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Form Revision Date: March 1, 2019

METHODS FOR MAKING L-GLUFOSINATE**CROSS-REFERENCE TO PRIORITY APPLICATIONS**

This application is a continuation of U.S. Application No. 18/179,136, filed March 6, 2023, which is a continuation of U.S. Application No. 17/530,018, filed November 18, 2021, which is a continuation of U.S. Application No. 16/997,133, filed August 19, 2020, now U.S. Patent No. 11,560,557, which is a continuation of U.S. Application No. 16/287,290, filed February 27, 2019, now U.S. Patent No. 10,781,465, which is a continuation of U.S. Application No. 15/787,448, filed October 18, 2017, now U.S. Patent No. 10,260,078, which is a division of U.S. Application No. 15/445,254, filed February 28, 2017, now U.S. Patent No. 9,834,802, which claims the benefit of U.S. Provisional Application No. 62/302,421, filed March 2, 2016; U.S. Provisional Application No. 62/336,989, filed May 16, 2016; and U.S. Provisional Application No. 62/413,240, filed October 26, 2016. The aforementioned applications are hereby incorporated herein by reference in their entireties.

INCORPORATION BY REFERENCE OF MATERIAL SUBMITTED ELECTRONICALLY

The Sequence Listing, which is a part of the present disclosure, is submitted concurrently with the specification as an .xml file. The name of the file containing the Sequence Listing is "M202647G_Seqlisting.xml", which was created on December 20, 2023 and is 5,022 bytes in size. The subject matter of the Sequence Listing is incorporated herein in its entirety by reference.

FIELD

Described herein are methods for producing a single stereoisomer of glufosinate, particularly for the production of L-glufosinate.

BACKGROUND

The herbicide glufosinate is a non-selective, foliarly-applied herbicide considered to be one of the safest herbicides from a toxicological or environmental standpoint. Current commercial chemical synthesis methods for glufosinate yield a racemic mixture of L- and D-glufosinate (Duke *et al.* 2010 *Toxins* 2:1943-1962). However, L-glufosinate (also known as phosphinothricin or (S)-2-amino-4-(hydroxy(methyl)phosphonoyl)butanoic acid) is much more potent than D-glufosinate (Ruhland *et al.* (2002) *Environ. Biosafety Res.* 1:29–37).

Therefore, methods are needed to produce only or primarily the active, L-glufosinate form. Previously, cost effective methods to generate pure L-glufosinate, or a mixture of D- and L-glufosinate enriched for L-glufosinate, have not been available. Described herein are new and cost-effective methods for the production of L-glufosinate.

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SUMMARY

Compositions and methods for making L-glufosinate are provided. The first step of the process involves the oxidative deamination of D-glufosinate to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). The second step involves the specific amination of PPO to L-glufosinate, using an amine group from one or more amine donors. In some
 10 embodiments, the method involves reacting D-glufosinate with a D-amino acid oxidase (DAAO) enzyme to form PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid); followed by aminating the PPO to L-glufosinate by a transaminase (TA) enzyme, using an amine group from one or more amine donors, wherein at least 70% of the D-glufosinate is eliminated and/or the yield of L-glufosinate is at least 85% of the input racemic glufosinate or at least 70 to 85% of the D-
 15 glufosinate is converted to L-glufosinate. In some embodiments, unreacted amine donor from one reaction can be reused in further rounds of reaction. Optionally, the D-glufosinate is originally present (i.e., in the reacting step) in a racemic mixture of D- and L-glufosinate.

The DAAO enzyme must have an increased activity of about 3 umol/min*mg or greater to drive the reaction. DAAO enzymes are available in the art and can be modified or mutated to
 20 have the necessary increased activity needed to drive the process. In this manner, mutant or modified enzymes from *Rhodospiridium toruloides* (UniProt P80324), *Trigonopsis variabilis* (UniProt Q99042), *Neolentinus lepideus* (GenBank KZT28066.1), *Trichoderma reesei* (GenBank XP_006968548.1), or *Trichosporon oleaginosus* (KLT40252.1) can be used. In some embodiments, the DAAO enzyme is a mutant DAAO based on the sequence from
 25 *Rhodospiridium toruloides*. While a number of mutations can be made and tested for the effect on activity, the mutant DAAO in some embodiments may comprise mutations at positions 54, 56, 58, 213, and/or 238. For example, the mutant DAAO can comprise one or more of the following mutations at position 54: N54C, N54L, N54T, or N54V. The mutant DAAO can optionally comprise the following mutation at position 56: T56M. The mutant DAAO can
 30 optionally comprise one or more of the following mutations at position 58: F58A, F58G, F58H, F58K, F58N, F58Q, F58R, F58S, or F58T. Optionally, the mutant DAAO can comprise the following mutation at position 213: M213S. In some embodiments, the mutant DAAO can

comprise one or more of the following combinations of mutations: F58K and M213S; N54T and T56M; N54V and F58Q; and/or N54V, F58Q, and M213S. In each case, the enzyme needs to have an activity of equal to or greater than about 3 $\mu\text{mol}/\text{min}\cdot\text{mg}$, greater than about 4 $\mu\text{mol}/\text{min}\cdot\text{mg}$, or higher. A wild type enzyme can be used in the methods of the invention as long as the enzyme has an activity level as set forth above.

The TA enzyme may be a gabT transaminase from *Escherichia coli* (UniProt P22256). Alternatively, the TA enzyme may be a transaminase with the sequence identified as SEQ ID NO: 1. The TA enzyme may also be selected on the basis of sequence similarity to SEQ ID NO: 1 and/or mutated to improve its activity in the desired reaction. Thus, sequences having 80%, 85%, 90%, 95% or greater sequence identity to SEQ ID NO: 1, based on the BLASTP method of alignment, and retain transaminase activity are encompassed by the present invention. Any DNA sequence encoding the enzyme sequence of SEQ ID NO: 1 or variants thereof are encompassed herein as well.

The reacting step and the aminating step can be performed in a single container or in separate containers. In one embodiment, all reagents are substantially added at the start of the reaction. Alternatively, the reagents for the reacting step and the reagents for the aminating step are added to the single container at different times.

Also described herein is a method for selectively controlling weeds in a field containing a crop of planted seeds or crops that may optionally be resistant to glufosinate, comprising applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate to the field. Also described herein is a method for selectively controlling weeds in a field, controlling weeds in non-field areas, defoliating plants or crops, and/or desiccating crops before harvest, comprising applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate and more than 0.01% but less than 15% PPO to the field.

The details of one or more embodiments are set forth in the drawings and the description below. Other features, objects, and advantages will be apparent from the description and drawings, and from the claims.

Some compositions of the invention comprise D-glufosinate, PPO, and L-glufosinate. In such compositions, L-glufosinate is present at a concentration equal to or greater than 80%, greater than 90%, greater than 95%, greater than 96%, greater than 97%, greater than 98%, or

Figure 1 is a schematic of an exemplary conversion of D-glufosinate to L-glufosinate. The amine donor and keto acid product are examples and are not intended to be limiting.

Figure 2 is a graph showing concentrations of L-glufosinate (circles), D-glufosinate (triangles), and PPO (squares) during a one-step de-racemization by a N54T, T56M, F58K, and M213S mutant variant of *Rhodospiridium toruloides* DAAO and the *E. coli* gabT transaminase.

Compositions and methods for the production of L-glufosinate (also known as phosphinothricin or (S)-2-amino-4-(hydroxy(methyl)phosphonoyl)butanoic acid) are provided. The methods comprise a two-step process, which may optionally occur in a single vessel and nearly simultaneously. The first step involves the oxidative deamination of D-glufosinate to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). The second step involves the specific amination of PPO to L-glufosinate, using an amine group from one or more amine donors. By combining these two reactions, the proportion of L-glufosinate can be substantially increased in a racemic glufosinate mixture. Thus, provided herein are methods to obtain a composition consisting substantially of L-glufosinate. Since L-glufosinate is more potent than D-glufosinate, smaller amounts of the composition are needed to be effective as a herbicide.

In one embodiment, described herein, is a composition comprising a mixture of L-glufosinate, PPO, and D-glufosinate, where L-glufosinate is the predominant compound among the mixture of L-glufosinate, PPO, and D-glufosinate. Such composition can be used directly as a herbicide as PPO can contribute herbicidal activity (EP0030424). In other embodiments, L-glufosinate can be purified or substantially purified and used as a herbicide.

Compositions of L-glufosinate may comprise D-glufosinate, PPO, and L-glufosinate. Optionally, the amount of L-glufosinate is 80% or greater, 85% or greater, 90% or greater, or about 95% or greater, 97% or greater, 98% or greater based on the combined weight of D-glufosinate, PPO, and L-glufosinate. Optionally, the amount of D-glufosinate is 10% or less, 5%

or less, 2.5% or less, or 1% or less based on the combined weight of D-glufosinate, PPO, and L-glufosinate. Optionally, the amount of PPO is more than 1% but less than 20%, less than 15%, less than 10%, or less than 5% based on the weight of D-glufosinate, PPO, and L-glufosinate. These compositions can optionally occur as dried powders or dissolved in aqueous or non-aqueous carrier and additional chemical species can optionally be present. Optionally, the composition is prepared and used in an ex vivo environment.

It is also recognized that the L-glufosinate can be further isolated and used in formulations as a herbicide.

Also described herein are formulations. The formulations comprise L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; one or more additional components selected from the group consisting of sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; dipropylene glycol in an amount from 4-18% by weight of the formulation; and alkyl polysaccharide in an amount from 4-20% by weight of the formulation; and water as the balance of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 24.5% by weight of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; alkylethersulfate, sodium salt in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an

amount of 1.0% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

While the methods can be used to produce a substantially purified L-glufosinate in a batch reaction, it is recognized that a continuous process can be used.

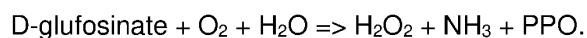
5 I. Methods of Synthesis

Methods for the conversion of D-glufosinate to L-glufosinate are provided. The methods described herein provide a means for converting a low cost feedstock of a racemic mixture of D- and L-glufosinate into a more valuable product that has been enriched for L-glufosinate. The methods for conversion includes two steps, which can occur in one or more separate
 10 containers. The first step is the oxidative deamination of D-glufosinate (which can be present in a racemic mixture of D- and L-glufosinate) to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). This step can be catalyzed by a D-amino acid oxidase (DAAO) enzyme, a D-amino acid dehydrogenase (DAAD) enzyme, or by chemical conversion. The second step is the specific
 15 amination of PPO to L-glufosinate, using an amine group from one or more amine donors. Such amine donors can be selected from glutamate, L-glutamate, lysine, alanine, isopropylamine, sec-butylamine, phenylethylamine and the like. This step can be catalyzed by a transaminase (TA) enzyme, an L-amino acid dehydrogenase (LAAD) enzyme, or by chemical conversion. Using the methods described herein, compositions of substantially purified L-glufosinate can be obtained.

20 Figure 1 sets forth an example of the conversion of D-glufosinate to L-glufosinate. As noted above, the method involves a two-step process. As illustrated, the first step is an oxidative deamination of D-glufosinate to PPO and the second step is an amination of PPO to L-glufosinate.

The first step, i.e., the oxidative deamination of D-glufosinate to PPO, can be catalyzed
 25 by several classes of enzymes or can occur non-enzymatically. Such enzymes include DAAO, DAAD, and D-amino acid dehydratase.

In one embodiment a DAAO enzyme is used to catalyze the conversion of D-glufosinate to PPO. Such a reaction has the following stoichiometry:



Since the solubility of oxygen in aqueous reaction buffer is typically low compared to that of D-glufosinate, for an efficient process, oxygen must be introduced throughout the time period of the DAAO reaction. This is in contrast to, for example, the Hawkes reaction set forth in US Patent Nos: 7,723,576; 7,939,709; 8,642,836; and 8,946,507 in which the reaction was

5 conducted in a sealed vessel. Initially, D-glufosinate is present at greater than 30 g/L up to as much as 140 g/L. The initial oxygen level is typically impacted by the reaction temperature, but is typically initially present at approximately 8 mg/L and is added throughout the reaction to allow for sufficient oxygen for the reaction to continue apace. Water is typically, but not obligately, present at greater than 500 g/L.

10 Several DAAO enzymes are known in the art and can be used in the methods described herein, as long as they are capable of accepting D-glufosinate as a substrate and provide an activity sufficient to level to drive the reaction. The DAAO enzymes useful in the methods of the invention have an activity of equal to or greater than about 3 $\mu\text{mol}/\text{min}\cdot\text{mg}$, greater than about 4 $\mu\text{mol}/\text{min}\cdot\text{mg}$, or higher. A wild type enzyme can be used in the methods of the invention as

15 long as the enzyme has an activity level as set forth above. Such DAAO enzymes that can be used in the method include those from *Rhodospiridium toruloides*, *Trigonopsis variabilis*, *Fusarium sp*, *Candida sp*, *Schizosaccharomyces sp*, *Verticillium sp*, *Neolentinus lepideus*, *Trichoderma reesei*, *Trichosporon oleaginosus*, and the like that have been modified to increase activity. Any DAAO enzyme can be used as a starting enzyme including those having

20 sequences corresponding to Swissprot accession numbers P80324, Q99042, P00371, and P24552 or SPTREMBL numbers Q9HGY3 and Q9Y7N4 or GenBank numbers KZT28066.1, XP_006968548.1, and KLT40252.1. The DNA sequences which encode the DAAO may be selected from sequences set forth in EMBL accessions A56901, RGU60066, Z50019, SSDA04, D00809, AB042032, RCDAAOX, A81420, and SPCC1450, or may be codon optimized from the

25 protein sequences indicated above for optimal expression in the chosen expression host(s). U.S. Patent No. 8,227,228 describes DAAO enzymes from *Candida intermedia*. Such sequences are herein incorporated by reference. These enzymes can be modified for increased activity and used in the methods of the invention.

30 Additional DAAO enzymes can be identified in a variety of ways, including sequence similarity and functional screens. The DAAO enzyme may be a mutant DAAO enzyme that is capable of accepting D-glufosinate as a substrate. In Hawkes *et al.*, *supra*, a mutant DAAO based on the sequence from *Rhodospiridium toruloides* (consisting of the F58K and M213S mutations) has been shown to accept D-glufosinate as a substrate (Hawkes *et al.* (2011) *Plant*

Biotechnol J. 9(3):301-14). Other DAAO enzymes can be similarly modified to accept D-glufosinate and have greater activity. i.e., the activity needed to drive the method of the invention. In the same manner, known DAAO enzymes may be improved by mutagenesis, and/or novel DAAO enzymes could be identified.

5 In some embodiments, mutant enzymes can be made and tested in the methods described herein. Mutant DAAO enzymes (e.g., from *Rhodotorula gracilis*) can include one mutation, two mutations, three mutations, or more than three mutations (e.g., four mutations, five mutations, six mutations, seven mutations, eight mutations, nine mutations, or ten mutations
10 or more) at positions in the mutant sequence as compared to the wild type sequence. The mutant DAAO can optionally comprise mutations at positions 54, 56, 58, 213, and/or 238. In some embodiments, such mutants can comprise amino acid substitutions at positions 54 and 56 when compared with the wild type sequence. In other embodiments, such mutants can comprise amino acid substitutions at positions 54 and 58 when compared to the wild type sequence. In other embodiments, such mutants can include amino acid substitutions at
15 positions 54, 213, and 238 when compared with the wild type sequence.

Optionally, at position 54, the wild type asparagine may be replaced by Ala, Cys, Gly, Ile, Ser, Leu, or, more preferably, Thr or Val. For example, the mutant DAAO can comprise one of the following mutations at position 54: N54C, N54L, N54T, or N54V.

Optionally, at position 56, the wild type threonine can be replaced by Ala, Cys, Gly, Ile,
20 Asn, Arg, Ser, Thr, Met, or Val. See, U.S. Patent No. 7,939,709, which is incorporated herein by reference. For example, the mutant DAAO can comprise the T56M mutation.

Additionally, at position 58, the wild type Phe can be replaced by Lys, Arg, Gln, Thr, Gly, Ser, Ala, Arg, Asn, or His. The mutant DAAO can optionally comprise one of the following mutations at position 58: F58A, F58G, F58H, F58K, F58N, F58Q, F58R, F58S, or F58T. In
25 some embodiments, the mutant DAAO does not include a mutation at position 58.

Optionally, at position 213, the wild type methionine is replaced by Arg, Lys, Ser, Cys, Asn, or Ala. In some examples, the mutant DAAO can comprise the mutation M213S.

Optionally, at position 238, the wild type tyrosine is replaced by His, Ser, Gys, Asn, or Ala.

In some embodiments, the mutant DAAO can comprise one or more of the following combinations of mutations: F58K and M213S; N54T and T56M; N54V and F58Q; N54C and F58H; N54T and F58T; N54T and F58G; N54T and F58Q; N54T and F58A; N54L and F58R; N54V and F58R; N54V and F58N; and/or N54V, F58Q, and M213S.

5 In one embodiment, the mutant DAAO comprises mutations in other DAAO enzymes in positions equivalent to positions 54, 56, 58, 213, and/or 238 of *Rhodospiridium toruloides* DAAO or *Trigonopsis variabilis* DAAO.

Other suitable D amino acid oxidases may be obtained preferably from fungal sources. Such DAAO enzymes can be identified and tested for use in the methods of the invention. To
 10 determine if the enzyme will accept D-glufosinate as a substrate, an oxygen electrode assay (Hawkes, 2011, *supra*), colorimetric assay (Berneman A, Alves-Ferreira M, Coatnoan N, Chamond N, Minoprio P (2010) Medium/High Throughput D-Amino Acid Oxidase Colorimetric Method for Determination of D-Amino Acids. Application for Amino Acid Racemases. *J Microbial Biochem Technol* 2: 139-146), and/or direct measurement (via high performance liquid
 15 chromatography (HPLC), liquid chromatography mass spectrometry (LC-MS), or similar) of product formation can be employed.

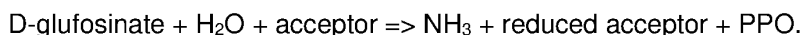
The reaction catalyzed by the DAAO enzyme requires oxygen. In some embodiments, oxygen, oxygen enriched air, an oxygen enriched gas stream, or air, is introduced to the reaction, either in the head space or by sparging gas through the reaction vessel, intermittently
 20 or continuously, to enhance the rate of reaction. Additionally, in other embodiments, optionally combined with sparging gas through the reaction vessel, a pressurized reactor may be used. That is, the reactor may be sealed and allowed to consume O₂. Using a sealed chamber would limit vapor emissions.

When a DAAO enzyme catalyzes the conversion of D-glufosinate to PPO, hydrogen
 25 peroxide (H₂O₂) evolves. This may be damaging to enzymes and other components of the biotransformation (e.g., products and/or substrates). Therefore, in one embodiment, an enzyme, such as catalase, can be used in addition to the DAAO enzyme to catalyze the elimination of hydrogen peroxide. Catalase catalyzes the decomposition of hydrogen peroxide with the following stoichiometry:

30
$$2\text{H}_2\text{O}_2 \Rightarrow 2\text{H}_2\text{O} + \text{O}_2.$$

In some embodiments, hydrogen peroxide can be eliminated using catalyzed and non-catalyzed decomposition reactions. For example, hydrogen peroxide can be eliminated by a non-catalyzed decomposition reaction using increased heat and/or pH. Hydrogen peroxide can also be eliminated by a catalyzed decomposition reaction using, for example, transition metals and other agents, such as potassium iodide. In addition to eliminating hydrogen peroxide, the use of catalase also produces oxygen (O₂). The production of oxygen by catalase can aid in facilitating the conversion of D-glufosinate to PPO using the DAAO enzyme, as DAAO requires oxygen to function.

Other enzymes can be used to catalyze the conversion of D-glufosinate to PPO. For example, a DAAD enzyme that accepts D-glufosinate as a substrate can be used with the following stoichiometry:



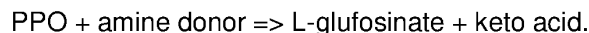
It is recognized that in methods where a DAAD is used, the DAAD catalyzed reaction can include redox cofactor recycling. This involves oxidizing the reduced acceptor so that it can accept more electrons from D-glufosinate.

In one embodiment, chemical oxidative deamination, wherein an intermediate α -keto acid is produced from the parent amino acid, can be used in the methods described herein to convert D-glufosinate to L-glufosinate. Chemical oxidative deamination involves the conversion of an amine group to a keto group with concomitant release of ammonia typically using metal ions such as those of copper or cobalt in an aqueous solution at temperatures between room temperature and the boiling point of the solution and at a pH in the range of about 4 – about 10. See, for example, Ikawa and Snell (1954) *J. Am. Chem. Soc.* 76 (19): 4900–4902, herein incorporated by reference.

The substantially complete (greater than 70%, greater than 75%, greater than 80%, greater than 85%, greater than 90%, or greater than 95%) conversion of D-glufosinate to PPO can occur within 24 hours, within 18 hours, within 12 hours, within 8 hours or less.

The second step of the method described herein involves the conversion of PPO to L-glufosinate using a transaminase (TA) enzyme, an L-amino acid dehydrogenase (LAAD) enzyme, or by chemical conversion. In one embodiment, the method is a reaction catalyzed by

a TA. A TA with the required stereospecificity that accepts PPO as a substrate catalyzes the amination of PPO to L-glufosinate with the following stoichiometry:



If the reaction is conducted as a two stage process where the D-glufosinate is
5 substantially converted to PPO in the absence of amine donor and/or transaminase, starting
amounts of PPO in the second stage typically range from 10g/L to 140g/L; 20g/L to 140g/L; or
from 30g/L to 140g/L. If the reaction is conducted in a single stage process, the starting
amounts of PPO are typically less than 1 g/L and the highest levels of PPO during the reaction
are typically less than 25 g/L. The amine donor is initially present at between 1 and 50 fold
10 molar excess over the starting amount of racemic glufosinate.

TAs useful in the methods described herein include the gabT transaminase from
Escherichia coli (UniProt P22256; identified herein as SEQ ID NO: 3), which has been shown to
catalyze the desired reaction with PPO as a substrate (Bartsch *et al.* (1990) *Appl Environ*
Microbiol. 56(1):7-12). Another enzyme has been evolved to catalyze the desired reaction at a
15 higher rate using isopropylamine as an amine donor (Bhatia *et al.* (2004) *Peptide Revolution:*
Genomics, Proteomics & Therapeutics, Proceedings of the Eighteenth American Peptide
Symposium, Ed. Michael Chorev and Tomi K. Sawyer, July 19-23, 2003, pp. 47-48). A
transaminase with the amino acid sequence of SEQ ID NO: 1 also catalyzes the desired
reaction with PPO and isopropylamine as the substrate (Example 11). Additionally, TA
20 enzymes from numerous microorganisms, such as *Streptomyces hygrosopicus*, *Streptomyces*
viridochromogenes, *Candida albicans*, and others can be used in the practice of the methods
described herein. In particular, see, for example, EP0249188, and U.S. Patent No. 5,162,212,
incorporated herein by reference. Where desired, the enzymes can be evolved by mutagenesis
to increase their activities. Mutant TA enzymes can be selected for desired activity by the
25 assays outlined in Schulz *et al.*, *Appl Environ Microbiol.* (1990) Jan. 56(1):1-6, and/or by direct
measurement of the products by HPLC, LC-MS, or similar products.

Additional TA enzymes for use in the methods can be identified by screening collections
of TAs, such as those sold by Prozomix Limited (Northumberland, United Kingdom),
SyncoZymes (Shanghai, China), Evocatal (Monheim am Rhein, Germany), Codexis (Redwood
30 City, CA), or Abcam (Cambridge, United Kingdom) for the desired activity. Alternatively,
sequence similarity can be used to identify novel TA enzymes. Finally, TA enzymes can also be
identified from organisms capable of catalyzing the desired reaction.

The selection of an appropriate amine donor is important for an economical conversion of D-glufosinate to L-glufosinate. A variety of issues may be considered, including the cost of the donor, equilibrium thermodynamics, potential recovery of the donor, separation of the keto acid product from the desired L-glufosinate, and others. Consequently, TA enzymes that accept
 5 several different amine donors can be used, including low cost amine donors such as L-aspartate or racemic aspartate, L-glutamate or racemic glutamate, L-alanine or racemic alanine, L-phenylethylamine or racemic phenylalanine, L-glycine or racemic glycine, L-lysine or racemic lysine, L-valine or racemic valine, L-serine or racemic serine, L-glutamine or racemic glutamine, isopropylamine, sec-butylamine, ethanolamine, 2-aminobutyric acid, and diaminopropionic
 10 acid. In some embodiments, the amine donor is not aspartate or aspartic acid (e.g., L-aspartic acid, D-aspartic acid, or racemic D,L-aspartic acid).

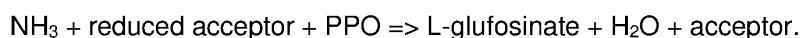
In embodiments where the amino donor is glutamate, the keto acid co-product that results from the transamination reaction is α -ketoglutarate (which is also referred to as α -ketoglutaric acid or α -KG). The α -ketoglutarate can be isolated and/or purified using methods
 15 known to those of skill in art, such as in EP Patent No. 0073711, CN Patent No. 10519873, CN Patent No. 105177065, CN Patent No. 104529755, and Zhan et al., *Shipin Yu Shengwu Jishu Xuebao*, 32(10): 1043-1048 (2013), each of which are incorporated herein by reference in their entireties. The produced and isolated α -ketoglutarate can be used in a variety of applications, including in synthesizing pharmaceutical agents, food additives, and biomaterials. Optionally,
 20 the α -ketoglutarate can be chemically converted to either racemic glutamate or L-glutamate, optionally for reuse in the reaction.

Chemical reductive amination involves the conversion of a keto group to an amine group via an imine compound typically by treatment of the keto compound with a suitable amine in an organic solution. Suitable amines include, for example, methylamine or ammonia. Suitable
 25 organic solvents for use in the organic solution include tetrahydrofuran, ethanol, or dichloromethane (DCM). The reductive amination can be performed at temperatures between room temperature and the boiling point of the solution. The produced imine can then be reduced using a reducing agent in an organic solution. Suitable reducing agents include, for example, NaBH_4 , $\text{NaHB}(\text{OAc})_3$, or $\text{Na}(\text{CN})\text{BH}_3$. Suitable organic solvents for use in the organic
 30 solution include tetrahydrofuran, ethanol, or DCM. The reduction reaction can be performed at temperatures between 0°C and the boiling point of the solution. Those skilled in the art will know that the process may be done in "one-pot" or in multiple containers, i.e., in separate transformations. Additionally, those skilled in the art will recognize that the described procedure

will provide racemic amino material where possible. The use of a chiral reducing agent such as $\text{RuCl}_2[(S)\text{-BINAP}]$ and hydrogen gas or latent source of hydrogen gas, or achiral hydride based reducing agents in the presence chiral ligands such as $(S)\text{-}$ or $(R)\text{-VAPOL}$ in a ratio between 1:1 to 1:0.05 can produce enantiomerically pure and/or enriched amino material. See, for example,
 5 Mignonac (1921) *Compt. Rend.* 172:223 and G. Li, Y. Liang, J. C. Antilla (2007) *J. Am. Chem. Soc.*, 129:5830-5831.

A wild type TA that accepts a desired amine donor can be identified, or a TA that does not normally accept a desired amine donor can be evolved to accept the desired substrate. Optionally, the transaminase is not an aspartate transaminase. Optionally, the transaminase is
 10 not 4-amino-butyrate: 2-ketoglutarate transaminase. In some embodiments, the transaminase is not a combination enzyme system that includes a PPT-specific transaminase and glutamate:oxaloacetate transaminase.

Other enzymes for use to catalyze the conversion of PPO to L-glufosinate include LAAD enzymes or imine reductases that accept PPO as a substrate. Such LAAD enzymes use the
 15 following stoichiometry:



LAAD catalyzed reactions can include redox cofactor recycling, which involves reducing the oxidized acceptor so that it can donate more electrons to PPO.

Chemical reductive amination, wherein an amino group is produced from the parent
 20 keto-compound, can also be used to produce glufosinate, in the case where no chiral reductant or ligand is used, or L-glufosinate, in the case where a chiral reductant or ligand is used. Chemical reductive amination can be affected as described above.

The substantially complete conversion of PPO to L-glufosinate may occur within 24 hours, within 18 hours, within 12 hours, within 8 hours, or within 4 hours. Substantially
 25 complete, in this context, means that the conversion of PPO to L-glufosinate is greater than about 70%, greater than about 75%, greater than about 80%, greater than about 85%, greater than about 90%, greater than about 95%, greater than about 98%, or greater than about 99%.

If the reaction occurs in a single container or vessel, the TA enzyme can be added with the DAAO enzyme or added at a later time, e.g., after the DAAO enzyme has been allowed to
 30 catalyze some or substantially all of the oxidative deamination.

Enzymes can be added to the reaction by a number of methods. One approach is to express the enzyme(s) in microorganism(s) such as *E. coli*, *S. cerevisiae*, *P. pastoris*, and others, and to add the whole cells to the reactions as whole cell biocatalysts. Another approach is to express the enzyme(s), lyse the microorganisms, and add the cell lysate. Yet another

5 approach is to purify, or partially purify, the enzyme(s) from a lysate and add pure or partially pure enzyme(s) to the reaction. If multiple enzymes are required for a reaction, the enzymes can be expressed in one or several microorganisms, including expressing all enzymes within a single microorganism.

A further approach, which can be combined with the above approaches, is to immobilize

10 enzyme(s) to a support (exemplary strategies are outlined in Datta *et al.* (2013) 3 Biotech. Feb; 3(1): 1–9). As outlined in Datta *et al.*, and not intending to be limiting, enzymes, either singly or in combination, can, for example, be adsorbed to, or covalently or non-covalently attached to, or entrapped within, natural or synthetic polymers or inorganic supports, including aggregates of the enzyme(s) themselves. Once immobilized, the enzyme(s) and support can be dispersed into

15 bulk solution or packed into beds, columns, or any number of similar approaches to interacting reaction solution with the enzymes. Since aeration is important for the DAAO reaction envisioned here, bubble columns or similar may be used for enzyme immobilization. As examples, reaction mixture can be flowed through a column of immobilized enzymes (flow reaction), added to a fixed bed or column of immobilized enzymes, allowed to react, and either

20 removed from the bottom or top of the reaction vessel (plug flow), or added to dispersed immobilized enzymes and allowed to react then the immobilized enzymes removed by filtration, centrifugation, or similar (batch). Thus, any method for immobilization of the enzymes may be employed in the methods of the invention.

The DAAO, TA, and/or other reactions can occur in a buffer. Exemplary buffers

25 commonly used in biotransformation reactions include Tris, phosphate, or any of Good's buffers, such as 2-(N-morpholino)ethanesulfonic acid (MES); N-(2-Acetamido)iminodiacetic acid (ADA); piperazine-N,N'-bis(2-ethanesulfonic acid) (PIPES); N-(2-Acetamido)-2-aminoethanesulfonic acid (ACES); β -Hydroxy-4-morpholinepropanesulfonic acid (MOPSO); choline chloride; 3-(N-morpholino)propanesulfonic acid (MOPS); N,N-Bis(2-hydroxyethyl)-2-aminoethanesulfonic acid

30 (BES); 2-[[1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl]amino]ethanesulfonic acid (TES); 4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid (HEPES); 3-(Bis(2-hydroxyethyl)amino)-2-hydroxypropane-1-sulfonic acid (DIPSO); acetamidoglycine, 3-(N-Tris(hydroxymethyl)methylamino)-2-hydroxypropanesulfonic acid (TAPSO); Piperazine-N,N'-

bis(2-hydroxypropanesulfonic acid) (POPSO); 4-(2-Hydroxyethyl)piperazine-1-(2-hydroxypropanesulfonic acid) (HEPPSO); 3-[4-(2-Hydroxyethyl)-1-piperazinyl]propanesulfonic acid (HEPPS); tricine; glycine; bicine; or 3-[[1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl]amino]propane-1-sulfonic acid (TAPS). Additional exemplary buffer recipes can be found in

5 Whittall, J. and Sutton, P. W. (eds) (2012) Front Matter, in Practical Methods for Biocatalysis and Biotransformations 2, John Wiley & Sons, Ltd, Chichester, UK. In some embodiments, ammonium can act as a buffer. One or more organic solvents can also be added to the reaction.

Surprisingly, the DAAO, TA, and/or other reactions can occur with no or low levels (less than 1 mM) of buffer added (other than ammonium that may optionally be present due to

10 addition of racemic glufosinate ammonium). In particular, immobilized DAAO and TA may be stable and active in the presence of less than 1 mM phosphate buffer and with no other buffer except any ammonium present due to the addition of racemic glufosinate ammonium.

The racemic glufosinate starting material can be provided in a number of forms. Various salts of racemic glufosinate, such as ammonium and hydrochloride, or the zwitterion, can be

15 used. The racemic glufosinate may be in the form of a solid powder (such as a powder of greater than 80%, 85%, 90%, or 95% purity) or an aqueous solution (such as a roughly 50% solution of racemic glufosinate).

In some embodiments, the reaction occurs within a defined pH range, which can be between pH 4 to pH 10 (e.g., between pH 6 and pH 9, such as approximately pH 7.5 to pH 8).

20 In some embodiments, the reaction occurs at a defined temperature. The temperature can be kept at a point between room temperature and the boiling point of the solvent, most typically between room temperature and 50 °C.

As indicated, the methods described herein provide a composition of substantially pure L-glufosinate (rather than a racemic mixture of L-glufosinate and D-glufosinate). Substantially

25 pure L-glufosinate means that greater than about 70%, greater than about 75%, greater than about 80%, greater than about 85%, greater than about 90%, greater than about 95%, greater than about 96%, greater than about 97%, greater than about 98%, or greater than about 99% of the D-glufosinate has been converted to L-glufosinate resulting in a composition having greater than about 80%, greater than about 85%, greater than about 90%, greater than about 95%,

30 greater than about 96%, greater than about 97%, greater than about 98%, or greater than about

99% L-glufosinate compared to the sum of the D-glufosinate and the L-glufosinate present in the composition.

In one embodiment, the L-glufosinate is not isolated from the biotransformation mixture and a composition comprising D-glufosinate, PPO, and L-glufosinate is obtained. This composition will contain at least 80% L-glufosinate by weight of the sum of L-glufosinate, D-glufosinate, and PPO, at least 90% L-glufosinate by weight of the sum of the components. This composition may be used directly as a herbicidal composition or as an ingredient in a formulated herbicidal product.

Alternatively, some or all of the components other than L-glufosinate can be removed from the biotransformation mixture, the mixture optionally concentrated, and then the mixture can be used directly (and/or with the addition of various adjuvants) for the prevention or control of weeds. The biotransformation mixture, in some instances, can be used directly (and/or with the addition of various adjuvants) for the prevention or control of weeds.

Additional steps to further purify the L-glufosinate can be added. Such further purification and isolation methods include ion exchange, extraction, salt formation, crystallization and filtration; each may be used multiple times or in suitable combination. Enzymes can be removed by simple filtration if supported, or if free in solution by the use of ultrafiltration, the use of absorbants like celite, cellulose or carbon, or denaturation via various techniques known to those skilled in the art.

Ion exchange processes effect separation by selective adsorption of solutes onto resins chosen for this purpose. Because products and impurities must be dissolved in a single solution prior to adsorption, concentration of the purified product stream by evaporation or distillation prior to isolation is usually required. Examples of the use of ion exchange for purification are described by Schultz et al., and in EP0249188(A2).

Purification may be achieved by the formation of an insoluble salt of L-glufosinate by the addition of a suitable acid, including hydrochloric acid, sulfuric acid, phosphoric acid, nitric acid, acetic acid and the like. Similarly, the purification may be achieved by the addition of a suitable base to form an insoluble salt. Useful bases include hydroxides, carbonates, sulfates and phosphates of alkali metals or hydroxides, carbonates, sulfates and phosphates of alkali earth metals. Other bases which contain nitrogen may be used, including ammonia, hydroxylamine, isopropylamine, triethylamine, tributylamine, pyridine, 2-picoline, 3-picoline, 4-picoline, 2,4-

lutidine, 2,6-lutidine, morpholine, N-methymorpholine, 1,8-diazabicyclo[5.4.0]undec-7-ene, and dimethylethanolamine. It may be advantageous to concentrate the mixture or to add a solvent (or both) to maximize yield and optimize purity of the desired salt. Solvents suitable for this purpose include those in which the solubility of the desired salt is very low (such solvents are often called "anti-solvents"). Salts of L-glufosinate can be transformed into forms of glufosinate suitable for formulation by standard methods known to those skilled in the art. Alternatively, the L-glufosinate can be isolated as a zwitterion.

US 9,255,115 B2 describes how the hydrochloric acid salt of L-glufosinate can be converted to the zwitterionic form with a base such as sodium hydroxide or sodium methoxide and then crystallized from aqueous alcohol solvent to afford L-glufosinate in relatively high purity. This method has the advantage of producing crystalline L-glufosinate that is not hygroscopic and therefore maintains a higher purity compared to amorphous L-glufosinate when exposed to humidity over time.

Other salts of L-glufosinate are known in the art. US 5,767,309 and US 5,869,668 teach the use of chiral alkaloid bases to form diastereomeric salts with racemic glufosinate. Purification is achieved because the salt of L-glufosinate precipitates from solution in much larger quantity than the corresponding salt of D-glufosinate. Therefore this method could be used with the present invention to obtain L-glufosinate with high enantiomeric excess, if desired.

Optionally, purification may be achieved by first crystallizing one or more impurities, removing the impurities by filtration and then further purifying L-glufosinate from the resulting filtrate by forming a salt as previously described. This is advantageous if unreacted amine donor can be partially or completely isolated and used in subsequent reactions. Similarly, unreacted PPO that is partially or completely isolated may be recycled for use in subsequent reactions.

Extraction may be used to purify the product. DE 3920570 C2 describes a process in which excess glutamic acid (used as the amine donor) is precipitated by adjusting the solution pH to 3.7 to 4.2 with sulfuric acid. After filtering the glutamic acid, the filtrate pH is lowered to 1-2 whereupon other impurities are extracted into a solvent. After extraction and concentration, ammonia is added to the aqueous solution to a pH of 5-7 whereupon ammonium sulfate precipitates. The ammonium sulfate is removed by filtration and the resulting filtrate is concentrated to afford the ammonium salt of L-glufosinate.

Isolation of L-glufosinate or its salts may be desirable, for example, for the purpose of shipping solids to the location of formulation or use. Typical industrial methods of isolation may be used, for example, a filtration, centrifugation, etc. Isolated product often requires the removal of water, volatile impurities and solvents (if present) and typical industrial drying equipment may be used for this purpose. Examples of such equipment include ovens, rotating drum dryers, agitated dryers, etc. In some cases, it may be advantageous to use a spray dryer.

It is not necessary to produce a solid product after purification. This may be advantageous if the formulation of L-glufosinate is to occur at the same site used for L-glufosinate production. L-glufosinate and many of its salts are readily soluble in water, and water is a convenient liquid to use for formulating products. For example, the amine donor is isolated by filtration and the resulting filtrate is concentrated by distillation. The pH of the filtrate may be adjusted to a desirable value and the resulting solution may be used as is or blended with formulation ingredients. In another example, a slurry of L-glufosinate or one of its salts may be prepared as described above and isolated by filtration. The solid could be dissolved directly on the filter by adding water or a suitable solvent to obtain a solution of L-glufosinate.

II. Compositions

Also described herein are compositions comprising the reaction products described above. In some embodiments, the composition substantially includes L-glufosinate and acceptable cationic or anionic salt forms such as the hydrochloride, ammonium, or isopropylammonium salts. In some embodiments, the composition comprises a mixture of L-glufosinate, PPO, and D-glufosinate.

Optionally, L-glufosinate is the predominant compound among L-glufosinate, PPO, and D-glufosinate. For example, L-glufosinate can be present in the composition in an amount of at least 80% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 85 % by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 90% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 95% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 96% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 97% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 98% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, or at least 99% by weight of the sum of L-glufosinate, PPO, and D-glufosinate.

The composition can include PPO in an amount up to 20% by weight of the sum of L-glufosinate, PPO, and D-glufosinate. Optionally, the composition includes from 0.001% to 20% PPO (e.g., from 0.05% to 15% or from more than 0.01% to less than 5% PPO). For example, the composition can include PPO in an amount of less than 20%, less than 19%, less than 18%,
 5 less than 17%, less than 16%, less than 15%, less than 14%, less than 13%, less than 12%, less than 11%, less than 10%, less than 9%, less than 8%, less than 7%, less than 6%, less than 5%, less than 4%, less than 3%, less than 2%, less than 1%, less than 0.5%, less than 0.1%, or less than 0.01% by weight of the sum of the masses of L-glufosinate, PPO, and D-glufosinate.

10 D-Glufosinate can be present in the composition in an amount of 15% or less by weight of the sum of L-glufosinate, PPO, and D-glufosinate. For example, D-glufosinate can be present in an amount of 14 % or less, 13 % or less, 12 % or less, 11 % or less, 10 % or less, 9 % or less, 8 % or less, 7 % or less, 6 % or less, 5 % or less, 4 % or less, 3 % or less, 2 % or less, 1 % or less, or 0.5 % or less by weight of the sum of L-glufosinate, PPO, and
 15 D-glufosinate.

In some embodiments, the composition can contain small amounts (e.g., about 10% or less, about 8% or less, about 5% or less, about 2% or less, or about 1% or less by weight of the composition) of D-glufosinate. In some embodiments, the composition can contain small amounts (e.g., about 15% or less, about 10% or less, about 8% or less, about 5% or less, about
 20 2% or less, or about 1% or less by weight of the composition) of PPO.

The compositions described herein are useful for application to a field of crop plants for the prevention or control of weeds. The composition may be formulated as a liquid for spraying on a field. The L-glufosinate is provided in the composition in effective amounts. As used herein, effective amount means from about 10 grams active ingredient per hectare to about
 25 1,500 grams active ingredient per hectare, e.g., from about 50 grams to about 400 grams or from about 100 grams to about 350 grams. In some embodiments, the active ingredient is L-glufosinate. For example, the amount of L-glufosinate in the composition can be about 10 grams, about 50 grams, about 100 grams, about 150 grams, about 200 grams, about 250 grams, about 300 grams, about 350 grams, about 400 grams, about 500 grams, about 550
 30 grams, about 600 grams, about 650 grams, about 700 grams, about 750 grams, about 800 grams, about 850 grams, about 900 grams, about 950 grams, about 1,000 grams, about 1,050 grams, about 1,100 grams, about 1,150 grams, about 1,200 grams, about 1,250 grams, about

1,300 grams, about 1,350 grams, about 1,400 grams, about 1,450 grams, or about 1,500 grams L-glufosinate per hectare.

The herbicidal compositions (including concentrates which require dilution prior to application to the plants) described herein contain L-glufosinate (i.e., the active ingredient), optionally some residual D-glufosinate and/or PPO, and one or more adjuvant components in liquid or solid form.

The compositions are prepared by admixing the active ingredient with one or more adjuvants, such as diluents, extenders, carriers, surfactants, organic solvents, humectants, or conditioning agents, to provide a composition in the form of a finely-divided particulate solid, pellet, solution, dispersion, or emulsion. Thus, the active ingredient can be used with an adjuvant, such as a finely-divided solid, a liquid of organic origin, water, a wetting agent, a dispersing agent, an emulsifying agent, or any suitable combination of these. From the viewpoint of economy and convenience, water is the preferred diluent. However, not all the compounds are resistant to hydrolysis and in some cases this may dictate the use of non-aqueous solvent media, as understood by those of skill in the art.

Optionally, one or more additional components can be added to the composition to produce a formulated herbicidal composition. Such formulated compositions can include L-glufosinate, carriers (e.g., diluents and/or solvents), and other components. The formulated composition includes an effective amount of L-glufosinate. Optionally, the L-glufosinate can be present in the form of L-glufosinate ammonium. The L-glufosinate ammonium can be present in an amount ranging from 10 % to 30 % by weight of the formulated composition. For example, the L-glufosinate ammonium can be present in an amount of 10 %, 12 %, 14 %, 16 %, 18 %, 20 %, 22 %, 24 %, 26 %, 28 %, or 30 % by weight of the formulated composition. Optionally, the L-glufosinate ammonium is present in an amount of 12.25 % or of 24.5%

In some examples, the formulated composition can include one or more surfactants. A suitable surfactant for use in the formulated composition includes sodium alkyl ether sulfate. The surfactant can be present in an amount from 10 % to 40 % by weight of the formulated composition. For example, the surfactant can be present in an amount of 10 %, 12 %, 14 %, 16 %, 18 %, 20 %, 22 %, 24 %, 26 %, 28 %, 30 %, 32 %, 34 %, 36 %, 38 %, or 40 % by weight of the formulated composition. Optionally, the sodium alkyl ether sulfate is present in an amount of 11.05%, 15.8 %, 22.1%, or 31.6 %.

The formulated composition can optionally include one or more solvents (e.g., organic solvents). Optionally, the solvent can be 1-methoxy-2-propanol, dipropylene glycol, ethylene glycol, and mixtures thereof. The one or more solvents can be present in an amount ranging from 0.5 % to 20 % by weight of the formulated composition. For example, the total amount of solvents in the composition can be present in an amount of 0.5% to 18 %, 5 % to 15 %, or 7.5 % to 10 % by weight of the formulated composition.

Optionally, the solvent includes a combination of two solvents. For example, the solvents in the formulation can include 1-methoxy-2-propanol and dipropylene glycol. The 1-methoxy-2-propanol can be present, for example, in an amount of 0.5 % to 2 % by weight of the formulated composition. For example, the 1-methoxy-2-propanol can be present in the amount of 0.5 %, 0.6 %, 0.7 %, 0.8 %, 0.9 %, 1.0 %, 1.1 %, 1.2 %, 1.3 %, 1.4 %, 1.5 %, 1.6 %, 1.7 %, 1.8 %, 1.9 %, or 2.0 % by weight of the formulated composition. Optionally, the 1-methoxy-2-propanol is present in an amount of 0.5 % or 1.0 % by weight of the formulated composition. The dipropylene glycol can be present in an amount of from 4 % to 18 % by weight of the formulated composition. For example, the dipropylene glycol can be present in an amount of 4 %, 6 %, 8 %, 10 %, 12 %, 14 %, 16 %, or 18 % by weight of the formulated composition. Optionally, the dipropylene glycol is present in an amount of 4.3 % or 8.6 % by weight of the formulated composition.

The formulated composition can also include one or more polysaccharide humectants. Examples of suitable polysaccharide humectants include, for example, alkyl polysaccharides, pentoses, high fructose corn syrup, sorbitol, and molasses. The polysaccharide humectant, such as alkyl polysaccharide, can be present in the formulated composition in an amount ranging from 4 % to 20 % by weight of the formulated composition. For example, the total amount of polysaccharide humectant in the composition can be present in an amount of 4 % to 18 %, 4.5 % to 15 %, or 5 % to 10 % by weight of the formulated composition. In some examples, the total amount of polysaccharide humectant, such as the alkyl polysaccharide, present in the formulated composition can be 4 %, 5 %, 6 %, 7 %, 8 %, 9 %, 10 %, 11 %, 12 %, 13 %, 14 %, 15 %, 16 %, 17 %, or 18 %. Optionally, the alkyl polysaccharide can be present in an amount of 3.2%, 4.9 %, 6.2%, or 9.8 %.

A diluent can also be included in the formulated composition. Suitable diluents include water and other aqueous components. Optionally, the diluents are present in an amount necessary to produce compositions ready for packaging or for use.

In one example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.

In another example, the formulated composition includes L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.

In another example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation.

In another example, the formulated composition includes L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

In another example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 58.45% by weight of the formulation.

In another example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 11.05% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of

the formulation; alkyl polysaccharide in an amount of 3.1% by weight of the formulation; and water in an amount of 73.1% by weight of the formulation.

Further components suitable for use in the formulated compositions provided herein are described in U.S. Patent Nos. 4,692,181 and 5,258,358, both of which are incorporated by
5 reference herein in their entireties.

The herbicidal compositions described herein, particularly liquids and soluble powders, can contain as further adjuvant components one or more surface-active agents in amounts sufficient to render a given composition readily dispersible in water or in oil. The incorporation of a surface-active agent into the compositions greatly enhances their efficacy. Surface-active
10 agent, as used herein, includes wetting agents, dispersing agents, suspending agents, and emulsifying agents are included therein. Anionic, cationic, and non-ionic agents can be used with equal facility.

Suitable wetting agents include alkyl benzene and alkyl naphthalene sulfonates, sulfated fatty alcohols, amines or acid amides, long chain acid esters of sodium isothionate, esters of
15 sodium sulfosuccinate, sulfated or sulfonated fatty acid esters petroleum sulfonates, sulfonated vegetable oils, ditertiary acetylenic glycols, polyoxyethylene derivatives of alkylphenols (particularly isooctylphenol and nonylphenol), and polyoxethylene derivatives of the mono-higher fatty acid esters of hexitol anhydrides (e.g. sorbitan). Exemplary dispersants include methyl cellulose, polyvinyl alcohol, sodium lignin sulfonates, polymeric alkyl naphthalene
20 sulfonates, sodium naphthalene sulfonate, polymethylene bisnaphthalenesulfonate, and sodium N-methyl-N- (long chain acid) laurates.

Water-dispersible powder compositions can be made containing one or more active ingredients, an inert solid extender, and one or more wetting and dispersing agents. The inert solid extenders are usually of mineral origin, such as the natural clays, diatomaceous earth, and
25 synthetic minerals derived from silica and the like. Examples of such extenders include kaolinites, attapulgite clay, and synthetic magnesium silicate. Water-dispersible powders described herein can optionally contain from about 5 to about 95 parts by weight of active ingredient (e.g., from about 15 to 30 parts by weight of active ingredient), from about 0.25 to 25 parts by weight of wetting agent, from about 0.25 to 25 parts by weight of dispersant, and from
30 4.5 to about 94.5 parts by weight of inert solid extender, all parts being by weight of the total

composition. Where required, from about 0.1 to 2.0 parts by weight of the solid inert extender can be replaced by a corrosion inhibitor or anti-foaming agent or both.

5 Aqueous suspensions can be prepared by dissolution or by mixing together and grinding an aqueous slurry of a water-insoluble active ingredient in the presence of a dispersing agent to obtain a concentrated slurry of very finely-divided particles. The resulting concentrated aqueous suspension is characterized by its extremely small particle size, so that when diluted and sprayed, coverage is very uniform.

10 Emulsifiable oils are usually solutions of active ingredient in water-immiscible or partially water-immiscible solvents together with a surface active agent. Suitable solvents for the active ingredient described herein include hydrocarbons and water-immiscible ethers, esters, or ketones. The emulsifiable oil compositions generally contain from about 5 to 95 parts active ingredient, about 1 to 50 parts surface active agent, and about 4 to 94 parts solvent, all parts being by weight based on the total weight of emulsifiable oil.

15 Compositions described herein can also contain other additaments, for example, fertilizers, phytotoxicants and plant growth regulants, pesticides, and the like used as adjuvants or in combination with any of the above-described adjuvants. The compositions described herein can also be admixed with the other materials, e.g., fertilizers, other phytotoxicants, etc., and applied in a single application.

20 In each of the formulation types described herein, e.g., liquid and solid formulations, the concentration of the active ingredients are the same.

25 In some embodiments, the composition can include α -ketoglutarate as the major component. α -ketoglutarate is an important dicarboxylic acid and one of the key intermediates in the tricarboxylic acid cycle and amino acid metabolism. α -ketoglutarate can be isolated from the reaction mixture by methods such as that set forth in French Patent No. 07199, herein incorporated by reference. The α -ketoglutarate composition can be formulated with pharmaceutical excipients and carriers, food additives, or components used to form biomaterials. The α -ketoglutarate composition can be used in a variety of applications, including in synthesizing pharmaceutical agents, food additives, and biomaterials, as described in Li et al., *Bioprocess Biosyst Eng*, 39:967-976 (2016).

It is recognized that the herbicidal compositions can be used in combination with other herbicides. The herbicidal compositions of the present invention are often applied in conjunction with one or more other herbicides to control a wider variety of undesirable vegetation. When used in conjunction with other herbicides, the presently claimed compounds can be formulated

5 with the other herbicide or herbicides, tank mixed with the other herbicide or herbicides or applied sequentially with the other herbicide or herbicides. Some of the herbicides that can be employed in conjunction with the compounds of the present invention include: amide herbicides such as allidochlor, beflubutamid, benzadox, benzipram, bromobutide, cafenstrole, CDEA, chlorthiamid, cyprazole, dimethenamid, dimethenamid-P, diphenamid, epronaz, etnipromid,

10 fentrazamide, flupoxam, fomesafen, halosafen, isocarbamid, isoxaben, napropamide, naptalam, pethoxamid, propyzamide, quinonamid and tebutam; anilide herbicides such as chloranocryl, cisanilide, clomeprop, cypromid, diflufenican, etobenzanid, fenasulam, flufenacet, flufenican, mefenacet, mefluidide, metamifop, monalide, naproanilide, pentanochlor, picolinafen and propanil; arylalanine herbicides such as benzoylprop, flamprop and flamprop-M;

15 chloroacetanilide herbicides such as acetochlor, alachlor, butachlor, butenachlor, delachlor, diethatyl, dimethachlor, metazachlor, metolachlor, S-metolachlor, pretilachlor, propachlor, propisochlor, prynachlor, terbuchlor, thenylchlor and xylachlor; sulfonanilide herbicides such as benzofluor, perfluidone, pyrimisulfan and profluazol; sulfonamide herbicides such as asulam, carbasulam, fenasulam and oryzalin; antibiotic herbicides such as bilanafos; benzoic acid

20 herbicides such as chloramben, dicamba, 2,3,6-TBA and tricamba; pyrimidinyloxybenzoic acid herbicides such as bispyribac and pyriminobac; pyrimidinylthiobenzoic acid herbicides such as pyrithiobac; phthalic acid herbicides such as chlorthal; picolinic acid herbicides such as aminopyralid, clopyralid and picloram; quinolinecarboxylic acid herbicides such as quinclorac and quinmerac; arsenical herbicides such as cacodylic acid, CMA, DSMA, hexaflurate, MAA,

25 MAMA, MSMA, potassium arsenite and sodium arsenite; benzoylcyclohexanedione herbicides such as mesotrione, sulcotrione, tefuryltrione and tembotrione; benzofuranyl alkylsulfonate herbicides such as benfuresate and ethofumesate; carbamate herbicides such as asulam, carboxazole chlorprocarb, dichlormate, fenasulam, karbutilate and terbucarb; carbanilate herbicides such as barban, BCPC, carbasulam, carbetamide, CEPC, chlorbufam,

30 chlorpropham, CPPC, desmedipham, phenisopham, phenmedipham, phenmedipham-ethyl, propham and swep; cyclohexene oxime herbicides such as alloxydim, butroxydim, clethodim, cloproxydim, cycloxydim, profoxydim, sethoxydim, tepraloxymid and tralkoxydim; cyclopropylisoxazole herbicides such as isoxachlortole and isoxaflutole; dicarboximide herbicides such as benzfendizone, cinidon-ethyl, flumezin, flumiclorac, flumioxazin and

flumipropyn; dinitroaniline herbicides such as benfluralin, butralin, dinitramine, ethalfluralin, fluchloralin, isopropalin, methalpropalin, nitratin, oryzalin, pendimethalin, prodiamine, profluralin and trifluralin; dinitrophenol herbicides such as dinofenat, dinoprop, dinosam, dinoseb, dinoterb, DNOC, etinofen and medinoterb; diphenyl ether herbicides such as ethoxyfen;

5 nitrophenyl ether herbicides such as acifluorfen, aclonifen, bifenox, chlomethoxyfen, chlomitrofen, etniproimid, fluorodifen, fluoroglycofen, fluoronitrofen, fomesafen, furyloxyfen, halosafen, lactofen, nitrofen, nitrofluorfen and oxyfluorfen; dithiocarbamate herbicides such as dazomet and metam; halogenated aliphatic herbicides such as alorac, chloropon, dalapon, flupropanate, hexachloroacetone, iodomethane, methyl bromide, monochloroacetic acid, SMA

10 and TCA; imidazolinone herbicides such as imazamethabenz, imazamox, imazapic, imazapyr, imazaquin and imazethapyr; inorganic herbicides such as ammonium sulfamate, borax, calcium chlorate, copper sulfate, ferrous sulfate, potassium azide, potassium cyanate, sodium azide, sodium chlorate and sulfuric acid; nitrile herbicides such as bromobonil, bromoxynil, chloroxynil, dichlobenil, iodobonil, ioxynil and pyraclozil; organophosphorus herbicides such as amiprofos-

15 methyl, anilofos, bensulide, bilanafos, butamifos, 2,4-DEP, DMPA, EBEP, fosamine, glyphosate and piperophos; phenoxy herbicides such as bromofenoxim, clomeprop, 2,4-DEB, 2,4-DEP, difenopenten, disul, erbon, etniproimid, fenteracol and trifopside; phenoxyacetic herbicides such as 4-CPA, 2,4-D, 3,4-DA, MCPA, MCPA-thioethyl and 2,4,5-T; phenoxybutyric herbicides such as 4-CPB, 2,4-DB, 3,4-DB, MCPB and 2,4,5-TB; phenoxypropionic herbicides such as cloprop,

20 4-CPP, dichlorprop, dichlorprop-P, 3,4-DP, fenoprop, mecoprop and mecoprop-P; aryloxyphenoxypropionic herbicides such as chlorazifop, clodinafop, clofop, cyhalofop, diclofop, fenoxaprop, fenoxaprop-P, fenthiaprop, fluazifop, fluazifop-P, haloxyfop, haloxyfop-P, isoxapyrifop, metamifop, propaquizafop, quizalofop, quizalofop-P and trifop; phenylenediamine herbicides such as dinitramine and prodiamine; pyrazolyl herbicides such as benzofenap,

25 pyrazolynate, pyrasulfotole, pyrazoxyfen, pyroxasulfone and topramezone; pyrazolylphenyl herbicides such as fluazolate and pyraflufen; pyridazine herbicides such as credazine, pyridafol and pyridate; pyridazinone herbicides such as brompyrazon, chloridazon, dimidazon, flufenpyr, metflurazon, norflurazon, oxapyrazon and pydanon; pyridine herbicides such as aminopyralid, clidinate, clopyralid, dithiopyr, fluroxypyr, haloxydine, picloram, picolinafen, pyriclor, thiazopyr

30 and triclopyr; pyrimidinediamine herbicides such as iprymidam and tioclorim; quaternary ammonium herbicides such as cyperquat, diethamquat, difenzoquat, diquat, morfamquat and paraquat; thiocarbamate herbicides such as butylate, cycloate, di-allate, EPTC, esprocarb, ethiolate, isopolinate, methiobencarb, molinate, orbencarb, pebulate, prosulfocarb, pyributicarb, sulfallate, thiobencarb, tiocarbamil, tri-allate and vemolate; thiocarbonate herbicides such as

dimexano, EXD and proxan; thiourea herbicides such as methiuron; triazine herbicides such as dipropetryn, triaziflam and trihydroxytriazine; chlorotriazine herbicides such as atrazine, chlorazine, cyanazine, cyprazine, eglazine, ipazine, mesoprazine, procyazine, proglazine, propazine, sebuthylazine, simazine, terbuthylazine and trietazine; methoxytriazine herbicides

5 such as atraton, methometon, prometon, secbumeton, simeton and terbumeton; methylthiotriazine herbicides such as ametryn, aziprotryne, cyanatryn, desmetryn, dimethametryn, methoprotryne, prometryn, simetryn and terbutryn; triazinone herbicides such as ametrudione, amibuzin, hexazinone, isomethiozin, metamitron and metribuzin; triazole herbicides such as amitrole, cafenstrole, epronaz and flupoxam; triazolone herbicides such as

10 amicarbazone, bencarbazone, carfentrazone, flucarbazone, propoxycarbazone, sulfentrazone and thienicarbazone-methyl; triazolopyrimidine herbicides such as cloransulam, diclosulam, florasulam, flumetsulam, metosulam, penoxsulam and pyroxsulam; uracil herbicides such as butafenacil, bromacil, flupropacil, isocil, lenacil and terbacil; 3-phenyluracils; urea herbicides such as benzthiazuron, cumyluron, cycluron, dichloralurea, diflufenzopyr, isonoruron, isouron,

15 methabenzthiazuron, monisouron and noruron; phenylurea herbicides such as anisuron, buturon, chlorbromuron, chloreturon, chlorotoluron, chloroxuron, daimuron, difenoxuron, dimefuron, diuron, fenuron, fluometuron, fluothiuron, isoproturon, linuron, methiuron, methylmymron, metobenzuron, metobromuron, metoxuron, monolinuron, monuron, neburon, parafluron, phenobenzuron, siduron, tetrafluron and thidiazuron; pyrimidinylsulfonyleurea

20 herbicides such as amidosulfuron, azimsulfuron, bensulfuron, chlorimuron, cyclosulfamuron, ethoxysulfuron, flazasulfuron, flucetosulfuron, flupyrsulfuron, foramsulfuron, halosulfuron, imazosulfuron, mesosulfuron, nicosulfuron, orthosulfamuron, oxasulfuron, primisulfuron, pyrazosulfuron, rimsulfuron, sulfometuron, sulfosulfuron and trifloxysulfuron; triazinylsulfonyleurea herbicides such as chlorsulfuron, cinosulfuron, ethametsulfuron,

25 iodosulfuron, metsulfuron, prosulfuron, thifensulfuron, triasulfuron, tribenuron, triflusulfuron and tritosulfuron; thiadiazolyleurea herbicides such as buthiuron, ethidimuron, tebuthiuron, thiazafluron and thidiazuron; and unclassified herbicides such as acrolein, allyl alcohol, aminocyclopyrachlor, azafenidin, benazolin, bentazone, benzobicyclon, buthidazole, calcium cyanamide, cambendichlor, chlorfenac, chlorfenprop, chlorflurazole, chlorflurenol, cinmethylin,

30 clomazone, CPMF, cresol, ortho-dichlorobenzene, dimepiperate, endothal, fluoromidine, fluridone, flurochloridone, flurtamone, fluthiacet, indanofan, methazole, methyl isothiocyanate, nipyraclufen, OCH, oxadiargyl, oxadiazon, oxaziclomefone, pentachlorophenol, pentoxazone, phenylmercury acetate, pinoxaden, prosulfalin, pyribenzoxim, pyriftalid, quinochloramine, rhodethanil, sulglycapin, thidiazimin, tridiphane, trimeturon, tripropindan and tritac. The

herbicidal compositions of the present invention can, further, be used in conjunction with glyphosate or 2,4-D on glyphosate-tolerant or 2,4-D-tolerant crops. It is generally preferred to use the compositions of the invention in combination with herbicides that are selective for the crop being treated and which complement the spectrum of weeds controlled by these compositions at the application rate employed. It is further generally preferred to apply the compositions of the invention and other complementary herbicides at the same time, either as a combination formulation or as a tank mix.

III. Methods of Use of L-Glufosinate Compositions

The compositions described herein can be used in methods for selectively controlling weeds in a field or any other area, including, for example, a railway, lawn, golf course, and others where the control of weeds is desired. Optionally, the field or other area can contain a crop of planted seeds or crops that are resistant to glufosinate. The methods can include applying an effective amount of a composition comprising L-glufosinate as described herein to the field.

The compositions described herein are useful for application to a field of crop plants for the prevention or control of weeds. The composition may be formulated as a liquid for spraying on a field. The L-glufosinate is provided in the composition in effective amounts. As used herein, effective amount means from about 10 grams active ingredient per hectare to about 1,500 grams active ingredient per hectare, e.g., from about 50 grams to about 400 grams or from about 100 grams to about 350 grams. In some embodiments, the active ingredient is L-glufosinate. For example, the amount of L-glufosinate in the composition can be about 10 grams, about 50 grams, about 100 grams, about 150 grams, about 200 grams, about 250 grams, about 300 grams, about 350 grams, about 400 grams, about 500 grams, about 550 grams, about 600 grams, about 650 grams, about 700 grams, about 750 grams, about 800 grams, about 850 grams, about 900 grams, about 950 grams, about 1,000 grams, about 1,050 grams, about 1,100 grams, about 1,150 grams, about 1,200 grams, about 1,250 grams, about 1,300 grams, about 1,350 grams, about 1,400 grams, about 1,450 grams, or about 1,500 grams L-glufosinate per hectare.

IV. Exemplary Embodiments

Non-limiting embodiments include:

1. A method for making L-glufosinate, comprising:
reacting D-glufosinate with a D-amino acid oxidase (DAAO) enzyme to form PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid); and
aminating the PPO to L-glufosinate by a transaminase (TA) enzyme, using an amine
5 group from one or more amine donors,
wherein at least 70% of the D-glufosinate is converted to L-glufosinate.
2. The method of embodiment 1, wherein the amine donor is selected from the group consisting of glutamate, L-glutamate, alanine, sec-butylamine, phenylethylamine, glycine, lysine, valine, serine, glutamine, isopropylamine, ethanolamine, 2-aminobutyric acid, and
10 diaminopropionic acid, or any secondary amine or amino acid.
3. The method of embodiment 1, wherein the D-glufosinate is originally present in a racemic mixture of D- and L-glufosinate or salts thereof.
4. The method of embodiment 1, wherein the DAAO enzyme is selected from the enzyme from *Rhodospiridium toruloides* or *Trigonopsis variabilis*, *Neolentinus lepideus*,
15 *Trichoderma reesei*, or *Trichosporon oleaginosus*. In an embodiment, the *Rhodospiridium toruloides* DAAO enzyme is UniProt P80324. In an embodiment, the *Trigonopsis variabilis* DAAO enzyme is UniProt Q99042. In an embodiment, the *Neolentinus lepideus* DAAO enzyme is KZT28066.1. In an embodiment, the *Trichoderma reesei* DAAO enzyme is XP_006968548.1. In an embodiment, the *Trichosporon oleaginosus* DAAO enzyme is KLT40252.1.
- 20 5. The method of embodiment 1, wherein the DAAO enzyme is a mutant DAAO.
6. The method of embodiment 5, wherein the mutant DAAO is a mutant DAAO based on the sequence from *Rhodospiridium toruloides*.
7. The method of embodiment claim 5, wherein the mutant DAAO comprises one or more mutations at positions 54, 56, 58, 213, and 238.
- 25 8. The method of embodiment 7, wherein the mutation at position 54 is selected from the group consisting of N54C, N54L, N54T, and N54V.
9. The method of embodiment 7, wherein the mutation at position 56 is T56M.

10. The method of embodiment 7, wherein the mutation at position 58 is selected from the group consisting of F58A, F58G, F58H, F58K, F58N, F58Q, F58R, F58S, and F58T.
11. The method of embodiment 7, wherein the mutation at position 213 is M213S.
12. The method of embodiment 5, wherein the mutant DAAO comprises mutations
5 F58K and M213S.
13. The method of embodiment 5, wherein the mutant DAAO comprises mutations at positions 54 and 56.
14. The method of embodiment 5, wherein the mutant DAAO comprises mutations N54T and T56M.
- 10 15. The method of embodiment 5, wherein the mutant DAAO comprises mutations F58Q or F58H.
16. The method of embodiment 5, wherein the mutant DAAO comprises mutations N54V and F58Q.
17. The method of embodiment 5, wherein the mutant DAAO comprises mutations
15 N54V, F58Q, and M213S.
18. The method of embodiment 1, wherein the TA enzyme is a gabT transaminase from *Escherichia coli*. In an embodiment, the *Escherichia coli* gabT transaminase is UniProt P22256.
19. The method of embodiment 1, wherein the TA enzyme is encoded by SEQ ID
20 NO: 1.
20. The method of embodiment 1, wherein the reacting step and the aminating step are performed in a single container.
21. The method of embodiment 20, wherein all reagents are substantially added at the start of the reaction.
- 25 22. The method of embodiment 20, wherein the reagents for the reacting step and the reagents for the aminating step are added to the single container at different times.

23. The method of embodiment 1, wherein the reacting step and the aminating step are performed in separate containers.

24. A composition comprising D-glufosinate, PPO, and L-glufosinate.

25. The composition of embodiment 24, wherein the amount of L-glufosinate is 90% or greater based on the total amount of D-glufosinate, PPO, and L-glufosinate.

26. The method of embodiment 1 wherein a solid is obtained having the composition of embodiment 24 or 25.

27. The method of embodiment 1 wherein a solution of L-glufosinate is obtained for use in a formulation which has herbicidal activity.

28. A formulation, comprising L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; one or more additional components selected from the group consisting of sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; dipropylene glycol in an amount from 4-18% by weight of the formulation; and alkyl polysaccharide in an amount from 4-20% by weight of the formulation; and water as the balance of the formulation.

29. The composition of embodiment 28, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.

30. The composition of embodiment 28, wherein the formulation comprises: L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 24.5% by weight of the formulation.

31. The composition of embodiment 28, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether

sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation.

5 32. A formulation, comprising L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; one or more additional components selected from the group consisting of sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; and alkyl polysaccharide in an amount from 3-10% by weight of the formulation; and water as the balance
10 of the formulation.

 33. The composition of embodiment 32, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the
15 formulation; and water in an amount of 58.45% by weight of the formulation.

 34. The composition of embodiment 32, wherein the formulation comprises: L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the
20 formulation; and water in an amount of 46.2% by weight of the formulation.

 35. The composition of embodiment 32, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 11.05% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; alkyl polysaccharide in an amount of 3.1% by
25 weight of the formulation; and water in an amount of 73.1% by weight of the formulation.

 36. A method for selectively controlling weeds in an area comprising:
 applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate to the area.

 37. The method of embodiment 36, wherein the amount of the composition is applied
30 at less than 400 grams of the sum of L-glufosinate and D-glufosinate per hectare.

38. A method for selectively controlling weeds in an area comprising:
applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate to the area and more than 0.01% but less than 10% PPO to the area.

5 39. The method of embodiment 38, wherein the amount of the composition is applied at less than 400 grams of the sum of L-glufosinate, D-glufosinate and PPO per hectare.

40. A method for selectively controlling weeds in an area containing a crop of planted seeds or crops that are resistant to glufosinate, comprising:
applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate and more than 0.01% but less than 10% PPO to the field.

The following examples are offered by way of illustration and not by way of limitation.

EXAMPLES

Example 1: DAAO enzyme purification

15 The coding sequence of a mutant DAAO from *Rhodospiridium toruloides* (for example, consisting of a MMARIRL leader sequence and the F58K and M213S mutations) was cloned into the pET14b vector to allow for expression of an N terminally 6xHis tagged protein. This pET14b-RgDAAO plasmid was transformed into BL21 (BE3) trxB pLysS cells. The sequence of the wild type DAAO from *Rhodospiridium toruloides* to which all numbering described here
20 corresponds, is:

MHSQKRVVVLGSGVIGLSSALILARKGYSVHILARDLPEDVSSQTFASPWAGANWTPF
MTLTDGPRQAKWEESTFKKWVELVPTGHAMWLKGTRRFAQNEDGLLGHWYKDITPNYRPLP
SSECPPGAIGVTYDTLSVHAPKYCQYLARELQKLGATFERRTVTSLEQAFDGADLVVNATGLG
AKSIAGIDDQAAEPIRGQTVLVKSPCKRCTMDSSDPASPAYIIPRPGGEVICGGTYGVGDWDL
25 VNPETVQRILKHCLRLDPTISSDGTIEGIEVLRHNVGLRPARRGGPRVEAERIVLPLDRTKSPLSL
GRGSARAAKEKEVTLVHAYGFSSAGYQQSWGAAEDVAQLVDEAFQRYHGAARESKL (SEQ
ID NO: 2).

To purify DAAO enzyme, cells were grown in 400 mL autoinducing medium (LB broth base with trace elements, Formedium) at 30°C for 20 to 24 hours. Cells were harvested in

precooled centrifuges and buckets, washed with cold water, centrifuged again, and stored at -80 °C until purification.

Cell pellets were then thawed in lysis buffer (50 mM potassium phosphate, pH 8.0, 20 mM imidazole and 1% Sigma protease inhibitor cocktail (PIC) w/o EDTA) at a volume of 5 mL of lysis buffer per 1 g cell pellet. While on ice, cells were sonicated 4 times for 30 seconds at an amplitude of 10. The cell lysate was clarified by centrifugation and then added to cobalt resin (HisPur Cobalt, ThermoScientific) at 4 times the bed volume. The cell lysate was incubated for 1 hour, with gentle shaking, at room temperature. The resin was added to a column and washed twice with 5 bed volumes of wash buffer (50 mM Kpi, pH 8.0, 20 mM imidazole). The elution was performed 4 times with 1 bed volume of elution buffer (50 mM Kpi, 200 mM imidazole).

Example 2: Colorimetric determination of DAAO activity

DAAO activity was determined similarly to Berneman et al. In brief, 100 uL of substrate and HRP (0.1 mg/mL HRP, Sigma P8375, and the desired amount of D-glufosinate or racemic D/L-glufosinate in 50 mM potassium phosphate, pH 8) was added to a Brand UV micro cuvette. To that, 50 uL of dyes (60 ug/mL TBHBA, Sigma 439533, and 1 mg/mL 4-aminoantipyrine, Sigma A4382, in 50 mM potassium phosphate, pH 8) was added and then 50 uL of enzyme mix (DAAO concentration as desired in 100 mM potassium phosphate, pH 8). The reaction was monitored on a spectrophotometer at 510 nm over an appropriate time to determine the enzyme kinetics. Although no flavin adenine dinucleotide (FAD) was added to the purification of DAAO or the reaction, this reagent can optionally be included. Two exemplary mutant variants of *Rhodospiridium toruloides* DAAO, AC201 (containing F58K and M213S) and AC263 (containing N54T, T56M, F58K, and M213S), purified as in Example 1, were tested using this assay and shown to produce hydrogen peroxide, demonstrating their activity in oxidizing D-glufosinate. AC201 and AC263 have similar V_{max} , but AC263 has a lower K_M .

Example 3: Purification of transaminases

To purify, for example, the *Escherichia coli* gabT transaminase (UniProt P22256), the gene was amplified from E. coli K12 strain ER2925 and cloned into pET-14b to generate an N-terminal 6xHis tagged version. This plasmid was then transformed into BL21 (DE3) cells for induction. After induction in autoinducing media, cells were lysed by sonication and the 6xHis tagged enzyme purified as described in Example 1.

Example 4: Demonstration of transaminase activity

In a non-limiting example, the source of PPO for a transamination assay can be D-glufosinate or racemic D/L-glufosinate that has been converted by a DAAO to PPO. In the first step, 39 mM racemic D/L-glufosinate was incubated with 0.5 mg/ml purified *Rhodospiridium*
5 *toruloides* DAAO F58K M213S and 10 ug/mL catalase in 50 mM Potassium phosphate buffer, pH8 for 20 hours at 30 C. This resulted in conversion of the majority of the D-glufosinate to PPO. Subsequently, purified *E. coli* gabT was added at 20 ug/mL and L-glutamate was added at 50 mM as the amine donor. At relevant points, samples were stopped by boiling for 10 minutes followed by precipitation with an equal volume of acetonitrile. The individual chemical species
10 were resolved on an HPLC with a Chirobiotic T2 column and quantified by comparison to authentic standards.

The combination of a mutant variant of DAAO and a transaminase resulted in an improvement of an enantiomeric enrichment that started at 0% for L-glufosinate over D-glufosinate (i.e., equal representation of D-glufosinate and L-glufosinate) to an enantiomeric
15 enrichment of 92%. These results demonstrate that *E. coli* gabT has transaminase activity, and this assay can be used to determine the activity of any number of wild type and/or mutant potential transaminases.

Example 5: De-racemization of racemic D/L-glufosinate in a single vessel

Reactions were set up similarly as in Example 4. The system (5.45 mL, 30 °C) was run
20 in phosphate buffer at a pH of 7.3. It was noted that 50 mM of phosphate buffer at a pH of 8.0 was inadequate to buffer the amino acid additions and that the unadjusted pH after amino acid additions in this system was pH 6.4. The pH was adjusted using 1M of the base salt K₂HPO₄ to a volume of 5.45 mL, meaning that the actual initial substrate concentration by addition was 275 mM. The following reagents were added essentially simultaneously at the start of the
25 reaction: 271 mg D,L-glufosinate, 420 mg glutamate, 15 mg AC263 DAAO, 50 µg catalase, and 1.0 mg *E. coli* gab T transaminase. Figure 2 shows that, when all reagents were added, the amount of D-PPT (D-glufosinate) diminished with only modest accumulation of PPO. This result indicates an efficient deracemisation of D/L-glufosinate into L-glufosinate by the RgDAAO/EcgabT enzyme couple.

Example 6: Demonstration of improved DAAO enzymes

Using protein mutagenesis strategies as outlined above, improved and variant DAAO enzymes were identified. The enzymes were assayed according to the procedures described below.

5 *Stock Solutions:*

The following dye stock solutions were prepared: a 20 mg/mL stock solution of 2,4,6-tribromo-3-hydroxybenzoic acid (TBHBA) in DMSO; and a 100 mg/mL stock solution of 4-aminoantipyrine (4-AAP) in water. The following enzyme stock solution was prepared: a 1 mg/mL stock solution of horseradish peroxidase (HRP) type 6 in a pH 8.0 potassium phosphate buffer. The following substrate stock solution was prepared: varying concentrations of D or DL amino acid in a pH 8.0 potassium phosphate buffer.

10

Reaction Mixes:

The following reaction mixtures were prepared:

Mix A is a combination of the substrate and HRP enzyme. Solutions were prepared for each substrate concentration to be assayed using reaction buffer. The solutions were two times the final substrate concentration and 0.2 mg/mL for the HRP solution.

15

Mix B is a dye mixture. To 5 mL of reaction buffer was added 120 μ L of TBHBA solution and 400 μ L of 4-AAP solution.

Mix C is an enzyme mixture. A 0.1 mg/mL solution of DAAO in reaction buffer was prepared. The final reaction concentration was 25 μ g/mL.

20

Protocol:

A spectrophotometer was used at a wavelength of 510 nm, which corresponds to the maximum absorbance for 4-AAP/TBHBA and is the point at which the extinction coefficient is 29400 M⁻¹ cm⁻¹. The temperature for performing the assays was 30 °C. The reaction kinetics were obtained by measuring every minute for 15 minutes. Between measurements, 20 seconds of orbital shaking at normal intensity was performed, followed by 10 seconds of settling time.

25

Using a 96-well plate, the following mixes (with replicates) were added in the following order using multi-channel: 100µl mix A, 50µl mix B, and 50µl mix C. The measurements were started immediately after the enzyme addition.

5 The enzyme kinetics were measured as described above, plotted on a Michaelis Menten graph, and used to calculate V_{max} and K_M . For the variant Ac302 (54V, 58Q, 213S), the V_{max} was 4.2 umol/min*mg.

This analysis was completed for a number of variant DAAO enzymes as above except that the Mix C stock was 0.2 mg/mL solution of DAAO and this final reaction concentration of DAAO was 50 ug/mL.

10 As shown below in Table 1, variant mutant DAAO enzymes showed a range of activities:

Table 1:

Variant	Mutations	Vmax (% of Ac302)
Ac263	54T, 56M, 58K, 213S	33
Ac302	54V, 58Q, 213S	100
Ac305	54C, 58H, 213S	88
Ac309	54T, 58T, 213S	71
Ac312	54T, 58G, 213S	74
Ac314	54T, 58Q, 213S	99
Ac316	54T, 58S, 213S	75
Ac318	54T, 58A, 213S	71
Ac319	54L, 58R, 213S	64
Ac320	54V, 58R, 213S	76
Ac322	54V, 58N, 213S	79

Example 7: De-racemization of racemic D/L-glufosinate at a 5 L reaction size

15 The scale of the de-racemization is increased using approaches familiar to those skilled in the art. Reagents and their relative ratios are substantially similar to Example 5, but the amounts are significantly greater. Rather than tubes in shakers, the reactions are performed in stirred jacketed reactors, including, optionally, air or oxygen sparging of the broth or headspace. These reactors vary in size, from less than 10 mL reaction to tens or hundreds of thousands of
 20 liters. Stirring rates are chosen to increase reaction mixing and rate while minimizing power consumption and shear.

In one example, the reaction was run at the 5 L scale. The system (5 L, 30 °C) was run in 200 mM phosphate buffer at a pH of 8.0 in a stirred, jacketed reactor. The following reagents were added essentially simultaneously at the start of the reaction: 300 mM D,L-glufosinate, 900 mM glutamate, 7.5 g AC302 DAAO, 0.2 g catalase, and 1.0 g *E. coli* gab T transaminase. In addition, 500 mL isopropanol was added to control foaming. During the course of the reaction, air was introduced at 0.3 VVM (volumes of air per volume of reaction mixture per minute).

HPLC analysis of the reaction demonstrated that equilibrium was reached within 8 hours, with the enantiomeric excess of L-glufosinate over D-glufosinate greater than 99% and the ratio of L-glufosinate to PPO 90% to 10%. This result indicates an efficient deracemisation of D/L-glufosinate into L-glufosinate by the RgDAAO/EcgabT enzyme couple at the larger scale.

Example 8: Impact of oxygen on reaction rate

Although stirred, jacketed reactors or immobilized columns typically allow for some oxygen transfer, the rate of oxygen uptake afforded by passive aeration is not sufficient for an efficient process. In one example, a reaction was run in the same vessel as Example 7 under substantially the same conditions, but under reduced (0.01 VVM), with twice the AC302 DAAO on a volumetric basis (3 g/L versus 1.5 g/L), and without the isopropanol. In this case, the reaction took more than 60 hours to achieve equilibrium, demonstrating the critical importance of aeration for an efficient reaction.

Example 9: Co-immobilization of DAAO and TA

DAAO and TA enzymes were co-immobilized on EziG controlled pore glass beads (EnginZyme). 100mg of EziG type 3 beads were shaken at room temperature with 3ml of solution containing 16 mg of purified AC302 DAAO and 1.6mg of purified gabT in 50 mM potassium phosphate buffer pH 7.5, 0.5M NaCl, 20mM imidazole in a 50 ml Falcon tube. After 30 minutes, beads were spun down, immobilizing solution was removed, and beads were washed 3 times with 10ml of 100 mM potassium phosphate buffer pH 7.5.

The reaction was started by adding all other components to the washed beads. The reaction mix contained 300 mM D/L-glufosinate, 900 mM L-glutamic acid, 50 ug catalase, 198 mM potassium phosphate in 2.5 mL. The reaction was incubated at 30C with shaking (250 rpm) in a 50 mL tube covered in parafilm with holes poked through for gas exchange.

After 1 hour, the depletion of D-glufosinate and formation of L-glufosinate was determined by HPLC and these rates calculated. After 6 hours, beads were spun down, reaction mixture was removed, and beads were washed 3 times with 10ml of 100 mM Potassium Phosphate buffer pH 7.5. Beads were then stored at 4 C for 18 to 72 hours before the reaction
5 was repeated, for a total of 15 times, after which the retained activity was greater than 50% of the initial activity.

Example 10: Effect of buffer on reaction

When soluble AC302 DAAO and *E. coli* gabT TA enzymes are used, phosphate buffer at >50 mM is required for full activity. A 100 mL reaction was incubated at 30C with shaking (250
10 rpm) in a 500 mL flask covered in parafilm. An air pump was used to bubble air through the reaction for the first 5 hours. The air pump was removed for overnight incubation so the reaction would not bubble over and new parafilm with air holes was used for gas exchange. The reaction mix contained 300 mM D/L-glufosinate, 905 mM L-glutamic acid, 80 mg AC302 DAAO (0.8 mg/mL), 14.5 mg gabT (0.145 mg/mL), 2 mg catalase, and isopropanol as anti-foam reagent
15 (10% initial concentration, isopropanol was additionally added at 2hr (2mL), 3hr (1mL), 3.5hr (1mL), and at 4hr (2mL)). 500 uL of 1N NaOH (added before enzymes) was used to adjust pH from about 6 to about 7. pH remained at about 7 for the entire reaction without further adjustment. Due to the potassium phosphate in the stock enzyme buffer, the final mixture was 45 mM phosphate buffer. When compared to a similar reaction with 200 mM phosphate buffer,
20 the reaction rate was 50-60% of that of the reaction with the 200 mM buffer.

When immobilized AC302 DOOA and *E. coli* gatT TA enzymes are used, phosphate buffer of less than 1 mM is sufficient for full activity. Immobilized proteins were prepared and reaction performed as in Example 9 for the "Buffered" reaction. In addition, immobilized proteins were prepared and reactions performed as in Example 9 for the "pH 7" reaction, except that
25 sodium hydroxide was used to adjust the pH of the reaction to pH 7 and no phosphate buffer was added (residual phosphate buffer from the enzyme storage buffer is less than 1 mM). This work demonstrated that the initial reaction rate for both the DAAO and combined DAAO and gabT reactions are very similar with and without the addition of phosphate buffer when immobilized enzymes are used.

Example 11: Isopropylamine as an amine donor

Isopropylamine can be used as an amine donor for conversion of PPO to L-glufosinate with the use of an appropriate TA. PPO was converted to L-glufosinate in a reaction with the following components:

- 5 • 0.25 mg/mL TA encoded by SEQ ID NO: 1
- 25 mM PPO
- 0.2 mM pyridoxal phosphate
- 250 mM isopropylamine (pHed to 8 w/ H₃PO₄)
- 100 mM Kphos buffer pH 8.0
- 10 The reaction was incubated at 25 to 30 °C for 30 hours with gentle shaking (250 rpm). At 0 hours, the amount of L-glufosinate as measured by HPLC was 0 mM, at 20 hours it was 14 mM, and at 30 hours it was 18 mM. This demonstrates that the enzyme encoded by SEQ ID NO: 1 can convert PPO into L-glufosinate.

Example 12: Lysine as an amine donor

- 15 Lysine can be used as an amine donor for conversion of PPO to L-glufosinate with the use of an appropriate TA. PPO was converted to L-glufosinate in a reaction with the following components:

- 0.4 mg/mL gabT (purified as in Example 3)
- 25 mM PPO (pHed to 8 w/ NaOH)
- 20 • 0.2 mM pyridoxal phosphate
- 75 mM L-Lysine dihydrochloride (pHed to 8 w/ NaOH)
- 100 mM Kphos buffer pH 8.0

- The reaction was incubated at 30 °C for 20 hours with shaking (250 rpm). L-glufosinate was formed at a rate of 0.4 mM/hr over the 20 hours. This demonstrates that L-lysine can be
- 25 used to convert PPO to L-glufosinate.

Example 13: Purification and isolation of L-glufosinate

Several batches prepared following the procedure described in Example 9 but at larger scale were generated. After the beads were removed, each batch was heated to 90 °C for at

least 10 minutes, and after cooling to 20-25 °C, filtered to remove a small amount of solids. To each individual batch was added 37% HCl, dropwise, to effect the precipitation of glutamic acid. The amount of 37% HCl added was approximately 10% of the volume of the batch. The resulting white solid was removed by filtration. The batches were combined and concentrated under vacuum to an oil; the oil contained approximately 153 grams of L-glufosinate. The oil was diluted with five volumes of water and 37% HCl was added to adjust the solution to pH 1. The solution was treated sequentially with two portions each of approximately 3.0 kg of prewashed DOWEX 50WX8 cation exchange resin. In each treatment, the solution was allowed to mix with the resin for 30 minutes after which the resin was isolated on a filter. Both portions of resins were combined and washed first with water and then eluted with 4M NH₄OH. The eluent was concentrated under vacuum to an oil; PPO and 2-oxoglutarate were not present in the oil. Approximately 100 grams of the oil was diluted with water and the aqueous ammonium hydroxide was added until the pH was approximately 9. To the batch was added 1.0 kg of prewashed DOWEX Monosphere (hydroxide form) anion exchange resin and the mixture was stirred for approximately 40 minutes. An equal amount of DOWEX Monosphere resin, prewashed, was charged to a glass column. The slurry of DOWEX resin in water was added to the column on top of the prewashed resin. 800 mL of water was charged to the column followed by 0.1 N acetic acid, which was kept flowing through the column until all of the glutamic acid had eluted as determined by HPLC. 4 N acetic acid was fed to the column until all of the L-glufosinate had eluted from the column as determined by HPLC. The solution of L-glufosinate was concentrated under vacuum. The resulting oil was diluted with water and concentrated under vacuum to minimum volume two times. Methanol was added until a clear solution was obtained and an equal volume of heptane was added. The mixture was concentrated under vacuum to minimum volume and the procedure was repeated. The remaining 168 grams of oil recovered from the cation exchange treatment was treated in a similar fashion to obtain an overall total of 108 grams of crude L-glufosinate. The ratio of L-glufosinate to glutamic acid was greater than 99:1 as determined by NMR. The resulting solid was mixed with aqueous ammonium hydroxide and concentrated to dryness to afford 111 grams of L-glufosinate ammonium. Neither methanol nor acetic acid was detected by NMR analysis of the product.

It is understood that the terminology used herein is for the purpose of describing particular embodiments only, and the terminology is not intended to be limiting. The scope of the invention will be limited only by the appended claims. Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by

one of ordinary skill in the art to which this invention belongs. Where a range of values is provided, it is understood that each intervening value, to the tenth of the unit of the lower limit unless the context clearly dictates otherwise, between the upper and lower limit of that range and any other stated or intervening value in that stated range, is encompassed within the invention. The upper and lower limits of these smaller ranges may independently be included in the smaller ranges and are also encompassed within the invention, subject to any specifically excluded limit in the stated range. Where the stated range includes one or both of the limits, ranges excluding either or both of those included limits are also included in the invention. Certain ranges are presented herein with numerical values being preceded by the term "about." The term "about" is used herein to provide literal support for the exact number that it precedes, as well as a number that is near to or approximately the number that the term precedes. In determining whether a number is near to or approximately a specifically recited number, the near or approximating unrecited number may be a number, which, in the context in which it is presented, provides the substantial equivalent of the specifically recited number.

All publications, patents, and patent applications cited in this specification are incorporated herein by reference to the same extent as if each individual publication, patent, or patent application were specifically and individually indicated to be incorporated by reference. Furthermore, each cited publication, patent, or patent application is incorporated herein by reference to disclose and describe the subject matter in connection with which the publications are cited. The citation of any publication is for its disclosure prior to the filing date and should not be construed as an admission that the invention described herein is not entitled to antedate such publication by virtue of prior invention. Further, the dates of publication provided might be different from the actual publication dates, which may need to be independently confirmed.

It is noted that the claims may be drafted to exclude any optional element. As such, this statement is intended to serve as antecedent basis for use of such exclusive terminology as "solely," "only," and the like in connection with the recitation of claim elements, or use of a "negative" limitation. As will be apparent to those of skill in the art upon reading this disclosure, each of the individual embodiments described and illustrated herein has discrete components and features which may be readily separated from or combined with the features of any of the other several embodiments without departing from the scope or spirit of the invention. Any recited method may be carried out in the order of events recited or in any other order that is logically possible. Although any methods and materials similar or equivalent to those described

herein may also be used in the practice or testing of the invention, representative illustrative methods and materials are now described.

SEQUENCES

SEQ ID NO: 1:

5 MNIAAQSWERREATSFFHTFTDLP SLKTDGPVIIDHGEGPYIIDTVGRRYFEGNSGLWNMTLGF
SERRLSDAALKQYQEFPGYHTFFGRNSKPTVELAERMLKLAPAPMSRVFFFTNSGSEANESIVK
LLWMMWAAEGRPERRKL TRKNAYHGATVMASALTGKD YVKA FGLPGPEIVTLDCPHAWRFA
LPGEGDDEFAARLAANLETRILQEGPETIAGMFAEPVMGAGGVIVPPATYFAKI QPVLQRYGIPL
IADEVICGFGR TGSLWGT LAVGQQPD IIVASKSMSAGYFPMGAVMLSADIDKRATAASEVWEEF
10 PHGFTTGGHPVGC AISLEAIRIITEEGVFENVKSVSETFQSGLRALADHPMIGEARGMGLMGAL
ETVADKKTQSFSGDLRIGERISKEARDRGFIIRPLGSSVVLAPPFISTHGQIEELLAVLKEVLDV
VYGT VKGEVA

SEQ ID NO: 2:

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15 GPRQAKWEESTFKKWVELVPTGHAMWLKGTRRFAQNEDG LLGHWYKDITPNYRPLPSSECP
PGAIGVTYDTLSVHAPKYCQYLARELQKLGATFERRTVTSLEQAFDGADLVVNATGLGAKSIAG
IDDQAAEPIRGQTVLVKSPCKRCTMDSSDPASPAYIIPRPGGEVICGGTYGVGDWDL SVN PETV
QRILKHCLRLDPTISSDGTIEGIEVLRHNVGLRPARRGGPRVEAERIVLPLDRTKSPLSLGRGSA
RAAKEKEVTLVHAYGFSSAGYQQSWGAAEDVAQLVDEAFQRYHGAARESKL

20 SEQ ID NO: 3:

MNSNKELMQRRSQAIPRGV GQIHPIFADRAENCRVWDVEGREYLDFAGGIAVLNTGHLHPKV V
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RSGTIAFSGAYHGRTHYTLALTGKVNPYSAGMGLMPGHVYRALYPCPLHGISEDDAIASIHRIFK
NDAAPEDIAAIVIEPVQGE GGFYASSPAFMQRLRALCDEHGIMLIADEVQSGAGRTGTLFAMEQ
25 MG VAPDLTTFAKSIAGGFPLAGVTGRAEVM DAVAPGGLGGTYAGNPIACVA AEVLKVFEQEN
LLQKANDLGQKLKDGLLAIAEKHPEIGDVRGLGAMIAIELFEDGDHNKPDAKLTA EIVARARDKG
LILLSCGPYYNVLRILVPLTIEDAQIRQGLEIISQCFDEAKQ

WHAT IS CLAIMED IS:

1. A formulation comprising:
L-glufosinate ammonium in an amount from 10-30% by weight of the formulation;
one or more additional components selected from the group consisting of:
 - 5 sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation;
 - 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation;
 - dipropylene glycol in an amount from 4-18% by weight of the formulation;
 - alkyl polysaccharide in an amount from 4-20% by weight of the formulation; and
- 10 water as the balance of the formulation.
2. The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 31.6%
15 by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.
- 20 3. The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl
25 polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 24.5% by weight of the formulation.

4. The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation.
5. The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 58.45% by weight of the formulation.
6. The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

ABSTRACT

Methods for the production of L-glufosinate (also known as phosphinothricin or (S)-2-amino-4-(hydroxy(methyl)phosphonoyl)butanoic acid) are provided. The methods comprise a two-step process. The first step involves the oxidative deamination of D-glufosinate to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). The second step involves the specific amination of PPO to L-glufosinate, using an amine group from one or more amine donors. By combining these two reactions, the proportion of L-glufosinate in a mixture of L-glufosinate and D-glufosinate can be substantially increased.

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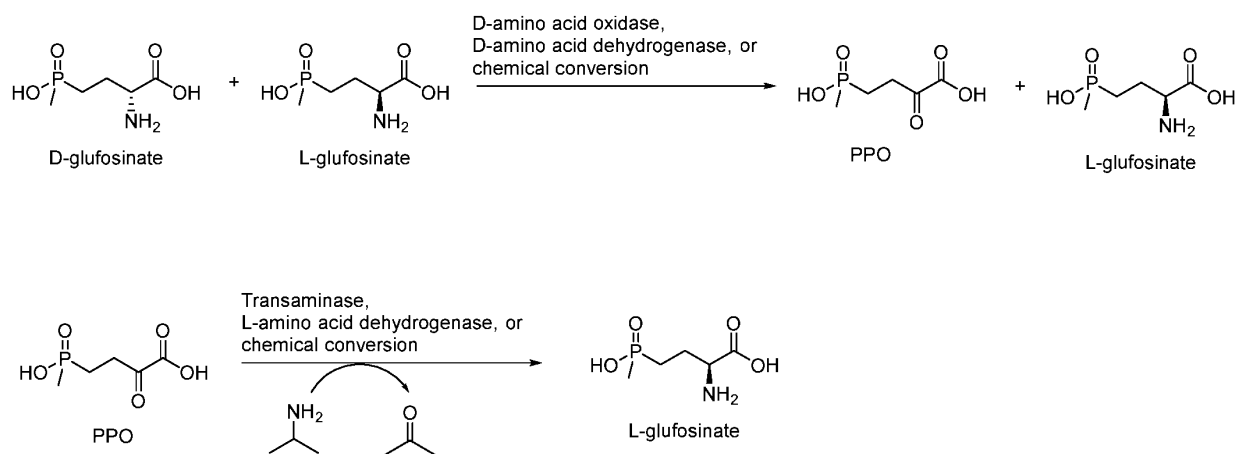
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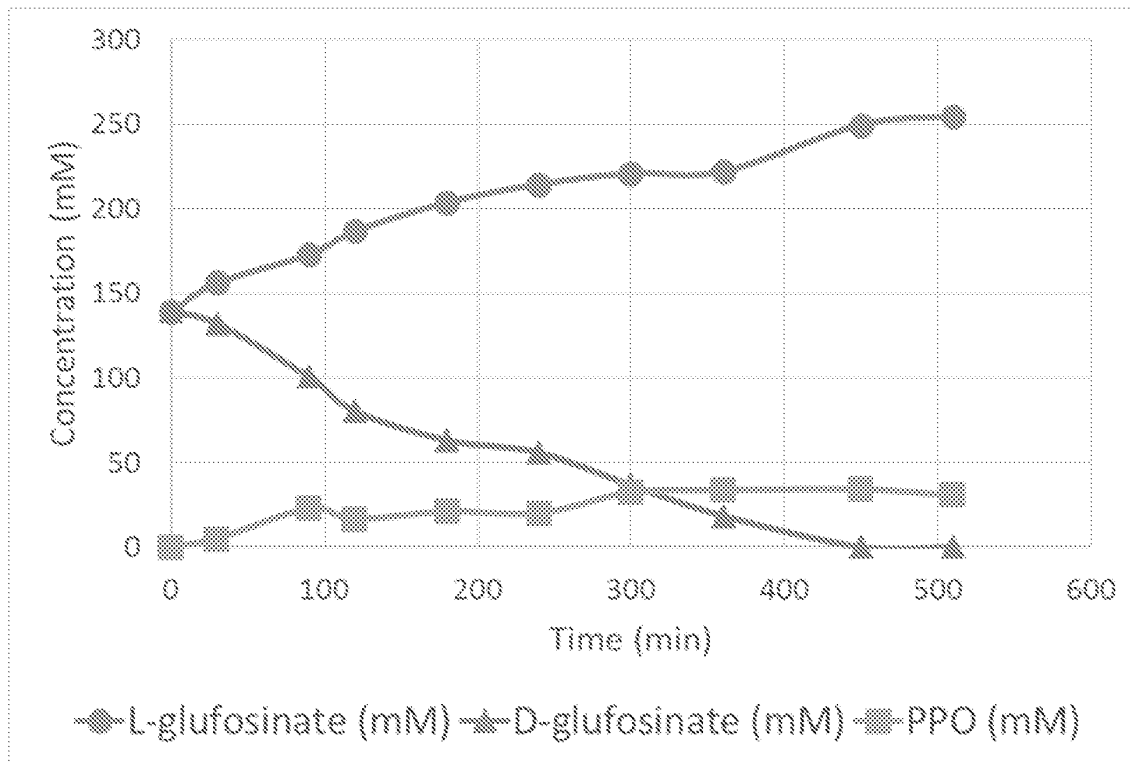
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**Figure 1**

**Figure 2**

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	32471/M202647G
		Application Number	
Title of Invention	METHODS FOR MAKING L-GLUFOSINATE		
The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76. This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.			

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City	Lutherville	State/Province	MD		
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Title of the Invention	METHODS FOR MAKING L-GLUFOSINATE		
Attorney Docket Number	32471/M202647G	Small Entity Status Claimed	☐
Application Type	Nonprovisional		
Subject Matter	Utility		
Total Number of Drawing Sheets (if any)	2	Suggested Figure for Publication (if any)	

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For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

Application number of the previously filed application	Filing date (YYYY-MM-DD)	Intellectual Property Authority or Country

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	32471/M202647G
		Application Number	
Title of Invention	METHODS FOR MAKING L-GLUFOSINATE		

Domestic Benefit/National Stage Information:

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, 365(c), or 386(c) or indicate National Stage entry from a PCT application. Providing benefit claim information in the Application Data Sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78.

When referring to the current application, please leave the "Application Number" field blank.

Prior Application Status	Pending	Remove			
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)		
	Continuation of	18/179136	2023-03-06		
Prior Application Status	Pending	Remove			
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)		
18/179136	Continuation of	17/530018	2021-11-18		
Prior Application Status	Patented	Remove			
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)	Patent Number	Issue Date (YYYY-MM-DD)
17/530018	Continuation of	16/997133	2020-08-19	11560577	2023-01-24
Prior Application Status	Patented	Remove			
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)	Patent Number	Issue Date (YYYY-MM-DD)
16/997133	Continuation of	16/287290	2019-02-27	10781465	2020-09-22
Prior Application Status	Patented	Remove			
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)	Patent Number	Issue Date (YYYY-MM-DD)
16/287290	Continuation of	15/787448	2017-10-18	10260078	2019-04-16
Prior Application Status	Patented	Remove			
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)	Patent Number	Issue Date (YYYY-MM-DD)
15/787448	Division of	15/445254	2017-02-28	9834802	2017-12-05
Prior Application Status	Expired	Remove			
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)		
15/445254	Claims benefit of provisional	62/413240	2016-10-26		
Prior Application Status	Expired	Remove			
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)		
15/445254	Claims benefit of provisional	62/336989	2016-05-16		

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76		Attorney Docket Number	32471/M202647G
		Application Number	
Title of Invention	METHODS FOR MAKING L-GLUFOSINATE		
Prior Application Status	Expired	Remove	
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)
15/445254	Claims benefit of provisional	62/302421	2016-03-02
Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the Add button.			Add

Foreign Priority Information:

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55. When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX)ⁱ the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(i)(1) and (2). Under the PDX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

Application Number	Country ⁱ	Filing Date (YYYY-MM-DD)	Access Code ⁱ (if applicable)
Additional Foreign Priority Data may be generated within this form by selecting the Add button.			Add

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

This application (1) claims priority to or the benefit of an application filed before March 16, 2013 and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013.

☐ NOTE: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.

Application Data Sheet 37 CFR 1.76		Attorney Docket Number	32471/M202647G
		Application Number	
Title of Invention	METHODS FOR MAKING L-GLUFOSINATE		

Authorization or Opt-Out of Authorization to Permit Access:

When this Application Data Sheet is properly signed and filed with the application, applicant has provided written authority to permit a participating foreign intellectual property (IP) office access to the instant application-as-filed (see paragraph A in subsection 1 below) and the European Patent Office (EPO) access to any search results from the instant application (see paragraph B in subsection 1 below).

Should applicant choose not to provide an authorization identified in subsection 1 below, applicant **must opt-out** of the authorization by checking the corresponding box A or B or both in subsection 2 below.

NOTE: This section of the Application Data Sheet is **ONLY** reviewed and processed with the **INITIAL** filing of an application. After the initial filing of an application, an Application Data Sheet cannot be used to provide or rescind authorization for access by a foreign IP office(s). Instead, Form PTO/SB/39 or PTO/SB/69 must be used as appropriate.

1. Authorization to Permit Access by a Foreign Intellectual Property Office(s)

A. Priority Document Exchange (PDX) - Unless box A in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the State Intellectual Property Office of the People's Republic of China (SIPO), the World Intellectual Property Organization (WIPO), and any other foreign intellectual property office participating with the USPTO in a bilateral or multilateral priority document exchange agreement in which a foreign application claiming priority to the instant patent application is filed, access to: (1) the instant patent application-as-filed and its related bibliographic data, (2) any foreign or domestic application to which priority or benefit is claimed by the instant application and its related bibliographic data, and (3) the date of filing of this Authorization. See 37 CFR 1.14(h)(1).

B. Search Results from U.S. Application to EPO - Unless box B in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the EPO access to the bibliographic data and search results from the instant patent application when a European patent application claiming priority to the instant patent application is filed. See 37 CFR 1.14(h)(2).

The applicant is reminded that the EPO's Rule 141(1) EPC (European Patent Convention) requires applicants to submit a copy of search results from the instant application without delay in a European patent application that claims priority to the instant application.

2. Opt-Out of Authorizations to Permit Access by a Foreign Intellectual Property Office(s)

☐ A. Applicant **DOES NOT** authorize the USPTO to permit a participating foreign IP office access to the instant application-as-filed. If this box is checked, the USPTO will not be providing a participating foreign IP office with any documents and information identified in subsection 1A above.

☐ B. Applicant **DOES NOT** authorize the USPTO to transmit to the EPO any search results from the instant patent application. If this box is checked, the USPTO will not be providing the EPO with search results from the instant application.

NOTE: Once the application has published or is otherwise publicly available, the USPTO may provide access to the application in accordance with 37 CFR 1.14.

Application Data Sheet 37 CFR 1.76		Attorney Docket Number	32471/M202647G
		Application Number	
Title of Invention	METHODS FOR MAKING L-GLUFOSINATE		

Applicant Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.			
Applicant 1			<input type="button" value="Remove"/>
If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section.			
<input type="button" value="Clear"/>			
☒ Assignee	☐ Legal Representative under 35 U.S.C. 117	☐ Joint Inventor	
☐ Person to whom the inventor is obligated to assign.		☐ Person who shows sufficient proprietary interest	
If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:			
Name of the Deceased or Legally Incapacitated Inventor: <input type="text"/>			
If the Applicant is an Organization check here. ☒			
Organization Name	BASF SE		
Mailing Address Information For Applicant:			
Address 1	Carl-Bosch-Strasse 38		
Address 2			
City	Ludwigshafen	State/Province	
Country	DE	Postal Code	67056
Phone Number		Fax Number	
Email Address			
Additional Applicant Data may be generated within this form by selecting the Add button. <input type="button" value="Add"/>			

Assignee Information including Non-Applicant Assignee Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	32471/M202647G
		Application Number	
Title of Invention	METHODS FOR MAKING L-GLUFOSINATE		

Assignee 1

Complete this section if assignee information, including non-applicant assignee information, is desired to be included on the patent application publication. An assignee-applicant identified in the "Applicant Information" section will appear on the patent application publication as an applicant. For an assignee-applicant, complete this section only if identification as an assignee is also desired on the patent application publication.

[Remove](#)If the Assignee or Non-Applicant Assignee is an Organization check here. ☐

Prefix	Given Name	Middle Name	Family Name	Suffix

Mailing Address Information For Assignee including Non-Applicant Assignee:

Address 1			
Address 2			
City		State/Province	
Country ⁱ		Postal Code	
Phone Number		Fax Number	
Email Address			

Additional Assignee or Non-Applicant Assignee Data may be generated within this form by selecting the Add button.

[Add](#)**Signature:**[Remove](#)

NOTE: This Application Data Sheet must be signed in accordance with 37 CFR 1.33(b). **However, if this Application Data Sheet is submitted with the INITIAL filing of the application and either box A or B is not checked in subsection 2 of the "Authorization or Opt-Out of Authorization to Permit Access" section, then this form must also be signed in accordance with 37 CFR 1.14(c).**

This Application Data Sheet **must** be signed by a patent practitioner if one or more of the applicants is a **juristic entity** (e.g., corporation or association). If the applicant is two or more joint inventors, this form must be signed by a patent practitioner, **all** joint inventors who are the applicant, or one or more joint inventor-applicants who have been given power of attorney (e.g., see USPTO Form PTO/AIA/81) on behalf of **all** joint inventor-applicants.

See 37 CFR 1.4(d) for the manner of making signatures and certifications.

Signature	/Joshua T. Elliott/		Date (YYYY-MM-DD)	2024-02-07	
First Name	Joshua	Last Name	Elliott	Registration Number	43,603

Additional Signature may be generated within this form by selecting the Add button.

[Add](#)

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	32471/M202647G
		Application Number	
Title of Invention	METHODS FOR MAKING L-GLUFOSINATE		

This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.



UNITED STATES
PATENT AND TRADEMARK OFFICE

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Alexandria, VA 22313 - 1450
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ELECTRONIC PAYMENT RECEIPT

APPLICATION # 18/435,340	RECEIPT DATE / TIME 02/07/2024 03:06:02 PM Z ET	ATTORNEY DOCKET # 32471/M202647G
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Title of Invention

METHODS FOR MAKING L-GLUFOSINATE

Application Information

APPLICATION TYPE	Utility - Nonprovisional Application under 35 USC 111(a)	PATENT #	-
CONFIRMATION #	4214	FILED BY	Rebecca Walsh
PATENT CENTER #	64244744	AUTHORIZED BY	Joshua Elliott
CUSTOMER #	4743	FILING DATE	-
CORRESPONDENCE ADDRESS	-	FIRST NAMED INVENTOR	Brian Michael Green

Payment Information

PAYMENT METHOD CARD / 0428	PAYMENT TRANSACTION ID E202427F06568618	PAYMENT AUTHORIZED BY Rebecca Walsh
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FEE CODE	DESCRIPTION	ITEM PRICE(\$)	QUANTITY	ITEM TOTAL(\$)
1111	UTILITY PATENT APPL. SEARCH FEE	700.00	1	700.00
1011	BASIC FILING FEE - UTILITY (PAPER FILING ALSO REQUIRES NON-ELECTRONIC FILING FEE UNDER 1.16(T))	320.00	1	320.00
1311	PATENT APPL. EXAMINATION FEE	800.00	1	800.00
TOTAL AMOUNT:				\$1,820.00

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.



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ELECTRONIC ACKNOWLEDGEMENT RECEIPT

APPLICATION #
18/435,340

RECEIPT DATE / TIME
02/07/2024 03:06:02 PM Z ET

ATTORNEY DOCKET #
32471/M202647G

Title of Invention

METHODS FOR MAKING L-GLUFOSINATE

Application Information

APPLICATION TYPE Utility - Nonprovisional Application
under 35 USC 111(a)

PATENT # -

CONFIRMATION # 4214

FILED BY Rebecca Walsh

PATENT CENTER # 64244744

FILING DATE -

CUSTOMER # 4743

FIRST NAMED INVENTOR Brian Michael Green

CORRESPONDENCE ADDRESS -

AUTHORIZED BY Joshua Elliott

Documents

TOTAL DOCUMENTS: 9

DOCUMENT	PAGES	DESCRIPTION	SIZE (KB)
Application Data Sheet Fillable PDF.pdf	9	Application Data Sheet	2180 KB
Utility Patent Application Transmittal.pdf	1	Transmittal of New Application	31 KB
POA or Authorization of Agent.pdf	1	Power of Attorney	16 KB
Declaration.pdf	2	Oath or Declaration filed	418 KB
BASF SE POA.pdf	1	Power of Attorney	87 KB
M202647G_SeqListing.xml	-	Sequence Listing Supp (XML File)	5 KB
Application-APP.TEXT.docx	46	Application body structured text document	70 KB

Warning: Bookmarks were found and have been removed.

Drawing.pdf	2	Drawings-only black and white line drawings	139 KB
Application.pdf	46	Auxiliary PDF of Application	518 KB

Digest

DOCUMENT	MESSAGE DIGEST(SHA-512)
Application Data Sheet Fillable PDF.pdf	4DB4C4CBB5BE2B05182EFC7C80A465C8E72CA58AE94A23D32A8EA4F9B108769A9A6E38A965E8D23CB52F33FCD33FCC80C0D153490AE47F493150183234A9D7EB
Utility Patent Application Transmittal.pdf	D6E39136BDC36CC0B26B9EE04A861C42C6E9B29425EDA1D13076F63624DD8C233D0A854E2B70AEC58AB8DF1543DB3E96A3701E226171F3DFA944449305C3570E
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Drawing.pdf	B9A694303D714C6F758AD9AF94A30AB5B4F8464101249887EC5E603FF44F76D5DEC2DFB86CD88C8711A5F6925B2564996A8197DA61523F053AC1730100C44C32
Application.pdf	130A806E2517903D967CE8869E202E14C037E6E77B1E3C89E900A4331935B0A4427352CB8713171FE2E837FAFD49C093B2735D854CB2F81B03A486F828164520

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Under the Paperwork Reduction Act of 1995 no persons are required to respond to a collection of information unless it displays a valid OMB control number.

UTILITY PATENT APPLICATION TRANSMITTAL <small>(Only for new nonprovisional applications under 37 CFR 1.53(b))</small>		Attorney Docket No. 32471/M202647G	
		First Named Inventor Brian Michael Green	
		Title	METHODS FOR MAKING L-GLUFOSINATE
		Priority Mail Express® Label No.	
APPLICATION ELEMENTS <small>See MPEP chapter 600 concerning utility patent application contents.</small>		ADDRESS TO: Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450	
<div>1. ☐ Fee Transmittal Form <small>(PTO/SB/17 or equivalent)</small></div> <div>2. ☐ Applicant asserts small entity status. <small>See 37 CFR 1.27</small></div> <div>3. ☐ Applicant certifies micro entity status. See 37 CFR 1.29. <small>Applicant must attach form PTO/SB/15A or B or equivalent.</small></div> <div>4. ☒ Specification [Total Pages <u>46</u>] <small>Both the claims and abstract must start on a new page. (See MPEP § 608.01(a) for information on the preferred arrangement)</small></div> <div>5. ☒ Drawing(s) (35 U.S.C. 113) [Total Sheets <u>2</u>]</div> <div>6. ☐ Inventor's Oath or Declaration [Total Pages <u>2</u>] <small>(including substitute statements under 37 CFR 1.64 and assignments serving as an oath or declaration under 37 CFR 1.63(e))</small><div>a. ☐ Newly executed (original or copy)</div><div>b. ☒ A copy from a prior application (37 CFR 1.63(d))</div></div> <div>7. ☒ Application Data Sheet * See note below. <small>See 37 CFR 1.76 (PTO/AIA/14 or equivalent)</small></div> <div>8. ☐ CD-ROM or CD-R <small>in duplicate, large table, or Computer Program (Appendix)</small> ☐ Landscape Table on CD</div> <div>9. Nucleotide and/or Amino Acid Sequence Submission <small>(if applicable, items a. – c. are required)</small><div>a. ☒ Computer Readable Form (CRF)</div><div>b. ☐ Specification Sequence Listing on:<div>i. ☐ CD-ROM or CD-R (2 copies); or</div><div>ii. ☐ Paper</div></div><div>c. ☐ Statements verifying identity of above copies</div></div>		ACCOMPANYING APPLICATION PAPERS <div>10. ☐ Assignment Papers <small>(cover sheet & document(s))</small> Name of Assignee <div style="border: 1px solid black; height: 30px; width: 100%;"></div></div> <div>11. ☐ 37 CFR 3.73(c) Statement <small>(when there is an assignee)</small> ☒ Power of Attorney</div> <div>12. ☐ English Translation Document (if applicable)</div> <div>13. ☐ Information Disclosure Statement <small>(PTO/SB/08 or PTO-1449)</small> ☐ Copies of citations attached</div> <div>14. ☐ Preliminary Amendment</div> <div>15. ☐ Return Receipt Postcard <small>(MPEP § 503) (Should be specifically itemized)</small></div> <div>16. ☐ Certified Copy of Priority Document(s) <small>(if foreign priority is claimed)</small></div> <div>17. ☐ Nonpublication Request <small>Under 35 U.S.C. 122(b)(2)(B)(i). Applicant must attach form PTO/SB/35 or equivalent.</small></div> <div>18. ☐ Other: <div style="border: 1px solid black; height: 40px; width: 100%;"></div></div>	
*Note: (1) Benefit claims under 37 CFR 1.78 and foreign priority claims under 1.55 must be included in an Application Data Sheet (ADS). (2) For applications filed under 35 U.S.C. 111, the application must contain an ADS specifying the applicant if the applicant is an assignee, person to whom the inventor is under an obligation to assign, or person who otherwise shows sufficient proprietary interest in the matter. See 37 CFR 1.46(b).			
19. CORRESPONDENCE ADDRESS			
☒ The address associated with Customer Number: <u>04743</u> OR ☐ Correspondence address below			
Name			
Address			
City		State	Zip Code
Country		Telephone	Email
Signature	/Joshua T. Elliott/		Date
Name (Print/Type)	Joshua T. Elliott		Registration No. (Attorney/Agent)
			February 7, 2024
			43,603

Doc Code: PA..
Document Description: Power of Attorney

PTO/AIA/82A (07-13)
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NOTE: This form is to be submitted with the Power of Attorney by Applicant form (PTO/AIA/82B) to identify the application to which the Power of Attorney is directed, in accordance with 37 CFR 1.5, unless the application number and filing date are identified in the Power of Attorney by Applicant form. If neither form PTO/AIA/82A nor form PTO/AIA/82B identifies the application to which the Power of Attorney is directed, the Power of Attorney will not be recognized in the application.

Application Number	Not Yet Assigned		
Filing Date	Concurrently Herewith		
First Named Inventor	Brian Michael Green		
Title	METHODS FOR MAKING L-GLUFOSINATE		
Art Unit	Not Yet Assigned		
Examiner Name	Not Yet Assigned		
Attorney Docket Number	32471/M202647G		
SIGNATURE of Applicant or Patent Practitioner			
Signature	/Joshua T. Elliott/	Date (Optional)	
Name	Joshua T. Elliott	Registration Number	43,603
Title (if Applicant is a juristic entity)			
Applicant Name (if Applicant is a juristic entity)			
NOTE: This form must be signed in accordance with 37 CFR 1.33. See 37 CFR 1.4(d) for signature requirements and certifications. If more than one applicant, use multiple forms.			
☐	*Total of <u>1</u> forms are submitted.		

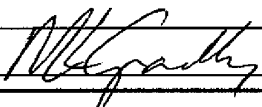
**DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN
APPLICATION DATA SHEET (37 CFR 1.76)**

Title of Invention	Methods for Making L-Glufosinate
<p>As the below named inventor, I hereby declare that:</p> <p>This declaration is directed to: ☐ The attached application, or ☒ United States application or PCT international application number <u>15/445,254</u> filed on <u>February 28, 2017</u></p> <p>The above-identified application was made or authorized to be made by me.</p> <p>I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.</p> <p>I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.</p> <p style="text-align: center;">WARNING:</p> <p>Petitioner/applicant is cautioned to avoid submitting personal information in documents filed in a patent application that may contribute to identity theft. Personal information such as social security numbers, bank account numbers, or credit card numbers (other than a check or credit card authorization form PTO-2036 submitted for payment purposes) is never required by the USPTO to support a petition or an application. If this type of personal information is included in documents submitted to the USPTO, petitioners/applicants should consider redacting such personal information from the documents before submitting them to the USPTO. Petitioner/applicant is advised that the record of a patent application is available to the public after publication of the application (unless a non-publication request in compliance with 37 CFR 1.213(a) is made in the application) or issuance of a patent. Furthermore, the record from an abandoned application may also be available to the public if the application is referenced in a published application or an issued patent (see 37 CFR 1.14). Checks and credit card authorization forms PTO-2036 submitted for payment purposes are not retained in the application file and therefore are not publicly available.</p> <p>LEGAL NAME OF INVENTOR</p> <p>Inventor: <u>Brian Michael Green</u> Date (Optional): <u>5/31/17</u></p> <p>Signature: <u>Brian Michael Green</u></p> <p>Note: An application data sheet (PTO/SB/14 or equivalent), including naming the entire inventive entity, must accompany this form or must have been previously filed. Use an additional PTO/AIA/01 form for each additional inventor.</p>	

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

**DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN
APPLICATION DATA SHEET (37 CFR 1.76)**

Title of Invention	Methods for Making L-Glufosinate
<p>As the below named inventor, I hereby declare that:</p> <p>This declaration is directed to: ☐ The attached application, or</p> <p style="margin-left: 150px;">☒ United States application or PCT international application number <u>15/445,254</u></p> <p style="margin-left: 150px;">filed on <u>February 28, 2017</u></p> <p>The above-identified application was made or authorized to be made by me.</p> <p>I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.</p> <p>I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.</p> <p style="text-align: center;">WARNING:</p> <p>Petitioner/applicant is cautioned to avoid submitting personal information in documents filed in a patent application that may contribute to identity theft. Personal information such as social security numbers, bank account numbers, or credit card numbers (other than a check or credit card authorization form PTO-2038 submitted for payment purposes) is never required by the USPTO to support a petition or an application. If this type of personal information is included in documents submitted to the USPTO, petitioners/applicants should consider redacting such personal information from the documents before submitting them to the USPTO. Petitioner/applicant is advised that the record of a patent application is available to the public after publication of the application (unless a non-publication request in compliance with 37 CFR 1.213(a) is made in the application) or issuance of a patent. Furthermore, the record from an abandoned application may also be available to the public if the application is referenced in a published application or an issued patent (see 37 CFR 1.14). Checks and credit card authorization forms PTO-2038 submitted for payment purposes are not retained in the application file and therefore are not publicly available.</p>	
<p>LEGAL NAME OF INVENTOR</p> <p>Inventor: _____ Date (Optional): <u>9/6/2017</u></p> <p>Signature: <u>Michelle Lorraine Gradley</u> </p>	
<p><small>Note: An application data sheet (PTO/SB/14 or equivalent), including naming the entire inventive entity, must accompany this form or must have been previously filed. Use an additional PTO/AIA/01 form for each additional inventor.</small></p>	

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Sequence Listing Report for Application No: 18435340

File Name: 18435340_1_1.xml (5025 B), **Created:** April 01, 2024, 04:12 PM

USPTO Receipt Date: February 07, 2024, 03:06 PM

Validation Date: April 01, 2024, 04:12 PM

Reviewed by: NKp6ZAWqxd5/YS+5kMWbeVihCo4/Q31f

DEFECTIVE: This validation report concerns the sequence listing filed 02-07-2024 15:06:02 and indicates the errors found by the USPTO during validation of the sequence listing. Any resolution of the errors identified in this validation report should not be construed as meaning that the sequence listing will comply with all of the requirements of 37 CFR §§ 1.821- 1.825, WIPO Standard ST.25, 37 CFR §§ 1.831-1.835 and 1.839, or WIPO Standard ST.26.

For any sequence listing related questions or concerns, the Applicant is invited to contact the USPTO Sequence Help Desk at 571-272-2510 or SequenceHelpDesk@USPTO.GOV

Reviewer's Comments

The "organism" qualifier value must be one of the following options:

1. A full Latin "Genus species" name or "Genus sp." (see WIPO ST.26 paragraphs 78-80), wherein the Genus name is capitalized and the species name is lower-case;
2. A virus name (see WIPO ST.26 paragraph 82);
3. "unidentified", for naturally occurring sequences where the Latin Genus and/or species is unknown. Any other taxonomic information should be contained in a "note" qualifier (see WIPO ST.26 paragraph 81);
4. "synthetic construct" for sequences that are not naturally occurring (see WIPO ST.26 paragraph 83).

SEQ ID NO: 1 contains the extra information "sp." in the organism name. "Sinorhizobium arboris" is already a "Genus species" name. Please remove "sp." from the organism name.

1. W261059 DataElement:PROPERTY_NAMES.QUALIFIER_VALUE
DetectedValue:Sinorhizobium arboris sp.
MessageKey:X_QUALIFIER_VALUE_UNRECOGNISED_ORGANISM
LocalizedMessage:This organism name has not been found in the list of predefined or custom organism names. Verify this name is a taxonomically acceptable organism name.
Sequence ID: 1

The messages shown below do not indicate non-compliance with the sequence rules, and do not require correction:

2. W261178 DataElement:PROPERTY_NAMES.ORIGINAL_FREE_TEXT_LANGUAGE_CODE
MessageKey:ORIGINAL_FREE_TEXT_LANGUAGE_CODE_MISSING
LocalizedMessage:An original free text language code has not been entered.

Validated By CRFValidator v 2.3.0

Application No: 18435340

Version No: 1.1

CRF Copy Request from application :

Version :

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Output Set: /slic/CRF_WORKFLOW/18/435/340/1.1/18435340_1_1.xml.pdf

Started: 2024-04-01 16:12:15.000

Finished: 2024-04-01 16:12:22.955

Elapsed: 0 hr(s) 0 min(s) 7 sec(s) 955 ms

Total Warnings: 2

Total Errors: 0

No. of SeqIDs Defined: 3

Actual SeqID Count: 3

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W 261178	DataElement:PROPERTY_NAMES.ORIGINAL_FREE_TEXT_LANGUAGE_CODE;MessageKey:ORIGINAL_FREE_TEXT_LANGUAGE_CODE_MISSING;LocalizedMessage:An original free text language code has not been entered.;

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UNITED STATES DEPARTMENT OF COMMERCE
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APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	TOT CLAIMS	IND CLAIMS
18/435,340	02/07/2024		1820	32471/M202647G	6	1

CONFIRMATION NO. 4214

FILING RECEIPT



00000000/0551192

4743

MARSHALL, GERSTEIN & BORUN LLP
233 SOUTH WACKER DRIVE
6300 WILLIS TOWER
CHICAGO, IL 60606-6357

Date Mailed: 04/03/2024

Receipt is acknowledged of this non-provisional utility patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF FIRST INVENTOR, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection.

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Inventor(s)

Brian Michael Green, Lutherville, MD;
Michelle Lorraine Gradley, Canterbury, UNITED KINGDOM;

Applicant(s)

BASF SE, Ludwigshafen, GERMANY;

Power of Attorney: The patent practitioners associated with Customer Number 04743

Domestic Priority data as claimed by applicant

This application is a CON of 18/179,136 03/06/2023 PAT 11,913,048
which is a CON of 17/530,018 11/18/2021 PAT 11,905,538
which is a CON of 16/997,133 08/19/2020 PAT 11,560,577
which is a CON of 16/287,290 02/27/2019 PAT 10,781,465
which is a CON of 15/787,448 10/18/2017 PAT 10,260,078
which is a DIV of 15/445,254 02/28/2017 PAT 9,834,802
which claims benefit of 62/413,240 10/26/2016 ABN
and claims benefit of 62/336,989 05/16/2016 ABN
and claims benefit of 62/302,421 03/02/2016 ABN

Foreign Applications for which priority is claimed (You may be eligible to benefit from the Patent Prosecution Highway program at the USPTO. Please see <http://www.uspto.gov> for more information.) - None.
Foreign application information must be provided in an Application Data Sheet in order to constitute a claim to foreign priority. See 37 CFR 1.55 and 1.76.

Permission to Access Application via Priority Document Exchange: Yes

Permission to Access Search Results: Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

If Required, Foreign Filing License Granted: 02/21/2024

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 18/435,340**

Projected Publication Date: To Be Determined - pending completion of Sequence Disclosures.

Non-Publication Request: No

Early Publication Request: No

Title

METHODS FOR MAKING L-GLUFOSINATE

Preliminary Class

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications: No

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Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at <http://www.uspto.gov/web/offices/pac/doc/general/index.html>.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, <http://www.stopfakes.gov>. Part of a Department of Commerce initiative,

page 2 of 4

this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

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Title 35, United States Code, Section 184

Title 37, Code of Federal Regulations, 5.11 & 5.15

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PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875						Application or Docket Number 18/435,340				
APPLICATION AS FILED - PART I										
		(Column 1)	(Column 2)		SMALL ENTITY		OR	OTHER THAN SMALL ENTITY		
FOR		NUMBER FILED		NUMBER EXTRA	RATE(\$)	FEE(\$)		RATE(\$)	FEE(\$)	
BASIC FEE (37 CFR 1.16(a), (b), or (c))		N/A		N/A	N/A			N/A	320	
SEARCH FEE (37 CFR 1.16(k), (l), or (m))		N/A		N/A	N/A			N/A	700	
EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))		N/A		N/A	N/A			N/A	800	
TOTAL CLAIMS (37 CFR 1.16(i))		6	minus 20 =	*			OR	x	= 0	
INDEPENDENT CLAIMS (37 CFR 1.16(h))		1	minus 3 =	*				x	= 0	
APPLICATION SIZE FEE (37 CFR 1.16(s))	If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$310 (\$155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).								0	
MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))									0	
					TOTAL			TOTAL	1820	
* If the difference in column 1 is less than zero, enter "0" in column 2.										
APPLICATION AS AMENDED - PART II										
		(Column 1)		(Column 2)	(Column 3)	SMALL ENTITY		OR	OTHER THAN SMALL ENTITY	
AMENDMENT A		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)		RATE(\$)	ADDITIONAL FEE(\$)
	Total (37 CFR 1.16(i))	*	Minus	**	=	x	=	OR	x	=
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x	=	OR	x	=
	Application Size Fee (37 CFR 1.16(s))							OR		
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	
		(Column 1)		(Column 2)	(Column 3)	SMALL ENTITY		OR	OTHER THAN SMALL ENTITY	
AMENDMENT B		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)		RATE(\$)	ADDITIONAL FEE(\$)
	Total (37 CFR 1.16(i))	*	Minus	**	=	x	=	OR	x	=
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x	=	OR	x	=
	Application Size Fee (37 CFR 1.16(s))							OR		
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	
* If the entry in column 1 is less than the entry in column 2, write "0" in column 3. ** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20". *** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3". The "Highest Number Previously Paid For" (Total or Independent) is the highest found in the appropriate box in column 1.										



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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
18/435,340	02/07/2024	Brian Michael Green	32471/M202647G

CONFIRMATION NO. 4214

4743

MARSHALL, GERSTEIN & BORUN LLP
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CHICAGO, IL 60606-6357

FORMALITIES LETTER



0000000070551194

Date Mailed: 04/03/2024

**NOTICE TO COMPLY WITH REQUIREMENTS FOR PATENT APPLICATIONS
CONTAINING NUCLEOTIDE AND/OR AMINO ACID SEQUENCE DISCLOSURES**

Filing Date Granted

Applicant is given **TWO MONTHS FROM THE DATE OF THIS NOTICE** within which to comply with the sequence rules, 37 CFR §§ 1.831-1.835. Failure to comply with these requirements will result in ABANDONMENT of the application under 37 CFR §1.835(d)(1). Extension of time may be obtained by filing a petition accompanied by the extension fee under the provisions of 37 CFR §1.136.

**Summary of Requirements for Patent Applications Filed
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37 CFR 1.831(a) requires that patent applications which contain disclosures of nucleotide and/or amino acid sequences that fall within the definitions of 37 CFR 1.831(b) must contain a "Sequence Listing XML", as a *separate part of the disclosure*, which presents the nucleotide and/or amino acid sequences and associated information using the symbols and format in accordance with the requirements of 37 CFR 1.831-1.835. This "Sequence Listing XML" part of the disclosure may be submitted:

1. In accordance with 37 CFR 1.831(a) using the symbols and format requirements of 37 CFR 1.832 through 1.834 via the USPTO's patent electronic filing system (currently Sequence Listing XML files may **only** be properly filed electronically using **Patent Center**) (see Section I.1 of the Legal Framework for Patent Electronic System (<https://www.uspto.gov/patents-application-process/filing-online/legal-framework-efs-web>), hereinafter "Legal Framework") in XML format, together with an incorporation by reference statement of the material in the XML file in a separate paragraph of the specification as required by 37 CFR 1.835(a)(2) or 1.835(b)(2) identifying:
 - a. the name of the XML file
 - b. the date of creation; and
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2. In accordance with 37 CFR 1.831(a) using the symbols and format requirements of 37 CFR 1.832 through 1.834 on read-only optical disc(s) as permitted by 37 CFR 1.52(e)(1)(ii), labeled according to 37 CFR 1.52(e)(5), with an incorporation-by-reference statement of the material in the XML format according to 37 CFR 1.52(e)(8) and 37 CFR 1.835(a)(2) or 1.835(b)(2) in a separate paragraph of the specification identifying:
 - a. the name of the XML file;
 - b. the date of creation; and
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SPECIFIC DEFICIENCIES AND THE REQUIRED RESPONSE TO THIS NOTICE ARE AS FOLLOWS:

- The "Sequence Listing XML" part of the disclosure filed with this application in accordance with 37 CFR 1.831-1.834 has been found to contain an error or errors as indicated on the PAIR document "Computer Readable Form (CRF) for Sequence Listing - Defective" dated 04/01/2024. Applicant must provide:
 - A replacement "Sequence Listing XML" part of the disclosure, as described above submitted in accordance with either item 1. or 2.; together with
 - A statement that identifies the location of all additions, deletions, or replacements of sequence information in the replacement "Sequence Listing XML" as required by 1.835(b)(3);
 - A statement that indicates support for the amendment in the application, as filed, as required by 37 CFR 1.835(b)(4);
 - A statement that the replacement "Sequence Listing XML" includes no new matter as required by 37 CFR 1.835(b)(5); and
 - A substitute specification in compliance with 37 CFR 1.52, 1.121(b)(3), and 1.125 inserting the required incorporation-by-reference paragraph as required by 37 CFR 1.835(b)(2), consisting of:
 - A copy of the previously-submitted specification, with deletions shown with strikethrough or brackets and insertions shown with underlining (marked-up version);
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/stzewdie/



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A handwritten signature in black ink, reading "Kathi Vidal".

Kathi Vidal

Under Secretary of Commerce for Intellectual Property and
Director of the United States Patent and Trademark Office

A handwritten signature in black ink, reading "Derrick Brent".

Derrick Brent

Deputy Under Secretary of Commerce for Intellectual Property and
Deputy Director of the United States Patent and Trademark Office

Docket No.: 32471/M202647G
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Brian Michael Green et al.

Application No.: 18/435,340

Confirmation No.: 4214

Filed: February 7, 2024

Art Unit: Not Yet Assigned

For: METHODS FOR MAKING L-GLUFOSINATE

Examiner: Not Yet Assigned

INFORMATION DISCLOSURE STATEMENT (IDS)

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Commissioner:

Pursuant to 37 C.F.R. §§ 1.56, 1.97, and 1.98, the attention of the United States Patent and Trademark Office is hereby directed to the references listed on the attached PTO/SB/08. It is respectfully requested that the information be expressly considered during the prosecution of the above-identified application, and that the references be made of record therein and appear among the "References Cited" on any patent to issue therefrom.

This Information Disclosure Statement is filed within three months of the filing date of a national application other than a continued prosecution application under 37 C.F.R. § 1.53(d) (37 C.F.R. § 1.97(b)(1)).

In accordance with 37 C.F.R. § 1.98(a)(2)(ii), copies of U.S. patents and U.S. patent application publications are not submitted. Submitted herewith are copies of foreign patents and non-patent literature in accordance with 37 C.F.R. § 1.98(a)(2).

In accordance with 37 C.F.R. § 1.98(d)(1) and as noted by a double-asterisk next to the Cite No. on the attached PTO/SB/08, the following references are not supplied because they were previously cited by or submitted to the Office in prior application numbers 18/179,136 filed March 6, 2023, 17/530,018 filed November 18, 2021, 16/997,133 filed August 19, 2020, 16/287,290 filed February 27, 2019, 15/787,448 filed October 18, 2017,

and 15/445,254 filed February 28, 2017 and relied on in the above-identified application for an earlier effective filing date under 35 U.S.C. § 120.

In accordance with 37 C.F.R. § 1.97(g), the filing of this Information Disclosure Statement shall not be construed as a representation that a search has been made. In accordance with 37 C.F.R. § 1.97(h), the filing of this Information Disclosure Statement shall not be construed to be an admission that the information cited in this Information Disclosure Statement is, or is considered to be, material to patentability as defined in 37 C.F.R. § 1.56(b).

It is submitted that the Information Disclosure Statement is in compliance with 37 C.F.R. § 1.98, and the Examiner is respectfully requested to consider the listed references.

Applicant believes no fee is due with this response. However, if a fee is due, please charge our Deposit Account No. 13-2855, under Order No. 32471/M202647G from which the undersigned is authorized to draw.

Dated: May 6, 2024

Respectfully submitted,

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Espacenet

Bibliographic data: CN102174632 (A) — 2011-09-07

Bio-catalytic deracemization preparation method of non-natural L-amino acid

Inventor(s): SHIWEN XIA; GUOLAN FANG; HONGMEI XU; CHAO GE; LIJUAN GUO; YANG LI; ZHIHUI YANG ± (XIA SHIWEN, ; FANG GUOLAN, ; XU HONGMEI, ; GE CHAO, ; GUO LIJUAN, ; LI YANG, ; YANG ZHIHUI)

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Classification: - **international:** C12P13/04; C12P41/00
- **cooperative:**

Application number: CN200910103883 20090519

Priority number(s): CN200910103883 20090519

Abstract of CN102174632 (A)

The invention provides a bio-catalytic deracemization preparation method of non-natural L-amino acid. In the method, immobilized D-amino acid oxidase with high enantioselectivity is used as a biocatalyst, D-enantiomer oxide in DL-amino acid catalytically and chemically synthesized in the presence of molecular oxygen is used as imino acid, and the L-amino acid is reserved. Pd-C is used as a catalyst, HCOONH₄ is used as a hydrogen donor, and the formed imino acid is converted into DL-amino acid in situ by chemical catalytic hydrogen transfer reaction, so that continuous conversion from the DL-amino acid to the L-amino acid is realized. The hydrogen peroxide generated in the reaction process is efficiently decomposed into water and molecular oxygen in situ by using catalase. The method is used for preparing chiral medicament intermediates such as L-2-aminobutyric acid, L-2-aminovaleric acid, L-2-aminoadipic acid, L-tert-leucine, L-cyclohexyl glycine, L-o-chlorobenzene glycine, L-p-fluorobenzene glycine, L-2-amino-4-phenyl butyric acid, L-piperidine-2-carboxylic acid and L-piperazine-2-carboxylic acid; the yield is 75 to 95 percent; and the ee is more than 99 percent.



(12) 发明专利申请

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限公司 50102

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(51) Int. Cl.

C12P 41/00(2006.01)

权利要求书 1 页 说明书 5 页

(54) 发明名称

一种非天然 L-氨基酸的生物催化去消旋化制备方法

(57) 摘要

本发明提供一种非天然 L-氨基酸的生物催化去消旋化制备方法。该方法以具有高对映选择性的固定化 D-氨基酸氧化酶为生物催化剂,在分子氧存在下催化化学合成的 DL-氨基酸中的 D-对映体氧化为亚氨基酸, L-氨基酸保留。以 Pd-C 为催化剂, HCOONH₄ 为氢供体通过化学催化氢转移反应将形成的亚氨基酸原位转化为 DL-氨基酸, 实现 DL-氨基酸向 L-氨基酸的连续转化。反应过程中产生的过氧化氢利用过氧化氢酶原位高效分解为水和分子氧。方法用于制备手性药物中间体 L-2-氨基丁酸、L-2-氨基戊酸、L-2-氨基己二酸、L-叔亮氨酸、L-环己基甘氨酸、L-邻氯苯甘氨酸、L-对氯苯甘氨酸、L-2-氨基-4-苯基丁酸、L-哌啶-2-羧酸、L-哌嗪-2-羧酸, 收率 75-95%, ee > 99%。

1. 一种非天然 L-氨基酸的生物催化去消旋化制备方法,其特征是非天然 DL-氨基酸中的 D-对映体以 D-氨基酸氧化酶为生物催化剂,在氧源存在下对映选择性氧化为亚氨基酸,L-氨基酸保留;亚氨基酸采用金属催化剂、在氢源存在下原位转化为 DL-氨基酸,反应过程中产生的过氧化氢利用氧化剂原位分解为水和分子氧;

方法步骤如下:

1) 选择固定化 D-氨基酸氧化酶为生物催化剂将化学合成的非天然 DL-氨基酸中的 D-对映体氧化为亚氨基酸,L-氨基酸保留;

2) 选择高活性的过氧化氢酶将上述反应过程中形成的过氧化氢原位分解为水和分子氧;

3) 选择 Pd-C 为化学氢转移催化剂将反应中形成的亚氨基酸原位转化为非天然 DL-氨基酸,用于非天然 DL-氨基酸向非天然 L-氨基酸的连续转化;

4) 反应体系构建:在 50ml 甲酸铵溶液 (1mol/L, pH7.2) 中加入 1-4g DL-氨基酸,1-4g 固定化 D-氨基酸氧化酶 (58U/g),1-2ml 过氧化氢酶 (50000U/ml),1g Pd-C(10%);

5) 反应条件:反应温度 30℃, pH7.2, 磁力搅拌,匀速通入氧气,反应时间 16-30 小时;

6) 反应液处理:反应完成后减压浓缩,调等电点,冷冻析出非天然 L-氨基酸,红外干燥。

2. 根据权利要求 1 所述的方法,其特征是所述非天然 L-氨基酸选自 L-脂肪族氨基酸中的 L-2-氨基丁酸、L-2-氨基戊酸、L-2-氨基己二酸、L-叔亮氨酸;或选自 L-脂环族氨基酸中的 L-环己基甘氨酸、L-哌啶-2-羧酸或 L-哌嗪-2-羧酸;或选自 L-芳香族氨基酸中的 L-邻氯苯甘氨酸、L-对氟苯甘氨酸、L-2-氨基-4-苯基丁酸。

3. 根据权利要求 1 所述的方法,其特征是底物非天然 DL-氨基酸的使用浓度为 0.1-0.5mol/L。

4. 根据权利要求 1 所述的方法,其特征是 D-氨基酸氧化酶为固定化酶。固定化 D-氨基酸氧化酶与 DL-氨基酸的质量比为 0.5-1.0。

5. 根据权利要求 1 所述的方法,其特征是氧化 DL-氨基酸中 D-对映体为亚氨基酸的氧源为空气氧或纯氧。

6. 根据权利要求 1 所述的方法,金属催化剂为 Pd-C、钨黑、钨 / 氧化铝;Pd-C 催化剂与固定化 D-氨基酸氧化酶的质量比为 0.3-1.0。

7. 根据权利要求 1 所述的方法,氢源为氢气或甲酸铵;甲酸铵既作为生物催化氧化反应的缓冲盐,也作为化学催化氢转移反应的氢供体;甲酸铵浓度为 1-3mol/L,最好为 1mol/L。缓冲液 pH 为 5-8,最好为 7.2。

8. 根据权利要求 1 所述的方法,分解反应过程中产生的过氧化氢的氧化剂为 Fe^{3+} 、过氧化氢酶,最好为过氧化氢酶。过氧化氢酶与固定化 D-氨基酸氧化酶的活性比为 300-1000。

9. 根据权利要求 1 所述的方法,反应温度为 10-40℃,最好为 30℃。

一种非天然 L-氨基酸的生物催化去消旋化制备方法

技术领域

[0001] 本发明涉及非天然 L-氨基酸的生物催化去消旋化制备方法,属于生物催化不对称氧化制备手性医药中间体技术领域。

背景技术

[0002] 非天然 L-氨基酸是自然界中不存在的非蛋白源氨基酸。非天然 L-氨基酸及其衍生物如保护非天然 L-氨基酸、非天然 L-氨基醇等是手性药物的关键中间体,在医药领域有广泛应用。例如,L-2-氨基丁酸是抗癫痫药物左乙拉西坦中间体,L-2-氨基戊酸是抗高血压药物培哚普利中间体,L-2-氨基己二酸是谷氨酰胺合成酶抑制剂中间体,L-叔亮氨酸是抗病毒、抗肿瘤和抗炎药物中间体,L-环己基甘氨酸是环氧合酶(COX-2)抑制剂 JTE-522 中间体,L-邻氯苯甘氨酸是抗血栓药物氯吡格雷中间体,L-对氯苯甘氨酸是抗癌药物中间体,L-2-氨基-4-苯基丁酸是普利类抗高血压药物中间体,L-哌啶-2-羧酸是局麻药罗哌卡因、抗精神病药物疏利哒嗪中间体,L-哌啶-2-羧酸是 HIV 蛋白酶抑制剂中间体。

[0003] 非天然 L-氨基酸不能像天然 L-氨基酸一样采用发酵法生产。尽管非天然 L-氨基酸可以采用化学法制备,但化学不对称合成采用价格昂贵的手性源、手性助剂或手性催化剂,化学拆分工艺路线复杂、环境污染大。无论是化学不对称合成或是化学拆分,都存在收率低、光学纯度不高、生产成本高的缺点。生物催化法具有立体选择性高、反应条件温和、环境友好等特征,是制备对映纯非天然 L-氨基酸极为有用的方法。目前大量具有商业价值的非天然 L-氨基酸都是用生物催化法制备的(Breuer M,Ditrich K,Habicher T,Hauer B. Angew Chem Int Ed,2004,43,788)。生物催化法包括酶拆分法和酶催化不对称合成法。外消旋体的酶催化动力学拆分是制备非天然 L-氨基酸的一个最成功的策略,但涉及消旋氨基酸的衍生以及游离氨基酸和衍生氨基酸的分离,工艺复杂,非天然 L-氨基酸的理论收率仅为 50%。因此,为了避免 D-对映体的浪费,有必要开发收率>50%的非天然 L-氨基酸酶催化或化学酶催化合成方法。这可以通过三个途径实现:前手性底物的酶催化不对称合成,酶催化动态动力学拆分,酶催化去消旋化。酶催化去消旋化可以采用两种策略:采用具有互补对映选择性的双酶或多酶系统如氨基酸脱氢酶/转氨酶;对映选择性酶催化氧化反应和非选择性、非酶还原反应组合。

[0004] D-氨基酸氧化酶在分子氧存在下,立体选择性催化 D-氨基酸氧化脱氢为相应的亚氨基酸,亚氨基酸经历非酶水解为酮酸。D-氨基酸氧化酶已成功用于从头孢菌素 C 工业合成 7-氨基头孢烷酸(7-ACA,半合成抗生素的关键母核)、去除 L-氨基酸中的少量 D-对映体和从 DL-氨基酸制备非天然 L-氨基酸。为了获得收率>50%的非天然 L-氨基酸,对 D-氨基酸氧化酶催化 DL-氨基酸氧化和化学还原剂组合体系进行了广泛的研究。组合 D-氨基酸氧化酶和 NaBH_4 用于 DL-哌啶-2-羧酸的去消旋化,L-对映体的产率和光学纯度均>98%(Soda K,Oikawa T,Yokoigawa K. J Mol Catal B:Enzymatic 2001,11,149.)。以 NaCNBH_3 替代 NaBH_4 为还原剂和猪肾 D-氨基酸氧化酶为生物催化剂,实现了哌啶-2-羧酸的去消旋化,收率和 ee 分别为 86%和>99%。以 NaBH_4 作还原剂,DL-苯甘氨酸、2-氨基

丁酸、环戊基甘氨酸等非环和环状氨基酸去消旋化产生的非天然 L-氨基酸收率 75-90%， $ee > 99\%$ (Beard TM, Turner NJ. Chem Commun, 2002, 246.)。在这些方法中采用 NaBH_4 或 NaCNBH_3 与 D-氨基酸氧化酶组合将 DL-氨基酸去消旋化转化为非天然 L-氨基酸有 3 个缺点：1) NaBH_4 对水敏感，在 D-氨基酸氧化酶表现最大活性和稳定性的 pH (酸性或中性) 条件下易分解。为使还原有效，必须多次缓慢加入且当量很大 (500 个当量以上)。虽然 NaCNBH_3 比 NaBH_4 的水稳定性好，但反应性较低，从而导致产生的非天然 L-氨基酸收率低；2) 大量 NaBH_4 的加入致使操作 pH 接近 10。在如此高 pH 下，D-氨基酸氧化酶容易通过不可逆变性快速失活，增加制备成本，难于规模化；3) 必须采用价格高昂的 D-氨基酸氧化酶作生物催化剂。因此有必要提出基于 D-氨基酸氧化酶的适合非天然 L-氨基酸规模化制备的生物催化去消旋化方法。

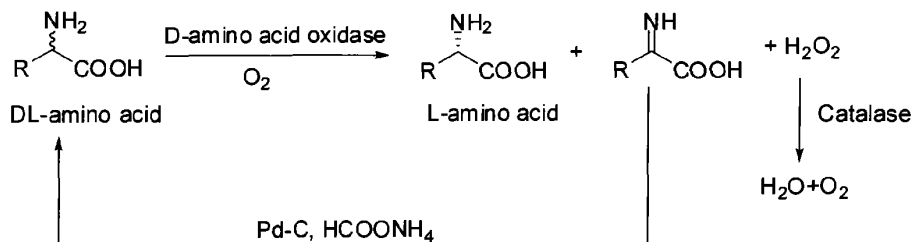
[0005] 以 D-氨基酸氧化酶为生物催化剂，组合化学催化还原制备非天然 L-氨基酸的方法未见报道。

发明内容

[0006] 本发明的目的是提供一种新的非天然 L-氨基酸生物催化去消旋化制备方法，利用固定化 D-氨基酸氧化酶高度的对映选择性，组合化学催化氢转移反应，以高的光学活性和高的收率制备非天然 L-氨基酸。

[0007] 方法的技术路线如下：

[0008]



[0009] 方法步骤如下：

[0010] 1) 选择固定化 D-氨基酸氧化酶为生物催化剂将化学合成的非天然 DL-氨基酸中的 D-对映体氧化为亚氨基酸，L-氨基酸保留；

[0011] 2) 选择高活性的过氧化氢酶将上述反应过程中形成的过氧化氢原位分解为水和分子氧；

[0012] 3) 选择 Pd-C 为化学氢转移催化剂将反应中形成的亚氨基酸原位转化为非天然 DL-氨基酸，用于非天然 DL-氨基酸向非天然 L-氨基酸的连续转化；

[0013] 4) 反应体系构建：在 50ml 甲酸铵溶液 (1mol/L, pH7.2) 中加入 1-4g DL-氨基酸，1-4g 固定化 D-氨基酸氧化酶 (58U/g)，1-2ml 过氧化氢酶 (50000U/ml)，1g Pd-C (10%)。

[0014] 5) 反应条件：反应温度 30℃，pH7.2，磁力搅拌，匀速通入氧气，反应时间 16-30 小时。

[0015] 6) 反应液处理：反应完成后减压浓缩，调等电点，冷冻析出非天然 L-氨基酸，红外干燥。

[0016] 分析方法

[0017] 生物催化去消旋化反应过程监测采用旋光法。间隔一定时间测定反应液旋光度,旋光度不再增加时反应完成。产物非天然 L-氨基酸的光学纯度以对映体过量值 (ee) 表示,采用手性液相色谱法测定。手性液相柱: Crownpak CR(+), 4.0×150mm, 5 μm, 流动相: pH1.5-2.0 HClO₄ 溶液, 流速: 0.4-1.0 ml/min, 检测: UV200nm。对映体色谱峰定位采用标准品对照。

[0018] 本发明的有益效果

[0019] 与传统的非天然 L-氨基酸化学和酶法制备技术相比,本发明提出的生物催化方法具有以下优点: 1) 工艺简单。与化学拆分和生物拆分不同, DL-氨基酸不经衍生, 直接作为酶催化反应底物。双酶反应和化学催化氢转移反应在同一反应体系中进行, 中间产物不经分离, 直接原位转化为 DL-氨基酸, 用于连续转化。终端产物仅为非天然 L-氨基酸, 无其他副产物, 分离过程简单。2) 方法的通用性好, 不仅适用于非天然 L-脂肪氨基酸的制备, 也适用于非天然 L-芳香氨基酸制备; 3) 反应条件温和, 环境友好。酶催化反应和化学催化反应在常温下进行, 不需要高温高压, 节约能源。反应体系以缓冲液为反应介质, 避免使用对环境有害的有机溶剂, 产生的废水只需经过简单处理即可排放; 4) 成本低廉。固定化 D-氨基酸氧化酶和 Pd-C 催化剂均为多相催化剂, 可回收重复使用多次, 有效降低了过程成本。

具体实施方式

[0020] 以下是以固定化 D-氨基酸氧化酶、过氧化氢酶为生物催化剂, Pd-C 为化学催化氢转移催化剂, 在水相体系中催化 DL-氨基酸对映选择性去消旋化制备非天然 L-氨基酸的实施例, 但本发明并不限于所列出的几个实例。

[0021] 实施例 1: L-2-氨基丁酸

[0022] 2.06g (0.02mol) DL-2-氨基丁酸溶于 50ml 甲酸铵 (1.0mol/L, 用氨水调节 pH 至 7.2) 溶液中, 加入 2.06g 固定化 D-氨基酸氧化酶 (58U/g)、1ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30℃ 下搅拌反应 16h, 反应液旋光度不再增加。过滤除去固定化 D-氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出白色晶体, 烘干得 1.8g L-2-氨基丁酸, 收率 87%, ee > 99%。

[0023] 实施例 2: L-2-氨基戊酸

[0024] 2.90g (0.025mol) DL-2-氨基戊酸溶于 50ml 甲酸铵 (1.0mol/L, 用氨水调节 pH 至 7.2) 溶液中, 加入 2.90g 固定化 D-氨基酸氧化酶 (58U/g)、1.5ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30℃ 下搅拌反应 16h, 反应液旋光度不再增加。过滤除去固定化 D-氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出白色晶体, 烘干得 2.6g L-2-氨基戊酸, 收率 90%, ee > 99%。

[0025] 实施例 3: L-2-氨基己二酸

[0026] 3.22g (0.02mol) DL-2-氨基己二酸溶于 50ml 甲酸铵 (1.0mol/L, 用氨水调节 pH 至 7.2) 溶液中, 加入 3.22g 固定化 D-氨基酸氧化酶 (58U/g)、1ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30℃ 下搅拌反应 20h, 反应液旋光度不再增加。过滤除去固定化 D-氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出白色晶体, 烘干得 2.8g L-2-氨基己二酸, 收率 87%, ee > 99%。

[0027] 实施例 4: L-叔亮氨酸

[0028] 1.31g (0.01mol) DL-叔亮氨酸溶于 50ml 甲酸铵 (1.0mol/L, 用氨水调节 pH 至 7.2) 溶液中, 加入 1.31g 固定化 D-氨基酸氧化酶 (58U/g)、1ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30°C 下反应 24h, 反应液旋光度不再增加。过滤除去固定化 D-氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出白色晶体, 烘干得 0.98g L-叔亮氨酸, 收率 75%, ee 99%。

[0029] 实施例 5: L-环己基甘氨酸

[0030] 1.57g (0.01mol) DL-环己基甘氨酸溶于 50ml 甲酸铵 (1.0mol/L, 用氨水调节 pH 至 7.2) 溶液中, 加入 1.57g 固定化 D-氨基酸氧化酶 (58U/g)、1ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30°C 下反应 24h, 反应液旋光度不再增加。过滤除去固定化 D-氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出白色晶体, 烘干得 1.4g L-环己基甘氨酸, 收率 89%, ee > 99%。

[0031] 实施例 6: L-邻氯苯甘氨酸

[0032] 1.85g (0.01mol) DL-邻氯苯甘氨酸溶于 50ml 甲酸铵 (1.0mol/L, 用氨水调节 pH 至 7.2) 溶液中, 加入 1.85g 固定化 D-氨基酸氧化酶 (58U/g)、1ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30°C 下反应 24h, 反应液旋光度不再增加。过滤除去固定化 D-氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出白色晶体, 烘干得 1.5g L-邻氯苯甘氨酸, 收率 81%, ee > 99%。

[0033] 实施例 7: L-对氟苯甘氨酸

[0034] 1.69g (0.01mol) DL-对氟苯甘氨酸溶于 50ml 甲酸铵 (1.0mol/L, 用氨水调节 pH 至 7.2) 溶液中, 加入 1.69g 固定化 D-氨基酸氧化酶 (58U/g)、1ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30°C 下反应 24h, 反应液旋光度不再增加。过滤除去固定化 D-氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出白色晶体, 烘干得 1.6g L-对氟苯甘氨酸, 收率 95%, ee > 99%。

[0035] 实施例 8: L-2-氨基-4-苯基丁酸

[0036] 1.79g (0.01mol) DL-2-氨基-4-苯基丁酸溶于 50ml 甲酸铵 (1.0mol/L, 用氨水调节 pH 至 7.2) 溶液中, 加入 1.79g 固定化 D-氨基酸氧化酶 (58U/g)、1ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30°C 下反应 24h, 反应液旋光度不再增加。过滤除去固定化 D-氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出白色晶体, 烘干得 1.6g L-2-氨基-4-苯基丁酸, 收率 89%, ee > 99%。

[0037] 实施例 9: L-哌啶-2-羧酸

[0038] 3.23g (0.025mol) DL-哌啶-2-羧酸溶于 50ml 甲酸铵溶液 (1.0mol/L, 用氨水调节 pH 至 7.2), 加入 1.62g 固定化 D-氨基酸氧化酶 (58U/g)、1.6ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30°C 下反应 24h, 反应液旋光度不再增加。过滤除去固定化 D-氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出白色晶体, 烘干得 2.6g L-哌啶-2-羧酸, 收率 80%, ee > 99%。

[0039] 实施例 10: L-哌嗪-2-羧酸

[0040] 3.25g (0.025mol) DL-哌嗪-2-羧酸溶于 50ml 甲酸铵溶液 (1.0mol/L, 用氨水调节 pH 至 7.2), 加入 1.63g 固定化 D-氨基酸氧化酶 (580U/g)、1.6ml 过氧化氢酶溶液 (50000U/ml)、1g Pd-C 催化剂 (10%), 连续通入氧气, 30°C 下反应 24h, 反应液旋光度不再增加。过

滤除去固定化 D- 氨基酸氧化酶和 Pd-C, 滤液浓缩至约 10ml, 调等电点, 冷冻析出淡黄色晶体, 烘干得 2.8g L- 哌嗪 -2- 羧酸, 收率 86%, ee > 99%。

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(54) Title: SOLVENT SYSTEMS FOR PRODUCING PUMPABLE FORMULATIONS OF HIGH ACTIVE ETHER SULFATES

(57) Abstract: Novel methods and compositions are provided for reducing the viscosity of high alkyl ether sulfate surfactants (e.g., 70 % sodium lauryl ether sulfate) without using highly flammable solvents or preservatives. The present invention alleviates the problems of the prior art by providing novel methods for reducing viscosity of high active alkyl ether sulfates (e.g., sodium lauryl ether sulfate or ammonium lauryl ether sulfate) by mixing high active alkyl ether sulfates with one or more solvents (solvent blend) and novel compositions with reduced the viscosity of the surfactants prior to use.

**SOLVENT SYSTEMS FOR PRODUCING PUMPABLE FORMULATIONS
OF HIGH ACTIVE ETHER SULFATES**

FIELD OF THE INVENTION

[0001] The present invention relates to surfactant compositions with improved ease of handling and pumping using non-specialized equipment which eliminates the use of flammable solvents.

BACKGROUND OF THE INVENTION

[0002] In the surfactant art, some water soluble products are produced in concentrated form for improved resistance to microbial degradation and energy efficiency, with both of these being related to the amount of water present in the composition. Lower water content inhibits microbial growth and eliminates the need for preservatives, but removing all traces of water can require large amounts of energy. In the industry, such forms of surfactants are known as “high active” and are used with surfactant products such as sodium lauryl ether sulfate (“SLES” or “sodium laureth sulfate”).

[0003] In concentrated forms of some surfactants, a gel phase occurs when the surfactant concentration passes a threshold level and there is not enough water present to ensure solubility of the surfactant. A particular example of this problem occurs with SLES, which forms a gel phase when the surfactant concentration exceeds approximately 27 % of an aqueous solution. When a gel phase forms, SLES is difficult to pump using conventional pumps (such as

diaphragm type pumps) and specialized equipment (such as positive displacement or tri-lobe pumps) is required.

[0004] Additionally, such concentrated forms of SLES can exhibit instability when unexpectedly heated above approximately 140 °F. The product can hydrolyze, releasing sulfuric acid, thus damaging process equipment. The heat of a product process reaction, once it is started, does not dissipate because of the gel nature of high active SLES. This can lead to very high temperatures in the process reaction, and further lead to a runaway reaction, which can destroy the whole batch of product. Of great interest to the surfactant community is a more dilute form(s) of SLES, while also eliminating the problems of (1) microbial contamination, (2) gel phase formation and attendant high viscosity, and (3) high reactivity.

[0005] One method for overcoming the high viscosity of high active surfactants has been the addition of volatile solvents (such as iso-propanol). However, this is a flammable solvent, which results in safety concerns in mixing and shipping operations. Other flammable solvents have been reported for some applications. *See U.S. Pat. Nos. 7,842,647 & 8,445,406* (the “Long patents”; both hereby incorporated by reference in their entirety). Additional solvents are needed to allow for dilution of high active surfactants for specific uses, including in cleaning and pesticide formulations.

SUMMARY OF THE INVENTION

[0006] The present invention alleviates the problems of the prior art discussed above by providing novel methods for reducing viscosity of high active alkyl ether sulfates (e.g., sodium lauryl ether sulfate or ammonium lauryl ether sulfate) by mixing said high active alkyl ether sulfates with one or more solvents (solvent blend) and novel compositions with reduced the viscosity of the surfactants prior to use. The present invention accomplishes these goals without

use of flammable solvents or the requirement of using a preservative. The resulting mixtures (surfactant-solvent blends, surfactant-solvent blend solutions, and liquid agrochemical compositions) can be easily pumped, once blended, over a temperature range of about 4-50 °C, without the need for special equipment. These resulting surfactant-solvent blends can be used to formulate cleaning products or pesticides, including herbicides. The surfactant-solvent blends described are stable and do not separate upon storage or at elevated (e.g., 50 °C) or low (e.g., 4 °C) temperatures. The described blends have viscosities in the range of 300-1000 cps at 20 °C and are generally clear in appearance. These blends can be readily transported in jugs, pails, drums, totes, or in bulk containers without the need for special equipment to unload the shipping and/or storage containers.

[0007] Therefore, according to one aspect, the invention comprises a method for reducing the viscosity of a high active (concentrated at 27-70%) alkyl ether sulfate surfactant solution comprising providing a high active concentrated alkyl ether sulfate surfactant solution and mixing hexylene glycol as a solvent into said high active concentrated alkyl ether sulfate surfactant solution. In some embodiments, said high active concentrated alkyl ether sulfate surfactant is selected from the group comprising sodium lauryl ether sulfate and ammonium lauryl ether sulfate. In other embodiments, at least one co-solvent in the amount of about 0.1 % to about 90 % of the total solvent amount by weight is further mixed into said high active concentrated alkyl ether sulfate surfactant solution. In yet further embodiments, at least one co-solvent is selected from the group consisting of diethylene glycol, ethylene glycol, propylene glycol, dipropylene glycol methyl ether, propylene glycol monomethyl ether, or combinations thereof is/are mixed into said high active concentrated alkyl ether sulfate surfactant solution. In preferred embodiments, said at least one co-solvent is diethylene glycol. In further preferred

embodiments, said at least one co-solvent is a combination of diethylene glycol and propylene glycol monomethyl ether. In still further preferred embodiments, at least one additional surfactant selected from the group consisting of alkylpolyglucosides and alcohol alkoxyates in amount of about 0.1 % to about 50 % by weight is further mixed into said high active concentrated alkyl ether sulfate surfactant solution.

[0008] According to another aspect, the invention comprises a method for treating an agricultural substrate comprising contacting said agricultural substrate with a sufficient amount of a liquid agrochemical composition spray solution containing a surfactant-solvent blend comprising a high active concentrated alkyl ether sulfate surfactant solution and a solvent mixed into said high active concentrated alkyl ether sulfate surfactant solution, wherein said solvent is hexylene glycol. In some embodiments, said liquid agrochemical composition spray solution comprises an active agrochemical agent selected from the group consisting of a herbicide, an insecticide, a fungicide, a miticide, a fertilizer, or a plant growth regulator, or combinations thereof. In preferred embodiments, said herbicide is glufosinate or salts thereof.

[0009] According to yet another aspect, the invention comprises a liquid agrochemical composition comprising an active agrochemical agent and a surfactant-solvent blend solution, wherein said surfactant-solvent blend solution comprises a high active (concentrated at 27-60 %) alkyl ether sulfate surfactant solution and a hexylene glycol solvent. In some embodiments, said high active concentrated alkyl ether sulfate surfactant is selected from the group comprising sodium lauryl ether sulfate and ammonium lauryl ether sulfate. In other embodiments, said hexylene glycol solvent comprises about 1 % to about 5 % by weight of said composition. In further embodiments, at least one co-solvent is selected from the group consisting of diethylene glycol, ethylene glycol, propylene glycol, dipropylene glycol methyl ether, propylene glycol

monomethyl ether, or combinations thereof is/are mixed into said high active concentrated alkyl ether sulfate surfactant solution. In preferred embodiments, said at least one co-solvent is diethylene glycol. In further preferred embodiments, said at least one co-solvent is a combination of diethylene glycol and propylene glycol monomethyl ether. In still further embodiments, said at least one co-solvent in the amount of about 5 % to about 80 % of the total solvent amount by weight is further mixed into said high active concentrated alkyl ether sulfate surfactant solution. In still further preferred embodiments, said high active concentrated alkyl ether sulfate surfactant comprises about 75 % to about 90 % of the total surfactant amount by weight in said composition. In yet other embodiments, at least one additional surfactant selected from the group consisting of alkylpolyglucosides and alcohol alkoxyates is further mixed into said high active concentrated alkyl ether sulfate surfactant solution. In yet further preferred embodiments, said at least one additional surfactant comprises about 0.1 to about 50 % by weight of said composition.

DETAILED DESCRIPTION OF INVENTION

[0010] The following detailed description is presented to enable any person skilled in the art to make and use the invention. For purposes of explanation, specific details are set forth to provide a thorough understanding of the present invention. However, it will be apparent to one skilled in the art that these specific details are not required to practice the invention. Descriptions of specific applications are provided only as representative examples. Various modifications to the preferred embodiments will be readily apparent to one skilled in the art, and the general principles defined herein may be applied to other embodiments and applications without departing from the scope of the invention. The present invention is not intended to be

limited to the embodiments shown, but is to be accorded the widest possible scope consistent with the principles and features disclosed herein.

[0011] A particular interest in the field is the development of a sodium lauryl ether sulfate (“SLES” or “sodium laureth sulfate”) or ammonium lauryl ether sulfate (“ALES” or “ammonium laureth sulfate”) solution for formulators (or “users”) who neither possess the specialized equipment required to handle high active surfactant nor the storage capability for large quantities of flammable material (i.e., volatile organic solvents). For these users, a pumpable and non-flammable version of SLES containing up to 60 % surfactant but greater than 27 % surfactant (maximum aqueous solution SLES concentration without a gel phase) that does not require preservatives is desirable. A formulation of SLES that fits these criteria will allow the surfactant to have a greater utility in terms of both more applications and a wider range of potential users.

[0012] The use of a non-flammable or reduced-flammability solvent is needed in order to produce a non-flammable, free-flowing solution of SLES that avoids a gel phase by diluting high active SLES (approximately 70 % SLES and approximately 30 % water). Preferably, the solvent selected should be efficient at reducing the viscosity of the surfactant and readily available. More preferably, the solvent selected should also result in a single phase or substantially single phase solution, where substantially single phase is defined as minimally or slightly cloudy in appearance.

[0013] During our research into this problem, we unexpectedly discovered that hexylene glycol was an effective solvent for creating surfactant-solvent blend solutions of high active alkyl ether sulfate surfactants (*see* Tables 1-3). In addition, we have discovered hexylene glycol is also useful for the purpose of making an easily pumpable solution of high active alkyl ether sulfate

surfactants that can be readily transported in common and non-hazardous shipping containers. Previously, blends of the solvents THFA and dipropylene glycol with the solvents iso-propanol or 1-butanol have been claimed to be useful solvent blends for SLES in concentrated herbicide formulations (*see* the Long patents). However, THFA, iso-propanol and 1-butanol pose significant fire safety hazards.

[0014] In addition to decreasing the viscosity of high active SLES, hexylene glycol also can be used as a component of pesticide and herbicide formulations because it is an approved inert ingredient according to the U.S. Environmental Protection Agency (*see* U.S. EPA *InertFinder*). Further anticipated applications for the hexylene glycol diluted high active SLES solutions of the present invention include formulations for cleaning products, such as soaps, detergents, shampoos, and similar products.

[0015] For the above reasons, hexylene glycol is uniquely qualified as a solvent to produce a surfactant (e.g., SLES) blend solution with properties not currently available (i.e., having improved safety characteristics as compared to current commercial formulations).

[0016] In one aspect, the present invention comprises a surfactant-solvent blend solution of a high active alkyl ether sulfate surfactant and hexylene glycol as a solvent. In another aspect, the surfactant-solvent blend solution of the present invention can then be further modified to obtain desired physical or chemical properties. In yet another aspect, the resulting surfactant-solvent blend solution can be used in manufacturing active agent solutions, with or without modification. In still another aspect, the surfactant-solvent blend solution of the present invention can be used to manufacture concentrated active agent solutions, such as concentrated glufosinate-ammonium herbicide solutions and mixtures of glufosinate with other herbicides.

[0017] With the decrease in viscosity afforded by hexylene glycol, other solvents and surfactants may then be added to the SLES solution without adversely affecting the viscosity of the solution, but modifying the physical and/or chemical properties of the resulting formulation. For example, non-ionic surfactants such as alkyl polyglucosides ("APG"), co-solvents such as diethylene glycol, and other additional components may be added without deleterious effect.

[0018] Suitable alkyl ether sulfate surfactants that may be used in the present invention include sodium lauryl ether sulfate, ammonium lauryl ether sulfate, and/or other salts of lauryl ether sulfate. Preferably, the alkyl ether sulfate surfactant is a high active concentrated alkyl ether sulfate surfactant, such as 70 % active SLES, ALES, or mixtures thereof, available from BASF. More preferably the alkyl ether sulfate surfactant is a high active SLES. The alkyl ether sulfate surfactant can be manufactured from petroleum or vegetable sources. The alkyl ether sulfate surfactant may be present in the present invention at about 25 % to about 60 % by weight. In embodiments of surfactant-solvent blend solutions, the preferable range is about 35 % to about 50 % by weight, and more preferably between about 40 % to about 45 % by weight. In embodiments of surfactant-solvent blend solution formulas blended with active agrochemical agents (liquid agrochemical compositions), the preferable range is about 25 % to about 35% by weight.

[0019] In addition to alkyl ether sulfate surfactants, the present invention may optionally contain one or more co-surfactant(s) or additional surfactant(s). Additional surfactants suitable for use in the present invention include non-ionic surfactants, such as alkyl polyglycosides and alcohol alkoxyates. Preferably, the alkyl polyglycosides used in the present invention are alkyl polyglucosides. Examples of suitable commercial alkyl polyglucosides for use in the present invention include AGNIQUE® PG series, such as AGNIQUE® PG-8105, from BASF or similar

surfactant products. Preferably, the alcohol alkoxylates used in the present invention are alcohol ethoxylates. Examples of suitable commercial alcohol alkoxylates for use in the present invention include SURFONIC® L series surfactants, such as SURFONIC® L-24-3, from Huntsman International LLC, ECOSURF® EH series surfactants, such as ECOSURF® EH-3 or ECOSURF® EH-6, from Dow Chemical Company, AGNIQUE IDA-6 from BASF Corp., or similar surfactant products. The additional surfactant may be present in the present invention at about 0.1 % to about 50 % by weight. Preferably, the additional surfactant is present at about 10 % to about 20 % by weight.

[0020] Previously, Long and others taught that the following solvents may be suitable for diluting high active SLES: cyclic alcohols such as tetrahydrofurfuryl alcohol (THFA); aliphatic alcohols, such as alkanols having 1 to 12 carbon atoms, usually 1 to 6 carbon atoms, such as methanol, ethanol, propanol, iso-propanol and butanol, for example, or polyhydric alcohols such as ethylene glycol, propylene glycol, dipropylene glycol, and glycerol; ethers such as diethyl ether, tetrahydrofuran (THF), and dioxane; alkylene glycol monoalkyl and dialkyl ethers, such as propylene glycol monomethyl ether, propylene glycol monoethyl ether, ethylene glycol monomethyl and monoethyl ether, diglyme, and tetraglyme, for example; amides such as dimethylformamide (DMF), dimethylacetamide, dimethylcaprylamide, dimethylcapramide, and N-alkylpyrrolidones; ketones such as acetone, cyclohexanone, acetophenone, butyrolactone; esters based on glyceryl and carboxylic acids, such as glyceryl mono-, di- and triacetate, phthalic esters, ethyl lactate, 2-ethylhexyl lactate; lactams; carbonic diesters; nitriles such as acetonitrile, propionitrile, butyronitrile, and benzonitrile; sulfoxides and sulfones such as dimethyl sulfoxide (DMSO) and sulfolane; carbonates such as propylene or butylene carbonate; and combinations of different solvents, additionally containing alcohols such as methanol, ethanol, n- and iso-

propanol, and n-, iso-, tert- and 2-butanol. However, many of these organic solvents pose serious fire hazards in storage, shipping, and/or use conditions. Therefore, most, if not all, of these solvents may not be suitable for the present invention. In one aspect, the present invention comprises a solvent that is non-flammable or only has low-flammability characteristics in order to provide a SLES solution without the use of fire hazard solvents. In preferred embodiments, the present invention comprises hexylene glycol ("2-methylpentane-2,4-diol," CAS 107-41-5 from Solvay) as a solvent. The solvent may be present in the present invention at about 1 % to about 30 % by weight.

[0021] In another aspect, the present invention additionally comprises one or more co-solvents. Preferably, the co-solvent comprises a non- or low-flammable solvent or a combination of non- and/or low-flammable solvents. In preferred embodiments, the present invention comprises hexylene glycol and at least one co-solvent selected from the group consisting of diethylene glycol, ethylene glycol, propylene glycol, dipropylene glycol methyl ether, propylene glycol monomethyl ether or combinations thereof. In more preferred embodiments, the present invention comprises hexylene glycol and co-solvents diethylene glycol (CAS 111-46-6 available from MEGlobal), dipropylene glycol methyl ether ("2-methoxymethylethoxy) propanol," "2-(2-methoxypropoxy)propanol," "dipropylene glycol monomethyl ether," "DPM," DOWANOL™ DPM glycol ether, CAS 34590-94-8 (mixed isomers) available from DOW), and/or propylene glycol monomethyl ether ("1-methoxy-2-propanol," or "propylene glycol methyl ether," DOWANOL™ PM, CAS 107-98-2 available from DOW). The co-solvent (including mixtures thereof) may be present in the present invention at about 0.1 % to about 90 % by weight. Preferably, the co-solvent (including mixtures thereof) may be present in the present invention at about 1 % to about 50 % by weight of said composition.

[0022] In yet another aspect, the present invention comprises a liquid agrochemical composition comprising an active agrochemical agent and a surfactant-solvent blend solution as described above. The active agrochemical agent may include at least one of the group consisting of a herbicide, insecticide, fungicide, miticide, fertilizer, or plant growth regulator, or combinations thereof. In some embodiments, the present invention may be a concentrated liquid agrochemical composition formulation. In preferred embodiments, the present invention is a concentrated liquid agrochemical composition solution capable of being stored and pumped or poured at freezing or near-freezing temperatures or it may be diluted with an appropriate aqueous solution capable of being sprayed at freezing or near-freezing temperatures.

[0023] In some embodiments, the liquid agrochemical composition of the present invention comprises a water-soluble active herbicide agent selected from the group consisting of glufosinate, salts thereof, and/or other similar active compounds. In preferred embodiments, the liquid agrochemical composition of the present invention comprises the ammonium salt of glufosinate (CAS 77182-82-2).

[0024] Preferably, the liquid agrochemical composition spray solution of the present invention is applied to an "agricultural substrate" in a "sufficient amount" to have an appropriate desired effect. An agricultural substrate is a living or growing plant that is intended to be affected by application of a liquid agrochemical composition spray solution. For example, a liquid agrochemical composition spray solution of the present invention comprising a herbicide active agrochemical agent will be applied in sufficient amount to damage or inhibit the growth of unwanted plants (i.e., the agricultural substrate) growing in an undesirable area. More preferably, the sufficient amount of the liquid agrochemical composition spray solution of the

present invention is an amount that will not adversely affect agricultural crops to be protected by application of a liquid agrochemical composition spray solution.

[0025] The resulting mixtures and liquid agrochemical compositions may optionally comprise secondary chemical agents, depending on the application of the mixture/composition. For example, a herbicide containing liquid agrochemical compositions of the present invention may optionally include such secondary chemical agents known in the art as defoamers, dispersants, wetting agents, emulsifiers, penetrants, antifreezes, dyes to provide color, pH buffers and/or modifiers, and other known compounds for altering or enhancing the performance or physical/chemical characteristics of said liquid agrochemical compositions.

EXAMPLES

[0026] The following Examples 1-7 are provided to illustrate the unexpected superior performance of surfactant-solvent blend solutions containing 30 % hexylene glycol solvent. Viscosity results of 70 % SLES diluted with various glycol are solvents shown in Table 1. High active 70 % SLES (BASF) was mixed with 10, 20, and 30 % of various glycol solvents (glycerin, propylene glycol, hexylene glycol, dipropylene glycol, diethylene glycol, ethylene glycol, and PEG-200), then viscosity was measured. Viscosity measurement conditions: viscosity in cps, Brookfield Viscometer, number 2 spindle at 12 rpm, 25 °C. Hexylene glycol is superior to all glycols tested at 30 % with 70 % high active SLES.

[0027] TABLE 1

	Solvent						
% solvent	1 Glycerin	2 Propylene glycol	3 Hexylene glycol	4 Dipropylene glycol	5 Diethylene glycol	6 Ethylene glycol	7 PEG- 200
10 %	gel	gel	gel	gel	gel	gel	gel
20 %	gel	gel	1000	600	gel	gel	gel
30 %	gel	188	50	625	gel	gel	gel

[0028] The following Examples 8-18 are provided to illustrate the unexpected superior performance of surfactant-solvent blend solutions containing a blend of hexylene glycol, diethylene glycol, and DPM solvents. Surfactant-solvent blend solutions were prepared containing 60 % of high active SLES 70 % (BASF), 10 to 15 % additional surfactant(s), and diluted with 25 to 30 % of various glycol solvents in Table 2. The prepared example solutions 8-18 were then observed for liquid or gel phase state at 22 °C, 4 °C, and 50 °C.

[0029] The data in Table 2 indicate that not all glycols are suitable solvents for high active SLES. Diethylene glycol, ethylene glycol, polyethylene glycol 200, and glycerin all form gels at 22 °C and are not suitable viscosity reducers. At 22 °C, hexylene glycol, propylene glycol dipropylene glycol and blends of hexylene glycol and diethylene glycol form clear solutions. At 4 °C, the only solvent blend that is fluid is a blend of diethylene glycol with hexylene glycol. Both individual components form gels at 4 °C. Only the surfactant-solvent blend solution containing hexylene glycol, diethylene glycol, and DPM solvents was at least a substantially single phase liquid at all temperatures tested. Unless indicated otherwise, all percentages are amounts by weight.

[0030] TABLE 2

Formula (%)	8	9	10	11	12	13	14	15	16	17	18
Diethylene glycol	23	28							18	18	18
Hexylene glycol	5		28						5	5	5
Ethylene glycol				28							
Propylene glycol					28						
Dipropylene glycol						28					
PEG 200							28				
Glycerin								28			
Ecosurf EH-6									5		
Surfonic L-24-3										5	
Ecosurf EH-3											5
DPM	2	2	2	2	2	2	2	2	2	2	2
AGNIQUE® PG 8105	10	10	10	10	10	10	10	10	10	10	10
SLES (70%) BASF	60	60	60	60	60	60	60	60	60	60	60
at 22 °C	clear	gel	clear	gel	clear	clear	gel	gel	clear	clear	clear
at 4 °C	slightly hazy	---	cloudy solid	---	cloudy solid	cloudy solid	---	---	solid	solid	paste flows
at 50 °C	clear	---	bottom separation	---	bottom separation	clear	---	---	floc	floc	floc

[0031] The data in Table 3 indicate that only formulas containing (a) a blend of hexylene glycol and diethylene glycol or (b) only propylene glycol form cold stable single phases when blended with the ammonium salt of glufosinate. Diethylene glycol, ethylene glycol, polyethylene glycol (PEG) 200, and glycerin all form gels at 22 °C and are not suitable viscosity reducers when blended with the ammonium salt of glufosinate. At 22 °C, hexylene glycol, propylene

glycol, dipropylene glycol, and blends of hexylene glycol and diethylene glycol form clear solutions. At 4 °C, the only solvent blend that is fluid is a blend of diethylene glycol with hexylene glycol. Both individual components form gels at 4 °C.

[0032] TABLE 3

Formula (%)	19	20	21	22	23	24	25	26	27	28	29
Diethylene glycol	23	28							18	18	18
Hexylene glycol	5		28						5	5	5
Ethylene glycol				28							
Propylene glycol					28						
Dipropylene glycol						28					
PEG 200							28				
Glycerin								28			
Ecosurf EH-6									5		
Surfonic L-24-3										5	
Ecosurf EH-3											5
DPM	2	2	2	2	2	2	2	2	2	2	2
Agnique PG 8105	10	10	10	10	10	10	10	10	10	10	10
SLES (70) BASF	60	60	60	60	60	60	60	60	60	60	60
51 % of the above formulations 19-29, respectively, 22.4 % water, 25.7 % glufosinate ammonium (97 % PURITY), and 0.90 % ammonium hydroxide (30%)											
Glufosinate 280 g/L SL at 4 °C	clear	---	Phase separation	---	clear	Phase separation	---	---	Phase separation	Phase separation	Phase separation
Viscosity #2 spindle 12 rpm at 6 °C	500 cps	---	not tested	---	800 cps	---	---	---	not tested	not tested	not tested

[0033] The terms "comprising," "including," and "having," as used in the claims and specification herein, shall be considered as indicating an open group that may include other elements not specified. The terms "a," "an," and the singular forms of words shall be taken to include the plural form of the same words, such that the terms mean that one or more of something is provided. The term "one" or "single" may be used to indicate that one and only one of something is intended. Similarly, other specific integer values, such as "two," may be used when a specific number of things is intended. The terms "preferably," "preferred," "prefer," "optionally," "may," and similar terms are used to indicate that an item, condition or step being referred to is an optional (not required) feature of the invention.

[0034] The invention has been described with reference to various specific and preferred embodiments and techniques. However, it should be understood that many variations and modifications may be made while remaining within the spirit and scope of the invention. It will be apparent to one of ordinary skill in the art that methods, devices, device elements, materials, procedures and techniques other than those specifically described herein can be applied to the practice of the invention as broadly disclosed herein without resort to undue experimentation. All art-known functional equivalents of methods, devices, device elements, materials, procedures and techniques described herein are intended to be encompassed by this invention. Whenever a range is disclosed, all sub-ranges and individual values are intended to be encompassed. This invention is not to be limited by the embodiments disclosed, including any exemplified in the specification, which are given by way of example and not of limitation.

[0035] All references throughout this application, for example patent documents including issued or granted patents or equivalents, patent application publications, and non-patent literature documents or other source material, are hereby incorporated by reference herein

in their entireties, as though individually incorporated by reference, to the extent each reference is at least partially not inconsistent with the disclosure in the present application (for example, a reference that is partially inconsistent is incorporated by reference except for the partially inconsistent portion of the reference).

[0036] While the invention has been described with respect to a limited number of embodiments, those skilled in the art, having benefit of this disclosure, will appreciate that other embodiments can be devised which do not depart from the scope of the invention as disclosed herein. Accordingly, the scope of the invention should be limited only by the attached claims.

CLAIMS

We Claim:

1. A method for reducing the viscosity of a high active concentrated alkyl ether sulfate surfactant solution comprising: providing a high active concentrated alkyl ether sulfate surfactant solution and mixing hexylene glycol as a solvent into said high active concentrated alkyl ether sulfate surfactant solution.
2. The method of claim 1, wherein said high active concentrated alkyl ether sulfate surfactant is selected from the group comprising sodium lauryl ether sulfate and ammonium lauryl ether sulfate.
3. The method of claim 1, further comprising mixing at least one co-solvent in the amount of about 0.1 % to about 90 % of the total solvent amount by weight into said high active concentrated alkyl ether sulfate surfactant solution.
4. The method of claim 3, wherein said at least one co-solvent is selected from the group consisting of diethylene glycol, ethylene glycol, propylene glycol, dipropylene glycol methyl ether, propylene glycol monomethyl ether, or combinations thereof.
5. The method of claim 3, wherein said at least one co-solvent is diethylene glycol.
6. The method of claim 3, wherein said at least one co-solvent is a combination of diethylene glycol and dipropylene glycol methyl ether.
7. The method of claim 4, further comprising mixing at least one additional surfactant selected from the group consisting of alkylpolyglucosides, alcohol alkoxylates, and combinations thereof in amount of about 0.1 % to about 50 % by weight into said high active concentrated alkyl ether sulfate surfactant solution.
8. A method for treating an agricultural substrate comprising:

- a. Contacting said agricultural substrate with a sufficient amount of a liquid agrochemical composition spray solution containing a surfactant-solvent blend comprising a high active concentrated alkyl ether sulfate surfactant solution and a solvent mixed into said high active concentrated alkyl ether sulfate surfactant solution, wherein said solvent is hexylene glycol.
9. The method of claim 8, wherein said liquid agrochemical composition spray solution comprises an active agrochemical agent selected from the group consisting of a herbicide, an insecticide, a fungicide, a miticide, a fertilizer, or a plant growth regulator, or combinations thereof.
10. The method of claim 9, wherein said herbicide is glufosinate or salts thereof.
11. A liquid agrochemical composition comprising:
 - a. an active agrochemical agent; and
 - b. a surfactant-solvent blend solution, wherein said surfactant-solvent blend solution comprises a high active concentrated alkyl ether sulfate surfactant solution and a hexylene glycol solvent.
12. The liquid agrochemical composition of claim 11, wherein said high active concentrated alkyl ether sulfate surfactant is selected from the group comprising sodium lauryl ether sulfate and ammonium lauryl ether sulfate.
13. The liquid agrochemical composition of claim 12, wherein said hexylene glycol solvent comprises about 1 % to about 5 % by weight of said composition.
14. The liquid agrochemical composition of claim 12, further comprising at least one co-solvent selected from the group consisting of diethylene glycol, ethylene glycol,

propylene glycol, dipropylene glycol methyl ether, propylene glycol monomethyl ether, or combinations thereof.

15. The liquid agrochemical composition of claim 14, wherein said at least one co-solvent is diethylene glycol.
16. The liquid agrochemical composition of claim 14, wherein said at least one co-solvent is a combination of diethylene glycol and dipropylene glycol methyl ether.
17. The liquid agrochemical composition of claim 14, wherein said at least one co-solvent comprises about 1 % to about 50 % by weight of said composition.
18. The liquid agrochemical composition of claim 12, wherein said high active concentrated alkyl ether sulfate surfactant comprises about 60 % to about 90 % of the total surfactant amount by weight in said composition.
19. The liquid agrochemical composition of claim 16, further comprising at least one additional surfactant selected from the group consisting of alkylpolyglucosides, alcohol alkoxylates, and combinations thereof.
20. The liquid agrochemical composition of claim 19, wherein said at least one additional surfactant comprises about 0.1 % to about 50 % by weight of said composition.

INTERNATIONAL SEARCH REPORT **PCT/US2014/071723 17.04.2015**

International application No.

PCT/US2014/071723

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - A01N 25/02 (2015.01)

CPC - A01N 25/02 (2015.01)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC(8) - A01N 25/02, 25/04, 25/30 (2015.01)

CPC - A01N 25/02, 25/04, 25/30 (2015.01) (keyword delimited)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

USPC - 47/1.5, 1.7; 504/358, 362, 364, 366 (keyword delimited)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Orbit, Google Patents, Google Scholar.

Search terms used: anionic alkyl ether sulfate hexylene glycol viscosity agriculture agrochemical

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6,313,085 B1 (LE HEN-FERRENBACH) 06 November 2001 (06.11.2001) entire document	1, 2
Y		3-7
X	US 2011/0172101 A1 (KRAPP et al) 14 July 2011 (14.07.2011) entire document	8, 9, 11-13, 18
Y		10, 14-17, 19, 20
Y	US 2007/0238631 A1 (GAMBOGI et al) 11 October 2007 (11.10.2007) entire document	3-7
Y	US 2011/0195839 A1 (SCHLOTTERBECK et al) 11 August 2011 (11.08.2011) entire document	10, 14-17, 19, 20

☐ Further documents are listed in the continuation of Box C.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

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Date of the actual completion of the international search

02 March 2015

Date of mailing of the international search report

17 APR 2015

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APPLICATION #
18/435,340RECEIPT DATE / TIME
05/06/2024 09:59:38 AM Z ETATTORNEY DOCKET #
32471/M202647G**Title of Invention**

METHODS FOR MAKING L-GLUFOSINATE

Application InformationAPPLICATION TYPE Utility - Nonprovisional Application
under 35 USC 111(a)

PATENT # -

CONFIRMATION # 4214

FILED BY Peter Adams

PATENT CENTER # 65384795

FILING DATE 02/07/2024

CUSTOMER # 4743

FIRST NAMED INVENTOR Brian Michael Green

CORRESPONDENCE ADDRESS -

AUTHORIZED BY Joshua Elliott

Documents**TOTAL DOCUMENTS: 8**

DOCUMENT	PAGES	DESCRIPTION	SIZE (KB)
Information Disclosure Statement.pdf	2	Transmittal Letter	18 KB
FOR_AU2005249191A1.pdf	1	Foreign Reference	84 KB
FOR_CN102174632.pdf	8	Foreign Reference	1547 KB
FOR_CN103269581.pdf	15	Foreign Reference	1946 KB
FOR_WO2015095824A1.pdf	22	Foreign Reference	3894 KB
NPL_Mehia549.pdf	13	Non Patent Literature	4856 KB
NPL_TransaminaseWikipedia	2	Non Patent Literature	610 KB

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1141A5D97F25AD57DC259CA473490163

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(19) AUSTRALIAN PATENT OFFICE

(54) Title
Concentrated aqueous formulations for crop protection

(51)⁶ International Patent Classification(s)
A01N 25/30 (2006.01)20060101ALI2006072
A01N 25/02 (2006.01)2BMEP A01N
A01N 25/16 (2006.01)25/16
A01N 57/20 (2006.01)20060101ALI2006050
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				Application Number	18/435,340-Conf. #4214
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				First Named Inventor	Brian Michael Green
				Art Unit	
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Sheet	1	of	10	Attorney Docket Number	32471/M202647G

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	**	CN-105218579-A	01-06-2016	Jiangsu Sevencontinent Green Chemical Co., Ltd		√

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	**	CN-105567780-A	05-11-2016	Chongqing Huijian Biotech Co., Ltd		
	**	CN-105603015-A	05-25-2016	Univ Zhejiang		√
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Examiner Signature		Date Considered	
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Stabilized liquid aqueous crop protection agent composition

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Abstract of CN103269581 (A)

Provided is a water-soluble herbicidal composition having stability at low temperatures, which is capable of maintaining a continuous single phase without causing precipitation when stored at low temperatures. Specifically provided is a liquid herbicidal composition which is characterized by containing the following components (a)-(d): (a) a water-soluble herbicidal component; (b) a polyoxyethylene alkyl ether sulfate; (c) an organic solvent; and (d) a polyoxyethylene alkyl ether.



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(54) 发明名称

被稳定化了的液状水性作物保护剂组合物

(57) 摘要

本发明提供低温保存时不发生沉淀而可以保持连续的单相的具有低温稳定性的水溶性除草剂组合物。本发明提供液体除草剂组合物,其特征在于,含有以下的(a)~(d):(a)水溶性除草剂成分;(b)聚氧乙烯烷基醚硫酸盐;(c)有机溶剂;以及(d)聚氧乙烯烷基醚。

1. 一种液体除草剂组合物,其包含以下的(a)~(d):
 - (a) 水溶性除草剂成分;
 - (b) 聚氧乙烯烷基醚硫酸盐;
 - (c) 有机溶剂;以及
 - (d) 聚氧乙烯烷基醚,并且,在-5℃能够保持连续的单相至少3天。
2. 根据权利要求1所述的组合物,其中,(a)水溶性除草剂成分是选自由草铵膦及其盐、草铵膦-P及其盐、草甘膦及其盐、百草枯,以及敌草快所组成的组中的1种或2种以上的组合。
3. 根据权利要求1所述的组合物,其中,(a)水溶性除草剂成分是选自由草铵膦及其盐、以及草铵膦-P及其盐所组成的组中的1种或2种以上的组合。
4. 根据权利要求1所述的组合物,其中,(a)水溶性除草剂成分是选自由草铵膦-P及其盐所组成的组中的1种或2种以上的组合。
5. 根据权利要求1所述的组合物,其中,(a)水溶性除草剂成分以组合物的总重量的1~50重量份存在。
6. 根据权利要求1所述的组合物,其中,(a)水溶性除草剂成分以组合物的总重量的6~17重量份存在。
7. 根据权利要求1所述的组合物,其中,(b)聚氧乙烯烷基醚硫酸盐中的烷基的碳原子数为8~16。
8. 根据权利要求1所述的组合物,其中,(b)聚氧乙烯烷基醚硫酸盐以组合物的总重量的5~50重量份存在。
9. 根据权利要求1所述的组合物,其中,(b)聚氧乙烯烷基醚硫酸盐以组合物的总重量的10~35重量份存在。
10. 根据权利要求1所述的组合物,其中,(c)有机溶剂为丙二醇单甲醚。
11. 根据权利要求1所述的组合物,其中,(c)有机溶剂以组合物的总重量的1~30重量份存在。
12. 根据权利要求1所述的组合物,其中,(c)有机溶剂以组合物的总重量的5~15重量份存在。
13. 根据权利要求1所述的组合物,其中,(d)聚氧乙烯烷基醚中的聚氧乙烯的加成摩尔数为1~20,烷基的碳原子数为8~16。
14. 根据权利要求1所述的组合物,其中,(d)聚氧乙烯烷基醚以组合物的总重量的0.1~20重量份存在。
15. 根据权利要求1所述的组合物,其中,(d)聚氧乙烯烷基醚以组合物的总重量的2~10重量份存在。
16. 根据权利要求1所述的组合物,其除了包含(a)~(d)以外还包含(e)消泡剂。

被稳定化了的液状水性作物保护剂组合物

技术领域

[0001] 相关申请的记载

[0002] 本发明是根据日本专利申请：日本特愿 2010 - 283920 号（2010 年 12 月 21 日提出申请）而主张优先权，并在本说明书中以引用的方式并入记载该申请的全部记载内容。

背景技术

[0003] 作为非选择性茎叶处理剂使用的水溶性除草剂成分，使用于许多的农业场合，作为农作物保护剂是非常有用的。一般而言，为了增强除草剂的生物活性，利用表面活性剂作为佐剂。其中，关于将草铵膦作为有效成分的除草剂，如日本特公平 01 - 24765 号公报（专利文献 1）所示可知，聚氧乙烯 C_{12} - C_{16} 烷基醚硫酸盐可增强草铵膦的生物活性。

[0004] 但是，使用聚氧乙烯 C_{12} - C_{16} 烷基醚硫酸盐所制成的制剂，在低温的保存稳定性极端地降低，并且有时会引起沉淀。如果发生沉淀，则各种制剂成分的浓度变得不均匀，因此可能会引起生物活性降低。因此即使在低温保存时也不会发生沉淀的制剂是必要的。

[0005] 以提高农作物保护剂，特别是草铵膦制剂的低温稳定性为目的，在日本特表 2009 - 525345 号公报（专利文献 2）中公开了配合有烷基聚葡萄糖苷的制剂。

[0006] 此外，以增强生物活性为目的，在日本特开 2006 - 117650 号公报（专利文献 3）中，公开了并用草铵膦、聚氧化烯烷基醚、聚氧乙烯月桂基醚硫酸钠 3 种成分，且含有总重量为 3.5 重量 % 的聚氧乙烯月桂基醚硫酸钠与聚氧化烯烷基醚的组合物。

[0007] 现有技术文献

[0008] 专利文献

[0009] 专利文献 1：日本特公平 01 - 24765 号公报

[0010] 专利文献 2：日本特表 2009 - 525345 号公报

[0011] 专利文献 3：日本特开 2006 - 117650 号公报

发明内容

[0012] 发明所要解决的问题

[0013] 另外，本说明书中以引用的方式并入记载上述专利文献 1、2 和 3 的全部公开内容。根据本发明而提供以下的分析。

[0014] 然而，有关专利文献 1，聚氧乙烯烷基醚在低温的抑制沉淀发生效果尚未被验证。

[0015] 然而，有关专利文献 2，烷基聚葡萄糖苷与聚氧乙烯烷基醚是完全不同的物质，无法从日本特表 2009 - 525345 号公报（专利文献 2）类推聚氧乙烯烷基醚的效果。

[0016] 然而，有关专利文献 3，并未针对上述成分的含量比这更多的组合物进行验证，且关于制剂物性方面的稳定性也尚未验证。

[0017] 鉴于上述情形，本发明以提供低温保存时不发生沉淀而可以保持连续的单相，具有低温稳定性的水溶性除草剂组合物为目的。

[0018] 解决本发明课题的方法

- [0019] 本发明的一个观点在于提供以下的组合物。
- [0020] 即,提供一种液体除草剂组合物,其包含以下的(a)~(d):
- [0021] (a) 水溶性除草剂成分;
- [0022] (b) 聚氧乙烯烷基醚硫酸盐;
- [0023] (c) 有机溶剂;以及
- [0024] (d) 聚氧乙烯烷基醚,
- [0025] 并且,在-5℃能够保持连续的单相至少3天。(基本组成)
- [0026] 发明的效果
- [0027] 根据本发明,由于在低温保存时不发生沉淀而可以保持连续的单相,因此可以提供具有低温稳定性的水溶性除草剂组合物。

具体实施方式

- [0028] 在本发明中,可以为以下表示的形态。
- [0029] 作为基本组成,如第一观点所示。
- [0030] 作为(a)水溶性除草剂成分,可举出草铵膦及其盐、草铵膦-P及其盐、草甘膦及其盐、百草枯以及敌草快等。
- [0031] 作为优选的除草剂成分,可举出草铵膦及其盐、草铵膦-P及其盐、草甘膦及其盐、百草枯,作为更优选的除草剂成分,可举出草铵膦及其盐、草铵膦-P及其盐,进一步优选为草铵膦-P及其盐。
- [0032] 作为草铵膦、草铵膦-P、草甘膦的盐的形态,可举出铵盐、钾盐、钠盐、二乙胺盐、三乙胺盐等,草铵膦时优选为铵盐,草铵膦-P时优选为钠盐或铵盐。
- [0033] 另外,(a)水溶性除草剂成分的含量,只要在该组合物100重量份中以成为1~50重量份的比例,优选为6~17重量份的比例进行配合即可。
- [0034] 作为(b)聚氧乙烯烷基醚硫酸盐的形态,可举出钠盐、铵盐、烷醇胺盐等,优选为钠盐。
- [0035] 另外,聚氧乙烯烷基醚硫酸盐的烷基的碳原子数优选为8~16,另外,更优选为碳原子数12~16,但也可以不限定此。
- [0036] 此外,关于此处的聚氧乙烯的加成摩尔数并没有规定。
- [0037] 作为(b)聚氧乙烯烷基醚硫酸盐,优选为聚氧乙烯月桂基醚硫酸盐,作为含有聚氧乙烯月桂基醚硫酸盐的表面活性剂,例如可举出サンノール(注册商标)LMT、サンノール(注册商标)NL-1430、サンノール(注册商标)TD-3130(以上ライオン社制)、シノリン(注册商标)SPE-1150、シノリン(注册商标)SPE-1250、シノリン(注册商标)SPE-1350(以上新日本理化社制)、エマール(注册商标)270J(以上花王社制)、Texapon(注册商标)N-70、Texapon(注册商标)N-70EG、Texapon(注册商标)ALES3-70A(以上Cognis社制)、タイポール(注册商标)NLES-227、タイポール(注册商标)NLEA-227、タイポール(注册商标)22HS、AO-3S、タイポール(注册商标)DBET-336、タイポール(注册商标)DBES-327N(以上泰光油脂化学工业社制)等,也可以含有月桂基硫酸盐作为混合存在物。
- [0038] (b)聚氧乙烯烷基醚硫酸盐只要在该组合物100重量份中以成为5~50重量份,

优选为 10 ~ 35 重量份的比例进行配合即可。

[0039] 作为 (c) 有机溶剂,可以以任何比例溶解于水的极性溶剂是适合的,优选为丙二醇单甲醚、异丙醇、1-丁醇、四氢糠醇、二丙二醇,更优选为丙二醇单甲醚,也可以为它们的混合。另外,含量只要在该组合物 100 重量份中以成为 1 ~ 30 重量份的比例,优选为 3 ~ 15 重量份的比例,更优选为 5 ~ 15 重量份的比例进行配合即可。

[0040] 所谓 (d) 聚氧乙烯烷基醚,是指聚氧乙烯加成摩尔数为 1 ~ 20,烷基碳原子数为 8 ~ 16 的聚氧乙烯烷基醚。另外,含量只要在该组合物 100 重量份中以成为 0.1 ~ 20 重量份的比例,优选为 2 ~ 10 重量份的比例进行配合即可。

[0041] 作为配合有辅助剂的本发明的组合物,优选可举出在上述含有 (a) ~ (d) 的组合物中还含有消泡剂作为 (e) 的组合物。作为消泡剂,可举出有机硅乳液、有机硅油、改性有机硅油、乙炔二醇、PO/E0 嵌段聚合物、氟系表面活性剂等。

[0042] 本发明的组合物中根据需要可以进一步配合农药上可容许的制剂用辅助剂,例如 1997 年在“農薬製剤ガイド(农药制剂指南)”(日本植物防疫协会(社)发行)中记载的辅助剂。(本说明书中以引用的方式并入该文献的公开内容)。

[0043] 作为制剂用辅助剂,作为润湿剂,可举出二辛基磺基琥珀酸盐、烷基硫酸盐、聚氧乙烯烯丙醚、乙炔二醇、PO/E0 嵌段聚合物等,作为抗冻剂,可举出丙二醇、乙二醇、甘油等极性溶剂,作为 pH 调节剂,可举出磷酸盐等。

[0044] 本发明的组合物的使用量,可以根据其使用环境、施用时期、施用方法进行适当变更,但通常为每 10 公亩的有效成分量成为 1 ~ 3000g,优选为 10 ~ 300g 那样的量。

[0045] 作为本发明的组合物的形态,除了上述举出的成分之外,也可以为与其他的水溶性除草剂成分混合成的组合物,作为其他的水溶性除草剂成分的例子,可举出灭草松(bentazone)、绿草定(triclopyr)、双草醚(bispyribac-sodium)、灭草烟(imazapyr)、四氟丙酸(flupropanate)等,优选可举出混合有草铵膦或其盐或者草铵膦-P 或其盐、与草甘膦或其盐和 / 或百草枯的组合物。

[0046] 按照本发明的水溶性除草剂组合物,可以通过例如,将上述成分 (a)、(b)、(c)、(d) 全部混合搅拌,使用水合并为 100 重量份而制造。

[0047] 实施例

[0048] 以下记载实施例,但本发明并未限定于这些实施例。

[0049] 实施例 1 ~ 11

[0050] 将通过按照下表 1 和 2 中记载的成分以及比率进行配合而制造的各制剂置入小瓶中。在 -5℃ 的冷冻库静置 3 天后,观察有无沉淀。

[0051] 比较例 1 ~ 11

[0052] 由实施例 1 ~ 11 中的组合物,制造配合水来代替聚氧乙烯烷基醚的制剂,作为比较例。将制造后的各制剂置于小瓶中,在 -5℃ 的冷冻库静置 3 天后,观察有无沉淀。

[0053] 其结果为,在实施例 1 ~ 11 的全部情况下,都未发生沉淀,相对于此,配合水来代替聚氧乙烯烷基醚的比较例 1 ~ 11 中,全部情况下都发生了沉淀。

[0054] 表 1 实施例 1 ~ 6

[0055]

实施例	1	2	3	4	5	6
草铵膦 P Na 盐	17	11		10	10	10
草铵膦 P-NH ₄ 盐			11			
POE(2) 月桂基 (C:12) 醚硫酸 Na	10.5	14	14	17.5	17.5	17.5
丙二醇单甲醚	10	5	10	10	10	10
POE(4) 辛基 (C:8) 醚	2					
POE(7) 异癸基 (C:10) 醚		5				
POE(7) 烷基 (C:12 ~ 14) 醚			5			
POE(6) 十三烷基 (C:13) 醚				5		
POE(7) 十三烷基 (C:13) 醚					5	
POE(10) 油基 (C:18) 醚						10
POE(7) 辛基 (C:8) 醚						
水	其余部分	其余部分	其余部分	其余部分	其余部分	其余部分
在 5℃ 保存 3 天后的沉淀	无	无	无	无	无	无

[0056] 表 2 实施例 7 ~ 11

[0057]

实施例	7	8	9	10	11
草铵膦 P-Na 盐	11			6	6
草铵膦 P-NH ₄ 盐		6	6		
POE(2) 月桂基 (C:12) 醚硫酸 Na	21	21	21	28	35
丙二醇单甲醚	10	10	15	10	10
POE(4) 辛基 (C:8) 醚					
POE(7) 异癸基 (C:10) 醚					10
POE(7) 烷基 (C:12 ~ 14) 醚		5	5		
POE(6) 十三烷基 (C:13) 醚					
POE(7) 十三烷基 (C:13) 醚					
POE(10) 油基 (C:18) 醚					
POE(7) 辛基 (C:8) 醚	5			5	
水	其余部分	其余部分	其余部分	其余部分	其余部分
在 -5℃ 保存 3 天后的沉淀	无	无	无	无	无

[0058] 表 3 比较例 1 ~ 6

[0059]

比较例	1	2	3	4	5	6
草铵膦 P-Na 盐	17	11		10	10	10
草铵膦 P-NH ₄ 盐			11			
POE(2) 月桂基 (C :12) 酰硫酸 Na	10.5	14	14	17.5	17.5	17.5
丙二醇单甲醚	10	5	10	10	10	10
水	其余部分	其余部分	其余部分	其余部分	其余部分	其余部分
在 -5℃保存 3 天后的沉淀	有	有	有	有	有	有

[0060] 表 4 比较例 7 ~ 11

[0061]

比较例	7	8	9	10	11
草铵膦 P-Na 盐	11			6	6
草铵膦 P-NH ₄ 盐		6	6		
POE(2) 月桂基 (C : 12) 酰硫酸 Na	21	21	21	28	35
丙二醇单甲醚	10	10	15	10	10
水	其余部分	其余部分	其余部分	其余部分	其余部分
在 -5℃保存 3 天后的沉淀	有	有	有	有	有

[0062] 实施例 12 ~ 14

[0063] 作为更具体的组合物,将添加消泡剂,并按照下表 5 中记载的成分以及比率制造的各制剂置于小瓶中,在 -5℃ 的冷冻库静置 3 天后,观察有无沉淀。

[0064] 比较例 12

[0065] 由实施例 12 ~ 14 中的组合物,制造配合水来代替聚氧乙烯烷基醚的制剂,作为比较例。将制造后的制剂置于小瓶中,在 -5℃ 的冷冻库静置 3 天后,观察有无沉淀。

[0066] 其结果为,实施例 12 ~ 14 的全部情况下,都未发生沉淀,相对于此,配合水来代替聚氧乙烯烷基醚的比较例 12 中,发生了沉淀。

[0067] 表 5 实施例 12 ~ 14

[0068]

实施例	12	13	14
草铵膦 P Na 盐	11	11	11
POE(2) 月桂基 (C:12) 醚硫酸 Na	17.5	17.5	17.5
丙二醇单甲醚	10	10	10
POE(7) 烷基 (C:12 ~ 14) 醚	6		
POE(7) 烷基 (C:10) 醚		3	
POE(4) 烷基 (C:10) 醚			10
有机硅乳液消泡剂	0.05	0.05	0.05
水	其余部分	其余部分	其余部分
在 5℃ 保存 3 天后的沉淀	无	无	无

[0069] 表 6 比较例 12

[0070]

比较例	12
草铵膦 P-Na 盐	11
POE(2) 月桂基 (C:12) 醚硫酸 Na	17.5
丙二醇单甲醚	10
有机硅乳液消泡剂	0.05
水	其余部分
在 5℃ 保存 3 天后的沉淀	有

[0071] 此外,本说明书中以引用的方式并入上述专利文献的各公开内容。在本发明的全

部公开内容（包含各权利要求的各要素、各实施例的各要素等）的范围内，进一步根据其基本技术思想，能够实行实施例或实施例的变更、调整，另外，在本发明的权利要求的范围内能够进行各种公开要素的多种组合或选择。即，本发明当然包含本领域技术人员根据包含权利要求在内的全部公开内容、技术思想而能够得到的各种变形、修正。

[0072] 产业可利用性

[0073] 根据本发明，可以提供低温保存时不发生沉淀而可以保持连续的单相，具有低温稳定性的水溶性除草剂组合物。

Doc Code - SEQ.XML
WIPO Type Code -

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Application Number: 18435340

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METHODS FOR MAKING L-GLUFOSINATE

CROSS-REFERENCE TO PRIORITY APPLICATIONS

This application is a continuation of U.S. Application No. 18/179,136, filed March 6, 2023, which is a continuation of U.S. Application No. 17/530,018, filed November 18, 2021, which is a continuation of U.S. Application No. 16/997,133, filed August 19, 2020, now U.S. Patent No. 11,560,557, which is a continuation of U.S. Application No. 16/287,290, filed February 27, 2019, now U.S. Patent No. 10,781,465, which is a continuation of U.S. Application No. 15/787,448, filed October 18, 2017, now U.S. Patent No. 10,260,078, which is a division of U.S. Application No. 15/445,254, filed February 28, 2017, now U.S. Patent No. 9,834,802, which claims the benefit of U.S. Provisional Application No. 62/302,421, filed March 2, 2016; U.S. Provisional Application No. 62/336,989, filed May 16, 2016; and U.S. Provisional Application No. 62/413,240, filed October 26, 2016. The aforementioned applications are hereby incorporated herein by reference in their entireties.

INCORPORATION BY REFERENCE OF MATERIAL SUBMITTED ELECTRONICALLY

The Sequence Listing, which is a part of the present disclosure, is submitted concurrently with the specification as an .xml file. The name of the file containing the Sequence Listing is "M202647G_SubSeqListing.xml", which was created on April 17, 2024 and is 5,116 bytes in size. The subject matter of the Sequence Listing is incorporated herein in its entirety by reference.

FIELD

Described herein are methods for producing a single stereoisomer of glufosinate, particularly for the production of L-glufosinate.

BACKGROUND

The herbicide glufosinate is a non-selective, foliarly-applied herbicide considered to be one of the safest herbicides from a toxicological or environmental standpoint. Current commercial chemical synthesis methods for glufosinate yield a racemic mixture of L- and D-glufosinate (Duke *et al.* 2010 *Toxins* 2:1943-1962). However, L-glufosinate (also known as phosphinothricin or (S)-2-amino-4-(hydroxy(methyl)phosphonoyl)butanoic acid) is much more potent than D-glufosinate (Ruhland *et al.* (2002) *Environ. Biosafety Res.* 1:29–37).

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Therefore, methods are needed to produce only or primarily the active, L-glufosinate form. Previously, cost effective methods to generate pure L-glufosinate, or a mixture of D- and L-glufosinate enriched for L-glufosinate, have not been available. Described herein are new and cost-effective methods for the production of L-glufosinate.

5

SUMMARY

Compositions and methods for making L-glufosinate are provided. The first step of the process involves the oxidative deamination of D-glufosinate to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). The second step involves the specific amination of PPO to L-glufosinate, using an amine group from one or more amine donors. In some
10 embodiments, the method involves reacting D-glufosinate with a D-amino acid oxidase (DAAO) enzyme to form PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid); followed by aminating the PPO to L-glufosinate by a transaminase (TA) enzyme, using an amine group from one or more amine donors, wherein at least 70% of the D-glufosinate is eliminated and/or the yield of L-glufosinate is at least 85% of the input racemic glufosinate or at least 70 to 85% of the D-
15 glufosinate is converted to L-glufosinate. In some embodiments, unreacted amine donor from one reaction can be reused in further rounds of reaction. Optionally, the D-glufosinate is originally present (i.e., in the reacting step) in a racemic mixture of D- and L-glufosinate.

The DAAO enzyme must have an increased activity of about 3 $\mu\text{mol}/\text{min}\cdot\text{mg}$ or greater to drive the reaction. DAAO enzymes are available in the art and can be modified or mutated to
20 have the necessary increased activity needed to drive the process. In this manner, mutant or modified enzymes from *Rhodospiridium toruloides* (UniProt P80324), *Trigonopsis variabilis* (UniProt Q99042), *Neolentinus lepideus* (GenBank KZT28066.1), *Trichoderma reesei* (GenBank XP_006968548.1), or *Trichosporon oleaginosus* (KLT40252.1) can be used. In some embodiments, the DAAO enzyme is a mutant DAAO based on the sequence from
25 *Rhodospiridium toruloides*. While a number of mutations can be made and tested for the effect on activity, the mutant DAAO in some embodiments may comprise mutations at positions 54, 56, 58, 213, and/or 238. For example, the mutant DAAO can comprise one or more of the following mutations at position 54: N54C, N54L, N54T, or N54V. The mutant DAAO can optionally comprise the following mutation at position 56: T56M. The mutant DAAO can
30 optionally comprise one or more of the following mutations at position 58: F58A, F58G, F58H, F58K, F58N, F58Q, F58R, F58S, or F58T. Optionally, the mutant DAAO can comprise the

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following mutation at position 213: M213S. In some embodiments, the mutant DAAO can comprise one or more of the following combinations of mutations: F58K and M213S; N54T and T56M; N54V and F58Q; and/or N54V, F58Q, and M213S. In each case, the enzyme needs to have an activity of equal to or greater than about 3 $\mu\text{mol}/\text{min}\cdot\text{mg}$, greater than about 4
5 $\mu\text{mol}/\text{min}\cdot\text{mg}$, or higher. A wild type enzyme can be used in the methods of the invention as long as the enzyme has an activity level as set forth above.

The TA enzyme may be a gabT transaminase from *Escherichia coli* (UniProt P22256). Alternatively, the TA enzyme may be a transaminase with the sequence identified as SEQ ID NO: 1. The TA enzyme may also be selected on the basis of sequence similarity to SEQ ID NO:
10 1 and/or mutated to improve its activity in the desired reaction. Thus, sequences having 80%, 85%, 90%, 95% or greater sequence identity to SEQ ID NO: 1, based on the BLASTP method of alignment, and retain transaminase activity are encompassed by the present invention. Any DNA sequence encoding the enzyme sequence of SEQ ID NO: 1 or variants thereof are encompassed herein as well.

15 The reacting step and the aminating step can be performed in a single container or in separate containers. In one embodiment, all reagents are substantially added at the start of the reaction. Alternatively, the reagents for the reacting step and the reagents for the aminating step are added to the single container at different times.

Also described herein is a method for selectively controlling weeds in a field containing a
20 crop of planted seeds or crops that may optionally be resistant to glufosinate, comprising applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate to the field. Also described herein is a method for selectively controlling weeds in a field, controlling weeds in non-field areas, defoliating plants or crops, and/or desiccating crops before harvest, comprising applying an effective amount of a
25 composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate and more than 0.01% but less than 15% PPO to the field.

The details of one or more embodiments are set forth in the drawings and the description below. Other features, objects, and advantages will be apparent from the description and drawings, and from the claims.

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Some compositions of the invention comprise D-glufosinate, PPO, and L-glufosinate. In such compositions, L-glufosinate is present at a concentration equal to or greater than 80%, greater than 90%, greater than 95%, greater than 96%, greater than 97%, greater than 98%, or greater than 99% by weight of the composition, based on the combined weight of D-glufosinate, PPO, and L-glufosinate. Other compositions comprise L-glufosinate at concentrations equal to 80% or greater after isolation of the L-glufosinate from the present reaction mixture.

DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic of an exemplary conversion of D-glufosinate to L-glufosinate. The amine donor and keto acid product are examples and are not intended to be limiting.

Figure 2 is a graph showing concentrations of L-glufosinate (circles), D-glufosinate (triangles), and PPO (squares) during a one-step de-racemization by a N54T, T56M, F58K, and M213S mutant variant of *Rhodospiridium toruloides* DAAO and the *E. coli* gabT transaminase.

DETAILED DESCRIPTION

Compositions and methods for the production of L-glufosinate (also known as phosphinothricin or (S)-2-amino-4-(hydroxy(methyl)phosphonoyl)butanoic acid) are provided. The methods comprise a two-step process, which may optionally occur in a single vessel and nearly simultaneously. The first step involves the oxidative deamination of D-glufosinate to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). The second step involves the specific amination of PPO to L-glufosinate, using an amine group from one or more amine donors. By combining these two reactions, the proportion of L-glufosinate can be substantially increased in a racemic glufosinate mixture. Thus, provided herein are methods to obtain a composition consisting substantially of L-glufosinate. Since L-glufosinate is more potent than D-glufosinate, smaller amounts of the composition are needed to be effective as a herbicide.

In one embodiment, described herein, is a composition comprising a mixture of L-glufosinate, PPO, and D-glufosinate, where L-glufosinate is the predominant compound among the mixture of L-glufosinate, PPO, and D-glufosinate. Such composition can be used directly as a herbicide as PPO can contribute herbicidal activity (EP0030424). In other embodiments, L-glufosinate can be purified or substantially purified and used as a herbicide.

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Compositions of L-glufosinate may comprise D-glufosinate, PPO, and L-glufosinate. Optionally, the amount of L-glufosinate is 80% or greater, 85% or greater, 90% or greater, or about 95% or greater, 97% or greater, 98% or greater based on the combined weight of D-glufosinate, PPO, and L-glufosinate. Optionally, the amount of D-glufosinate is 10% or less, 5% or less, 2.5% or less, or 1% or less based on the combined weight of D-glufosinate, PPO, and L-glufosinate. Optionally, the amount of PPO is more than 1% but less than 20%, less than 15%, less than 10%, or less than 5% based on the weight of D-glufosinate, PPO, and L-glufosinate. These compositions can optionally occur as dried powders or dissolved in aqueous or non-aqueous carrier and additional chemical species can optionally be present. Optionally, the composition is prepared and used in an ex vivo environment.

It is also recognized that the L-glufosinate can be further isolated and used in formulations as a herbicide.

Also described herein are formulations. The formulations comprise L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; one or more additional components selected from the group consisting of sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; dipropylene glycol in an amount from 4-18% by weight of the formulation; and alkyl polysaccharide in an amount from 4-20% by weight of the formulation; and water as the balance of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 24.5% by weight of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the

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formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; alkylethersulfate, sodium salt in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1.0% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

While the methods can be used to produce a substantially purified L-glufosinate in a batch reaction, it is recognized that a continuous process can be used.

I. Methods of Synthesis

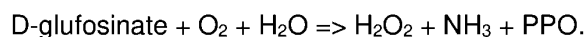
Methods for the conversion of D-glufosinate to L-glufosinate are provided. The methods described herein provide a means for converting a low cost feedstock of a racemic mixture of D- and L-glufosinate into a more valuable product that has been enriched for L-glufosinate. The methods for conversion includes two steps, which can occur in one or more separate containers. The first step is the oxidative deamination of D-glufosinate (which can be present in a racemic mixture of D- and L-glufosinate) to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). This step can be catalyzed by a D-amino acid oxidase (DAAO) enzyme, a D-amino acid dehydrogenase (DAAD) enzyme, or by chemical conversion. The second step is the specific amination of PPO to L-glufosinate, using an amine group from one or more amine donors. Such amine donors can be selected from glutamate, L-glutamate, lysine, alanine, isopropylamine, sec-butylamine, phenylethylamine and the like. This step can be catalyzed by a transaminase (TA) enzyme, an L-amino acid dehydrogenase (LAAD) enzyme, or by chemical conversion. Using the methods described herein, compositions of substantially purified L-glufosinate can be obtained.

Figure 1 sets forth an example of the conversion of D-glufosinate to L-glufosinate. As noted above, the method involves a two-step process. As illustrated, the first step is an oxidative deamination of D-glufosinate to PPO and the second step is an amination of PPO to L-glufosinate.

The first step, i.e., the oxidative deamination of D-glufosinate to PPO, can be catalyzed by several classes of enzymes or can occur non-enzymatically. Such enzymes include DAAO, DAAD, and D-amino acid dehydratase.

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In one embodiment a DAAO enzyme is used to catalyze the conversion of D-glufosinate to PPO. Such a reaction has the following stoichiometry:



Since the solubility of oxygen in aqueous reaction buffer is typically low compared to that
5 of glufosinate, for an efficient process, oxygen must be introduced throughout the time period of
the DAAO reaction. This is in contrast to, for example, the Hawkes reaction set forth in US
Patent Nos: 7,723,576; 7,939,709; 8,642,836; and 8,946,507 in which the reaction was
conducted in a sealed vessel. Initially, D-glufosinate is present at greater than 30 g/L up to as
much as 140 g/L. The initial oxygen level is typically impacted by the reaction temperature, but
10 is typically initially present at approximately 8 mg/L and is added throughout the reaction to
allow for sufficient oxygen for the reaction to continue apace. Water is typically, but not
obligately, present at greater than 500 g/L.

Several DAAO enzymes are known in the art and can be used in the methods described
herein, as long as they are capable of accepting D-glufosinate as a substrate and provide an
15 activity sufficient to level to drive the reaction. The DAAO enzymes useful in the methods of the
invention have an activity of equal to or greater than about 3 umol/min*mg, greater than about 4
umol/min*mg, or higher. A wild type enzyme can be used in the methods of the invention as
long as the enzyme has an activity level as set forth above. Such DAAO enzymes that can be
used in the method include those from *Rhodospiridium toruloides*, *Trigonopsis variabilis*,
20 *Fusarium sp*, *Candida sp*, *Schizosaccharomyces sp*, *Verticillium sp*, *Neolentinus lepideus*,
Trichoderma reesei, *Trichosporon oleaginosus*, and the like that have been modified to increase
activity. Any DAAO enzyme can be used as a starting enzyme including those having
sequences corresponding to Swissprot accession numbers P80324, Q99042, P00371, and
P24552 or SPTREMBL numbers Q9HGY3 and Q9Y7N4 or GenBank numbers KZT28066.1,
25 XP_006968548.1, and KLT40252.1. The DNA sequences which encode the DAAO may be
selected from sequences set forth in EMBL accessions A56901, RGU60066, Z50019, SSDA04,
D00809, AB042032, RCDAAOX, A81420, and SPCC1450, or may be codon optimized from the
protein sequences indicated above for optimal expression in the chosen expression host(s).
U.S. Patent No. 8,227,228 describes DAAO enzymes from *Candida intermedia*. Such
30 sequences are herein incorporated by reference. These enzymes can be modified for
increased activity and used in the methods of the invention.

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Additional DAAO enzymes can be identified in a variety of ways, including sequence similarity and functional screens. The DAAO enzyme may be a mutant DAAO enzyme that is capable of accepting D-glufosinate as a substrate. In Hawkes *et al.*, *supra*, a mutant DAAO based on the sequence from *Rhodospiridium toruloides* (consisting of the F58K and M213S mutations) has been shown to accept D-glufosinate as a substrate (Hawkes *et al.* (2011) *Plant Biotechnol J.* 9(3):301-14). Other DAAO enzymes can be similarly modified to accept D-glufosinate and have greater activity. i.e., the activity needed to drive the method of the invention. In the same manner, known DAAO enzymes may be improved by mutagenesis, and/or novel DAAO enzymes could be identified.

In some embodiments, mutant enzymes can be made and tested in the methods described herein. Mutant DAAO enzymes (e.g., from *Rhodotorula gracilis*) can include one mutation, two mutations, three mutations, or more than three mutations (e.g., four mutations, five mutations, six mutations, seven mutations, eight mutations, nine mutations, or ten mutations or more) at positions in the mutant sequence as compared to the wild type sequence. The mutant DAAO can optionally comprise mutations at positions 54, 56, 58, 213, and/or 238. In some embodiments, such mutants can comprise amino acid substitutions at positions 54 and 56 when compared with the wild type sequence. In other embodiments, such mutants can comprise amino acid substitutions at positions 54 and 58 when compared to the wild type sequence. In other embodiments, such mutants can include amino acid substitutions at positions 54, 213, and 238 when compared with the wild type sequence.

Optionally, at position 54, the wild type asparagine may be replaced by Ala, Cys, Gly, Ile, Ser, Leu, or, more preferably, Thr or Val. For example, the mutant DAAO can comprise one of the following mutations at position 54: N54C, N54L, N54T, or N54V.

Optionally, at position 56, the wild type threonine can be replaced by Ala, Cys, Gly, Ile, Asn, Arg, Ser, Thr, Met, or Val. See, U.S. Patent No. 7,939,709, which is incorporated herein by reference. For example, the mutant DAAO can comprise the T56M mutation.

Additionally, at position 58, the wild type Phe can be replaced by Lys, Arg, Gln, Thr, Gly, Ser, Ala, Arg, Asn, or His. The mutant DAAO can optionally comprise one of the following mutations at position 58: F58A, F58G, F58H, F58K, F58N, F58Q, F58R, F58S, or F58T. In some embodiments, the mutant DAAO does not include a mutation at position 58.

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Optionally, at position 213, the wild type methionine is replaced by Arg, Lys, Ser, Cys, Asn, or Ala. In some examples, the mutant DAAO can comprise the mutation M213S.

Optionally, at position 238, the wild type tyrosine is replaced by His, Ser, Gys, Asn, or Ala.

5 In some embodiments, the mutant DAAO can comprise one or more of the following combinations of mutations: F58K and M213S; N54T and T56M; N54V and F58Q; N54C and F58H; N54T and F58T; N54T and F58G; N54T and F58Q; N54T and F58A; N54L and F58R; N54V and F58R; N54V and F58N; and/or N54V, F58Q, and M213S.

10 In one embodiment, the mutant DAAO comprises mutations in other DAAO enzymes in positions equivalent to positions 54, 56, 58, 213, and/or 238 of *Rhodospiridium toruloides* DAAO or *Trigonopsis variabilis* DAAO.

Other suitable D amino acid oxidases may be obtained preferably from fungal sources. Such DAAO enzymes can be identified and tested for use in the methods of the invention. To determine if the enzyme will accept D-glufosinate as a substrate, an oxygen electrode assay
15 (Hawkes, 2011, *supra*), colorimetric assay (Berneman A, Alves-Ferreira M, Coatnoan N, Chamond N, Minoprio P (2010) Medium/High Throughput D-Amino Acid Oxidase Colorimetric Method for Determination of D-Amino Acids. Application for Amino Acid Racemases. *J Microbial Biochem Technol* 2: 139-146), and/or direct measurement (via high performance liquid chromatography (HPLC), liquid chromatography mass spectrometry (LC-MS), or similar) of
20 product formation can be employed.

The reaction catalyzed by the DAAO enzyme requires oxygen. In some embodiments, oxygen, oxygen enriched air, an oxygen enriched gas stream, or air, is introduced to the reaction, either in the head space or by sparging gas through the reaction vessel, intermittently or continuously, to enhance the rate of reaction. Additionally, in other embodiments, optionally
25 combined with sparging gas through the reaction vessel, a pressurized reactor may be used. That is, the reactor may be sealed and allowed to consume O₂. Using a sealed chamber would limit vapor emissions.

When a DAAO enzyme catalyzes the conversion of D-glufosinate to PPO, hydrogen peroxide (H₂O₂) evolves. This may be damaging to enzymes and other components of the

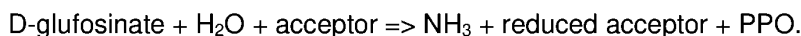
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biotransformation (e.g., products and/or substrates). Therefore, in one embodiment, an enzyme, such as catalase, can be used in addition to the DAAO enzyme to catalyze the elimination of hydrogen peroxide. Catalase catalyzes the decomposition of hydrogen peroxide with the following stoichiometry:



In some embodiments, hydrogen peroxide can be eliminated using catalyzed and non-catalyzed decomposition reactions. For example, hydrogen peroxide can be eliminated by a non-catalyzed decomposition reaction using increased heat and/or pH. Hydrogen peroxide can also be eliminated by a catalyzed decomposition reaction using, for example, transition metals
10 and other agents, such as potassium iodide. In addition to eliminating hydrogen peroxide, the use of catalase also produces oxygen (O_2). The production of oxygen by catalase can aid in facilitating the conversion of D-glufosinate to PPO using the DAAO enzyme, as DAAO requires oxygen to function.

Other enzymes can be used to catalyze the conversion of D-glufosinate to PPO. For
15 example, a DAAD enzyme that accepts D-glufosinate as a substrate can be used with the following stoichiometry:



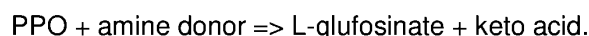
It is recognized that in methods where a DAAD is used, the DAAD catalyzed reaction can include redox cofactor recycling. This involves oxidizing the reduced acceptor so that it can
20 accept more electrons from D-glufosinate.

In one embodiment, chemical oxidative deamination, wherein an intermediate α -keto acid is produced from the parent amino acid, can be used in the methods described herein to convert D-glufosinate to L-glufosinate. Chemical oxidative deamination involves the conversion of an amine group to a keto group with concomitant release of ammonia typically using metal
25 ions such as those of copper or cobalt in an aqueous solution at temperatures between room temperature and the boiling point of the solution and at a pH in the range of about 4 – about 10. See, for example, Ikawa and Snell (1954) *J. Am. Chem. Soc.* 76 (19): 4900–4902, herein incorporated by reference.

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The substantially complete (greater than 70%, greater than 75%, greater than 80%, greater than 85%, greater than 90%, or greater than 95%) conversion of D-glufosinate to PPO can occur within 24 hours, within 18 hours, within 12 hours, within 8 hours or less.

The second step of the method described herein involves the conversion of PPO to L-glufosinate using a transaminase (TA) enzyme, an L-amino acid dehydrogenase (LAAD) enzyme, or by chemical conversion. In one embodiment, the method is a reaction catalyzed by a TA. A TA with the required stereospecificity that accepts PPO as a substrate catalyzes the amination of PPO to L-glufosinate with the following stoichiometry:



If the reaction is conducted as a two stage process where the D-glufosinate is substantially converted to PPO in the absence of amine donor and/or transaminase, starting amounts of PPO in the second stage typically range from 10g/L to 140g/L; 20g/L to 140g/L; or from 30g/L to 140g/L. If the reaction is conducted in a single stage process, the starting amounts of PPO are typically less than 1 g/L and the highest levels of PPO during the reaction are typically less than 25 g/L. The amine donor is initially present at between 1 and 50 fold molar excess over the starting amount of racemic glufosinate.

TAs useful in the methods described herein include the gabT transaminase from *Escherichia coli* (UniProt P22256; identified herein as SEQ ID NO: 3), which has been shown to catalyze the desired reaction with PPO as a substrate (Bartsch *et al.* (1990) *Appl Environ Microbiol.* 56(1):7-12). Another enzyme has been evolved to catalyze the desired reaction at a higher rate using isopropylamine as an amine donor (Bhatia *et al.* (2004) *Peptide Revolution: Genomics, Proteomics & Therapeutics*, Proceedings of the Eighteenth American Peptide Symposium, Ed. Michael Chorev and Tomi K. Sawyer, July 19-23, 2003, pp. 47-48). A transaminase with the amino acid sequence of SEQ ID NO: 1 also catalyzes the desired reaction with PPO and isopropylamine as the substrate (Example 11). Additionally, TA enzymes from numerous microorganisms, such as *Streptomyces hygroscopicus*, *Streptomyces viridochromogenes*, *Candida albicans*, and others can be used in the practice of the methods described herein. In particular, see, for example, EP0249188, and U.S. Patent No. 5,162,212, incorporated herein by reference. Where desired, the enzymes can be evolved by mutagenesis to increase their activities. Mutant TA enzymes can be selected for desired activity by the

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assays outlined in Schulz *et al.*, Appl Environ Microbiol. (1990) Jan. 56(1):1-6, and/or by direct measurement of the products by HPLC, LC-MS, or similar products.

Additional TA enzymes for use in the methods can be identified by screening collections of TAs, such as those sold by Prozomix Limited (Northumberland, United Kingdom),
5 SyncoZymes (Shanghai, China), Evocatal (Monheim am Rhein, Germany), Codexis (Redwood City, CA), or Abcam (Cambridge, United Kingdom) for the desired activity. Alternatively, sequence similarity can be used to identify novel TA enzymes. Finally, TA enzymes can also be identified from organisms capable of catalyzing the desired reaction.

The selection of an appropriate amine donor is important for an economical conversion
10 of D-glufosinate to L-glufosinate. A variety of issues may be considered, including the cost of the donor, equilibrium thermodynamics, potential recovery of the donor, separation of the keto acid product from the desired L-glufosinate, and others. Consequently, TA enzymes that accept several different amine donors can be used, including low cost amine donors such as L-aspartate or racemic aspartate, L-glutamate or racemic glutamate, L-alanine or racemic alanine,
15 L-phenylethylamine or racemic phenylalanine, L-glycine or racemic glycine, L-lysine or racemic lysine, L-valine or racemic valine, L-serine or racemic serine, L-glutamine or racemic glutamine, isopropylamine, sec-butylamine, ethanolamine, 2-aminobutyric acid, and diaminopropionic acid. In some embodiments, the amine donor is not aspartate or aspartic acid (e.g., L-aspartic acid, D-aspartic acid, or racemic D,L-aspartic acid).

20 In embodiments where the amino donor is glutamate, the keto acid co-product that results from the transamination reaction is α -ketoglutarate (which is also referred to as α -ketoglutaric acid or α -KG). The α -ketoglutarate can be isolated and/or purified using methods known to those of skill in art, such as in EP Patent No. 0073711, CN Patent No. 10519873, CN Patent No. 105177065, CN Patent No. 104529755, and Zhan et al., *Shipin Yu Shengwu Jishu Xuebao*, 32(10): 1043-1048 (2013), each of which are incorporated herein by reference in their
25 entireties. The produced and isolated α -ketoglutarate can be used in a variety of applications, including in synthesizing pharmaceutical agents, food additives, and biomaterials. Optionally, the α -ketoglutarate can be chemically converted to either racemic glutamate or L-glutamate, optionally for reuse in the reaction.

30 Chemical reductive amination involves the conversion of a keto group to an amine group via an imine compound typically by treatment of the keto compound with a suitable amine in an

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organic solution. Suitable amines include, for example, methylamine or ammonia. Suitable organic solvents for use in the organic solution include tetrahydrofuran, ethanol, or dichloromethane (DCM). The reductive amination can be performed at temperatures between room temperature and the boiling point of the solution. The produced imine can then be

5 reduced using a reducing agent in an organic solution. Suitable reducing agents include, for example, NaBH_4 , $\text{NaHB}(\text{OAc})_3$, or $\text{Na}(\text{CN})\text{BH}_3$. Suitable organic solvents for use in the organic solution include tetrahydrofuran, ethanol, or DCM. The reduction reaction can be performed at temperatures between 0°C and the boiling point of the solution. Those skilled in the art will know that the process may be done in "one-pot" or in multiple containers, i.e., in separate

10 transformations. Additionally, those skilled in the art will recognize that the described procedure will provide racemic amino material where possible. The use of a chiral reducing agent such as $\text{RuCl}_2[(S)\text{-BINAP}]$ and hydrogen gas or latent source of hydrogen gas, or achiral hydride based reducing agents in the presence chiral ligands such as $(S)\text{-}$ or $(R)\text{-VAPOL}$ in a ratio between 1:1 to 1:0.05 can produce enantiomerically pure and/or enriched amino material. See, for example,

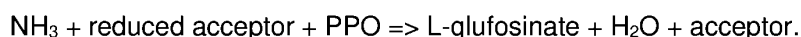
15 Mignonac (1921) *Compt. Rend.* 172:223 and G. Li, Y. Liang, J. C. Antilla (2007) *J. Am. Chem. Soc.*, 129:5830-5831.

A wild type TA that accepts a desired amine donor can be identified, or a TA that does not normally accept a desired amine donor can be evolved to accept the desired substrate. Optionally, the transaminase is not an aspartate transaminase. Optionally, the transaminase is

20 not 4-amino-butyrate: 2-ketoglutarate transaminase. In some embodiments, the transaminase is not a combination enzyme system that includes a PPT-specific transaminase and glutamate:oxaloacetate transaminase.

Other enzymes for use to catalyze the conversion of PPO to L-glufosinate include LAAD enzymes or imine reductases that accept PPO as a substrate. Such LAAD enzymes use the

25 following stoichiometry:



LAAD catalyzed reactions can include redox cofactor recycling, which involves reducing the oxidized acceptor so that it can donate more electrons to PPO.

Chemical reductive amination, wherein an amino group is produced from the parent

30 keto-compound, can also be used to produce glufosinate, in the case where no chiral reductant

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or ligand is used, or L-glufosinate, in the case where a chiral reductant or ligand is used. Chemical reductive amination can be affected as described above.

The substantially complete conversion of PPO to L-glufosinate may occur within 24 hours, within 18 hours, within 12 hours, within 8 hours, or within 4 hours. Substantially
5 complete, in this context, means that the conversion of PPO to L-glufosinate is greater than about 70%, greater than about 75%, greater than about 80%, greater than about 85%, greater than about 90%, greater than about 95%, greater than about 98%, or greater than about 99%.

If the reaction occurs in a single container or vessel, the TA enzyme can be added with the DAAO enzyme or added at a later time, e.g., after the DAAO enzyme has been allowed to
10 catalyze some or substantially all of the oxidative deamination.

Enzymes can be added to the reaction by a number of methods. One approach is to express the enzyme(s) in microorganism(s) such as *E. coli*, *S. cerevisiae*, *P. pastoris*, and others, and to add the whole cells to the reactions as whole cell biocatalysts. Another approach is to express the enzyme(s), lyse the microorganisms, and add the cell lysate. Yet another
15 approach is to purify, or partially purify, the enzyme(s) from a lysate and add pure or partially pure enzyme(s) to the reaction. If multiple enzymes are required for a reaction, the enzymes can be expressed in one or several microorganisms, including expressing all enzymes within a single microorganism.

A further approach, which can be combined with the above approaches, is to immobilize
20 enzyme(s) to a support (exemplary strategies are outlined in Datta *et al.* (2013) 3 Biotech. Feb; 3(1): 1–9). As outlined in Datta *et al.*, and not intending to be limiting, enzymes, either singly or in combination, can, for example, be adsorbed to, or covalently or non-covalently attached to, or entrapped within, natural or synthetic polymers or inorganic supports, including aggregates of the enzyme(s) themselves. Once immobilized, the enzyme(s) and support can be dispersed into
25 bulk solution or packed into beds, columns, or any number of similar approaches to interacting reaction solution with the enzymes. Since aeration is important for the DAAO reaction envisioned here, bubble columns or similar may be used for enzyme immobilization. As examples, reaction mixture can be flowed through a column of immobilized enzymes (flow reaction), added to a fixed bed or column of immobilized enzymes, allowed to react, and either
30 removed from the bottom or top of the reaction vessel (plug flow), or added to dispersed immobilized enzymes and allowed to react then the immobilized enzymes removed by filtration,

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centrifugation, or similar (batch). Thus, any method for immobilization of the enzymes may be employed in the methods of the invention.

The DAAO, TA, and/or other reactions can occur in a buffer. Exemplary buffers commonly used in biotransformation reactions include Tris, phosphate, or any of Good's buffers, such as 2-(N-morpholino)ethanesulfonic acid (MES); N-(2-Acetamido)iminodiacetic acid (ADA); piperazine-N,N'-bis(2-ethanesulfonic acid) (PIPES); N-(2-Acetamido)-2-aminoethanesulfonic acid (ACES); β -Hydroxy-4-morpholinepropanesulfonic acid (MOPSO); choline chloride; 3-(N-morpholino)propanesulfonic acid (MOPS); N,N-Bis(2-hydroxyethyl)-2-aminoethanesulfonic acid (BES); 2-[[1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl]amino]ethanesulfonic acid (TES); 4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid (HEPES); 3-(Bis(2-hydroxyethyl)amino)-2-hydroxypropane-1-sulfonic acid (DIPSO); acetamidoglycine, 3-(N-Tris(hydroxymethyl)methylamino)-2-hydroxypropanesulfonic acid (TAPSO); Piperazine-N,N'-bis(2-hydroxypropanesulfonic acid) (POPSO); 4-(2-Hydroxyethyl)piperazine-1-(2-hydroxypropanesulfonic acid) (HEPPSO); 3-[4-(2-Hydroxyethyl)-1-piperazinyl]propanesulfonic acid (HEPPS); tricine; glycine; bicine; or 3-[[1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl]amino]propane-1-sulfonic acid (TAPS). Additional exemplary buffer recipes can be found in Whittall, J. and Sutton, P. W. (eds) (2012) Front Matter, in Practical Methods for Biocatalysis and Biotransformations 2, John Wiley & Sons, Ltd, Chichester, UK. In some embodiments, ammonium can act as a buffer. One or more organic solvents can also be added to the reaction.

Surprisingly, the DAAO, TA, and/or other reactions can occur with no or low levels (less than 1 mM) of buffer added (other than ammonium that may optionally be present due to addition of racemic glufosinate ammonium). In particular, immobilized DAAO and TA may be stable and active in the presence of less than 1 mM phosphate buffer and with no other buffer except any ammonium present due to the addition of racemic glufosinate ammonium.

The racemic glufosinate starting material can be provided in a number of forms. Various salts of racemic glufosinate, such as ammonium and hydrochloride, or the zwitterion, can be used. The racemic glufosinate may be in the form of a solid powder (such as a powder of greater than 80%, 85%, 90%, or 95% purity) or an aqueous solution (such as a roughly 50% solution of racemic glufosinate).

In some embodiments, the reaction occurs within a defined pH range, which can be between pH 4 to pH 10 (e.g., between pH 6 and pH 9, such as approximately pH 7.5 to pH 8).

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In some embodiments, the reaction occurs at a defined temperature. The temperature can be kept at a point between room temperature and the boiling point of the solvent, most typically between room temperature and 50 °C.

As indicated, the methods described herein provide a composition of substantially pure L-glufosinate (rather than a racemic mixture of L-glufosinate and D-glufosinate). Substantially pure L-glufosinate means that greater than about 70%, greater than about 75%, greater than about 80%, greater than about 85%, greater than about 90%, greater than about 95%, greater than about 96%, greater than about 97%, greater than about 98%, or greater than about 99% of the D-glufosinate has been converted to L-glufosinate resulting in a composition having greater than about 80%, greater than about 85%, greater than about 90%, greater than about 95%, greater than about 96%, greater than about 97%, greater than about 98%, or greater than about 99% L-glufosinate compared to the sum of the D-glufosinate and the L-glufosinate present in the composition.

In one embodiment, the L-glufosinate is not isolated from the biotransformation mixture and a composition comprising D-glufosinate, PPO, and L-glufosinate is obtained. This composition will contain at least 80% L-glufosinate by weight of the sum of L-glufosinate, D-glufosinate, and PPO, at least 90% L-glufosinate by weight of the sum of the components. This composition may be used directly as a herbicidal composition or as an ingredient in a formulated herbicidal product.

Alternatively, some or all of the components other than L-glufosinate can be removed from the biotransformation mixture, the mixture optionally concentrated, and then the mixture can be used directly (and/or with the addition of various adjuvants) for the prevention or control of weeds. The biotransformation mixture, in some instances, can be used directly (and/or with the addition of various adjuvants) for the prevention or control of weeds.

Additional steps to further purify the L-glufosinate can be added. Such further purification and isolation methods include ion exchange, extraction, salt formation, crystallization and filtration; each may be used multiple times or in suitable combination. Enzymes can be removed by simple filtration if supported, or if free in solution by the use of ultrafiltration, the use of absorbants like celite, cellulose or carbon, or denaturation via various techniques known to those skilled in the art.

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Ion exchange processes effect separation by selective adsorption of solutes onto resins chosen for this purpose. Because products and impurities must be dissolved in a single solution prior to adsorption, concentration of the purified product stream by evaporation or distillation prior to isolation is usually required. Examples of the use of ion exchange for purification are
5 described by Schultz et al., and in EP0249188(A2).

Purification may be achieved by the formation of an insoluble salt of L-glufosinate by the addition of a suitable acid, including hydrochloric acid, sulfuric acid, phosphoric acid, nitric acid, acetic acid and the like. Similarly, the purification may be achieved by the addition of a suitable base to form an insoluble salt. Useful bases include hydroxides, carbonates, sulfates and
10 phosphates of alkali metals or hydroxides, carbonates, sulfates and phosphates of alkali earth metals. Other bases which contain nitrogen may be used, including ammonia, hydroxylamine, isopropylamine, triethylamine, tributylamine, pyridine, 2-picoline, 3-picoline, 4-picoline, 2,4-lutidine, 2,6-lutidine, morpholine, N-methymorpholine, 1,8-diazabicyclo[5.4.0]undec-7-ene, and dimethylethanolamine. It may be advantageous to concentrate the mixture or to add a solvent
15 (or both) to maximize yield and optimize purity of the desired salt. Solvents suitable for this purpose include those in which the solubility of the desired salt is very low (such solvents are often called "anti-solvents"). Salts of L-glufosinate can be transformed into forms of glufosinate suitable for formulation by standard methods known to those skilled in the art. Alternatively, the L-glufosinate can be isolated as a zwitterion.

US 9,255,115 B2 describes how the hydrochloric acid salt of L-glufosinate can be
20 converted to the zwitterionic form with a base such as sodium hydroxide or sodium methoxide and then crystallized from aqueous alcohol solvent to afford L-glufosinate in relatively high purity. This method has the advantage of producing crystalline L-glufosinate that is not hygroscopic and therefore maintains a higher purity compared to amorphous L-glufosinate when
25 exposed to humidity over time.

Other salts of L-glufosinate are known in the art. US 5,767,309 and US 5,869,668 teach the use of chiral alkaloid bases to form diastereomeric salts with racemic glufosinate. Purification is achieved because the salt of L-glufosinate precipitates from solution in much larger quantity than the corresponding salt of D-glufosinate. Therefore this method could be
30 used with the present invention to obtain L-glufosinate with high enantiomeric excess, if desired.

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Optionally, purification may be achieved by first crystallizing one or more impurities, removing the impurities by filtration and then further purifying L-glufosinate from the resulting filtrate by forming a salt as previously described. This is advantageous if unreacted amine donor can be partially or completely isolated and used in subsequent reactions. Similarly, unreacted PPO that is partially or completely isolated may be recycled for use in subsequent reactions.

Extraction may be used to purify the product. DE 3920570 C2 describes a process in which excess glutamic acid (used as the amine donor) is precipitated by adjusting the solution pH to 3.7 to 4.2 with sulfuric acid. After filtering the glutamic acid, the filtrate pH is lowered to 1-2 whereupon other impurities are extracted into a solvent. After extraction and concentration, ammonia is added to the aqueous solution to a pH of 5-7 whereupon ammonium sulfate precipitates. The ammonium sulfate is removed by filtration and the resulting filtrate is concentrated to afford the ammonium salt of L-glufosinate.

Isolation of L-glufosinate or its salts may be desirable, for example, for the purpose of shipping solids to the location of formulation or use. Typical industrial methods of isolation may be used, for example, a filtration, centrifugation, etc. Isolated product often requires the removal of water, volatile impurities and solvents (if present) and typical industrial drying equipment may be used for this purpose. Examples of such equipment include ovens, rotating drum dryers, agitated dryers, etc. In some cases, it may be advantageous to use a spray dryer.

It is not necessary to produce a solid product after purification. This may be advantageous if the formulation of L-glufosinate is to occur at the same site used for L-glufosinate production. L-glufosinate and many of its salts are readily soluble in water, and water is a convenient liquid to use for formulating products. For example, the amine donor is isolated by filtration and the resulting filtrate is concentrated by distillation. The pH of the filtrate may be adjusted to a desirable value and the resulting solution may be used as is or blended with formulation ingredients. In another example, a slurry of L-glufosinate or one of its salts may be prepared as described above and isolated by filtration. The solid could be dissolved directly on the filter by adding water or a suitable solvent to obtain a solution of L-glufosinate.

SUBSTITUTE SPECIFICATION - CLEAN**II. Compositions**

Also described herein are compositions comprising the reaction products described above. In some embodiments, the composition substantially includes L-glufosinate and acceptable cationic or anionic salt forms such as the hydrochloride, ammonium, or isopropylammonium salts. In some embodiments, the composition comprises a mixture of L-glufosinate, PPO, and D-glufosinate.

Optionally, L-glufosinate is the predominant compound among L-glufosinate, PPO, and D-glufosinate. For example, L-glufosinate can be present in the composition in an amount of at least 80% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 85 % by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 90% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 95% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 96% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 97% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 98% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, or at least 99% by weight of the sum of L-glufosinate, PPO, and D-glufosinate.

The composition can include PPO in an amount up to 20% by weight of the sum of L-glufosinate, PPO, and D-glufosinate. Optionally, the composition includes from 0.001% to 20% PPO (e.g., from 0.05% to 15% or from more than 0.01% to less than 5% PPO). For example, the composition can include PPO in an amount of less than 20%, less than 19%, less than 18%, less than 17%, less than 16%, less than 15%, less than 14%, less than 13%, less than 12%, less than 11%, less than 10%, less than 9%, less than 8%, less than 7%, less than 6%, less than 5%, less than 4%, less than 3%, less than 2%, less than 1%, less than 0.5%, less than 0.1%, or less than 0.01% by weight of the sum of the masses of L-glufosinate, PPO, and D-glufosinate.

D-Glufosinate can be present in the composition in an amount of 15% or less by weight of the sum of L-glufosinate, PPO, and D-glufosinate. For example, D-glufosinate can be present in an amount of 14 % or less, 13 % or less, 12 % or less, 11 % or less, 10 % or less, 9 % or less, 8 % or less, 7 % or less, 6 % or less, 5 % or less, 4 % or less, 3 % or less, 2 % or less, 1 % or less, or 0.5 % or less by weight of the sum of L-glufosinate, PPO, and D-glufosinate.

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In some embodiments, the composition can contain small amounts (e.g., about 10% or less, about 8% or less, about 5% or less, about 2% or less, or about 1% or less by weight of the composition) of D-glufosinate. In some embodiments, the composition can contain small amounts (e.g., about 15% or less, about 10% or less, about 8% or less, about 5% or less, about 2% or less, or about 1% or less by weight of the composition) of PPO.

The compositions described herein are useful for application to a field of crop plants for the prevention or control of weeds. The composition may be formulated as a liquid for spraying on a field. The L-glufosinate is provided in the composition in effective amounts. As used herein, effective amount means from about 10 grams active ingredient per hectare to about 1,500 grams active ingredient per hectare, e.g., from about 50 grams to about 400 grams or from about 100 grams to about 350 grams. In some embodiments, the active ingredient is L-glufosinate. For example, the amount of L-glufosinate in the composition can be about 10 grams, about 50 grams, about 100 grams, about 150 grams, about 200 grams, about 250 grams, about 300 grams, about 350 grams, about 400 grams, about 500 grams, about 550 grams, about 600 grams, about 650 grams, about 700 grams, about 750 grams, about 800 grams, about 850 grams, about 900 grams, about 950 grams, about 1,000 grams, about 1,050 grams, about 1,100 grams, about 1,150 grams, about 1,200 grams, about 1,250 grams, about 1,300 grams, about 1,350 grams, about 1,400 grams, about 1,450 grams, or about 1,500 grams L-glufosinate per hectare.

The herbicidal compositions (including concentrates which require dilution prior to application to the plants) described herein contain L-glufosinate (i.e., the active ingredient), optionally some residual D-glufosinate and/or PPO, and one or more adjuvant components in liquid or solid form.

The compositions are prepared by admixing the active ingredient with one or more adjuvants, such as diluents, extenders, carriers, surfactants, organic solvents, humectants, or conditioning agents, to provide a composition in the form of a finely-divided particulate solid, pellet, solution, dispersion, or emulsion. Thus, the active ingredient can be used with an adjuvant, such as a finely-divided solid, a liquid of organic origin, water, a wetting agent, a dispersing agent, an emulsifying agent, or any suitable combination of these. From the viewpoint of economy and convenience, water is the preferred diluent. However, not all the

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compounds are resistant to hydrolysis and in some cases this may dictate the use of non-aqueous solvent media, as understood by those of skill in the art.

Optionally, one or more additional components can be added to the composition to produce a formulated herbicidal composition. Such formulated compositions can include L-glufosinate, carriers (e.g., diluents and/or solvents), and other components. The formulated composition includes an effective amount of L-glufosinate. Optionally, the L-glufosinate can be present in the form of L-glufosinate ammonium. The L-glufosinate ammonium can be present in an amount ranging from 10 % to 30 % by weight of the formulated composition. For example, the L-glufosinate ammonium can be present in an amount of 10 %, 12 %, 14 %, 16 %, 18 %, 20 %, 22 %, 24 %, 26 %, 28 %, or 30 % by weight of the formulated composition. Optionally, the L-glufosinate ammonium is present in an amount of 12.25 % or of 24.5%

In some examples, the formulated composition can include one or more surfactants. A suitable surfactant for use in the formulated composition includes sodium alkyl ether sulfate. The surfactant can be present in an amount from 10 % to 40 % by weight of the formulated composition. For example, the surfactant can be present in an amount of 10 %, 12 %, 14 %, 16 %, 18 %, 20 %, 22 %, 24 %, 26 %, 28 %, 30 %, 32 %, 34 %, 36 %, 38 %, or 40 % by weight of the formulated composition. Optionally, the sodium alkyl ether sulfate is present in an amount of 11.05%, 15.8 %, 22.1%, or 31.6 %.

The formulated composition can optionally include one or more solvents (e.g., organic solvents). Optionally, the solvent can be 1-methoxy-2-propanol, dipropylene glycol, ethylene glycol, and mixtures thereof. The one or more solvents can be present in an amount ranging from 0.5 % to 20 % by weight of the formulated composition. For example, the total amount of solvents in the composition can be present in an amount of 0.5% to 18 %, 5 % to 15 %, or 7.5 % to 10 % by weight of the formulated composition.

Optionally, the solvent includes a combination of two solvents. For example, the solvents in the formulation can include 1-methoxy-2-propanol and dipropylene glycol. The 1-methoxy-2-propanol can be present, for example, in an amount of 0.5 % to 2 % by weight of the formulated composition. For example, the 1-methoxy-2-propanol can be present in the amount of 0.5 %, 0.6 %, 0.7 %, 0.8 %, 0.9 %, 1.0 %, 1.1 %, 1.2 %, 1.3 %, 1.4 %, 1.5 %, 1.6 %, 1.7 %, 1.8 %, 1.9 %, or 2.0 % by weight of the formulated composition. Optionally, the 1-methoxy-2-propanol is present in an amount of 0.5 % or 1.0 % by weight of the formulated composition.

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The dipropylene glycol can be present in an amount of from 4 % to 18 % by weight of the formulated composition. For example, the dipropylene glycol can be present in an amount of 4 %, 6 %, 8 %, 10 %, 12 %, 14 %, 16 %, or 18 % by weight of the formulated composition.

5 Optionally, the dipropylene glycol is present in an amount of 4.3 % or 8.6 % by weight of the formulated composition.

 The formulated composition can also include one or more polysaccharide humectants. Examples of suitable polysaccharide humectants include, for example, alkyl polysaccharides, pentoses, high fructose corn syrup, sorbitol, and molasses. The polysaccharide humectant, such as alkyl polysaccharide, can be present in the formulated composition in an amount
10 ranging from 4 % to 20 % by weight of the formulated composition. For example, the total amount of polysaccharide humectant in the composition can be present in an amount of 4 % to 18 %, 4.5 % to 15 %, or 5 % to 10 % by weight of the formulated composition. In some examples, the total amount of polysaccharide humectant, such as the alkyl polysaccharide, present in the formulated composition can be 4 %, 5 %, 6 %, 7 %, 8 %, 9 %, 10 %, 11 %, 12 %, 15
15 13 %, 14 %, 15 %, 16 %, 17 %, or 18 %. Optionally, the alkyl polysaccharide can be present in an amount of 3.2%, 4.9 %, 6.2%, or 9.8 %.

 A diluent can also be included in the formulated composition. Suitable diluents include water and other aqueous components. Optionally, the diluents are present in an amount necessary to produce compositions ready for packaging or for use.

20 In one example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of
25 36.75% by weight of the formulation.

 In another example, the formulated composition includes L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl
30 polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.

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5 In another example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation.

10 In another example, the formulated composition includes L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

15 In another example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 58.45% by weight of the formulation.

20 In another example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 11.05% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; alkyl polysaccharide in an amount of 3.1% by weight of the formulation; and water in an amount of 73.1% by weight of the formulation.

Further components suitable for use in the formulated compositions provided herein are described in U.S. Patent Nos. 4,692,181 and 5,258,358, both of which are incorporated by reference herein in their entireties.

25 The herbicidal compositions described herein, particularly liquids and soluble powders, can contain as further adjuvant components one or more surface-active agents in amounts sufficient to render a given composition readily dispersible in water or in oil. The incorporation of a surface-active agent into the compositions greatly enhances their efficacy. Surface-active agent, as used herein, includes wetting agents, dispersing agents, suspending agents, and

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emulsifying agents are included therein. Anionic, cationic, and non-ionic agents can be used with equal facility.

Suitable wetting agents include alkyl benzene and alkyl naphthalene sulfonates, sulfated fatty alcohols, amines or acid amides, long chain acid esters of sodium isothionate, esters of sodium sulfosuccinate, sulfated or sulfonated fatty acid esters petroleum sulfonates, sulfonated vegetable oils, ditertiary acetylenic glycols, polyoxyethylene derivatives of alkylphenols (particularly isooctylphenol and nonylphenol), and polyoxethylene derivatives of the mono-higher fatty acid esters of hexitol anhydrides (e.g. sorbitan). Exemplary dispersants include methyl cellulose, polyvinyl alcohol, sodium lignin sulfonates, polymeric alkyl naphthalene sulfonates, sodium naphthalene sulfonate, polymethylene bisnaphthalenesulfonate, and sodium N-methyl-N- (long chain acid) laurates.

Water-dispersible powder compositions can be made containing one or more active ingredients, an inert solid extender, and one or more wetting and dispersing agents. The inert solid extenders are usually of mineral origin, such as the natural clays, diatomaceous earth, and synthetic minerals derived from silica and the like. Examples of such extenders include kaolin, attapulgite clay, and synthetic magnesium silicate. Water-dispersible powders described herein can optionally contain from about 5 to about 95 parts by weight of active ingredient (e.g., from about 15 to 30 parts by weight of active ingredient), from about 0.25 to 25 parts by weight of wetting agent, from about 0.25 to 25 parts by weight of dispersant, and from 4.5 to about 94.5 parts by weight of inert solid extender, all parts being by weight of the total composition. Where required, from about 0.1 to 2.0 parts by weight of the solid inert extender can be replaced by a corrosion inhibitor or anti-foaming agent or both.

Aqueous suspensions can be prepared by dissolution or by mixing together and grinding an aqueous slurry of a water-insoluble active ingredient in the presence of a dispersing agent to obtain a concentrated slurry of very finely-divided particles. The resulting concentrated aqueous suspension is characterized by its extremely small particle size, so that when diluted and sprayed, coverage is very uniform.

Emulsifiable oils are usually solutions of active ingredient in water-immiscible or partially water-immiscible solvents together with a surface active agent. Suitable solvents for the active ingredient described herein include hydrocarbons and water-immiscible ethers, esters, or ketones. The emulsifiable oil compositions generally contain from about 5 to 95 parts active

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ingredient, about 1 to 50 parts surface active agent, and about 4 to 94 parts solvent, all parts being by weight based on the total weight of emulsifiable oil.

Compositions described herein can also contain other additaments, for example, fertilizers, phytotoxicants and plant growth regulants, pesticides, and the like used as adjuvants or in combination with any of the above-described adjuvants. The compositions described herein can also be admixed with the other materials, e.g., fertilizers, other phytotoxicants, etc., and applied in a single application.

In each of the formulation types described herein, e.g., liquid and solid formulations, the concentration of the active ingredients are the same.

In some embodiments, the composition can include α -ketoglutarate as the major component. α -ketoglutarate is an important dicarboxylic acid and one of the key intermediates in the tricarboxylic acid cycle and amino acid metabolism. α -ketoglutarate can be isolated from the reaction mixture by methods such as that set forth in French Patent No. 07199, herein incorporated by reference. The α -ketoglutarate composition can be formulated with pharmaceutical excipients and carriers, food additives, or components used to form biomaterials. The α -ketoglutarate composition can be used in a variety of applications, including in synthesizing pharmaceutical agents, food additives, and biomaterials, as described in Li et al., *Bioprocess Biosyst Eng*, 39:967-976 (2016).

It is recognized that the herbicidal compositions can be used in combination with other herbicides. The herbicidal compositions of the present invention are often applied in conjunction with one or more other herbicides to control a wider variety of undesirable vegetation. When used in conjunction with other herbicides, the presently claimed compounds can be formulated with the other herbicide or herbicides, tank mixed with the other herbicide or herbicides or applied sequentially with the other herbicide or herbicides. Some of the herbicides that can be employed in conjunction with the compounds of the present invention include: amide herbicides such as allidochlor, beflubutamid, benzadox, benzipram, bromobutide, cafenstrole, CDEA, chlorthiamid, cyprazole, dimethenamid, dimethenamid-P, diphenamid, epronaz, etnipromid, fentrazamide, flupoxam, fomesafen, halosafen, isocarbamid, isoxaben, napropamide, naptalam, pethoxamid, propyzamide, quinonamid and tebutam; anilide herbicides such as chloranocryl, cisanilide, clomeprop, cypromid, diflufenican, etobenzanid, fenasulam, flufenacet, flufenican, mefenacet, mefluidide, metamifop, monalide, naproanilide, pentanochlor, picolinafen and

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propanil; arylalanine herbicides such as benzoylprop, flamprop and flamprop-M;
 chloroacetanilide herbicides such as acetochlor, alachlor, butachlor, butenachlor, delachlor,
 diethatyl, dimethachlor, metazachlor, metolachlor, S-metolachlor, pretilachlor, propachlor,
 propisochlor, prynachlor, terbuchlor, thenylchlor and xylachlor; sulfonanilide herbicides such as
 5 benzo-fluor, perfluidone, pyrimisulfan and profluazol; sulfonamide herbicides such as asulam,
 carbasulam, fenasulam and oryzalin; antibiotic herbicides such as bilanafos; benzoic acid
 herbicides such as chloramben, dicamba, 2,3,6-TBA and tricamba; pyrimidinyloxybenzoic acid
 herbicides such as bispyribac and pyriminobac; pyrimidinylthiobenzoic acid herbicides such as
 pyri-thiobac; phthalic acid herbicides such as chlorthal; picolinic acid herbicides such as
 10 aminopyralid, clopyralid and picloram; quinolinecarboxylic acid herbicides such as quinclorac
 and quinmerac; arsenical herbicides such as cacodylic acid, CMA, DSMA, hexaflurate, MAA,
 MAMA, MSMA, potassium arsenite and sodium arsenite; benzoylcyclohexanedione herbicides
 such as mesotrione, sulcotrione, tefuryltrione and tembotrione; benzofuranyl alkylsulfonate
 herbicides such as benfuresate and ethofumesate; carbamate herbicides such as asulam,
 15 carboxazole chlorprocarb, dichlormate, fenasulam, karbutilate and terbucarb; carbanilate
 herbicides such as barban, BCPC, carbasulam, carbetamide, CEPC, chlorbufam,
 chlorpropham, CPPC, desmedipham, phenisopham, phenmedipham, phenmedipham-ethyl,
 propham and swep; cyclohexene oxime herbicides such as alloxymid, butoxydim, clethodim,
 cloproxydim, cycloxydim, profoxydim, sethoxydim, tepraloxymid and tralkoxydim;
 20 cyclopropylisoxazole herbicides such as isoxachlortole and isoxaflutole; dicarboximide
 herbicides such as benzfendazole, cinidon-ethyl, flumezin, flumiclorac, flumioxazin and
 flumipropyn; dinitroaniline herbicides such as benfluralin, butralin, dinitramine, ethalfluralin,
 fluchloralin, isopropalin, methalpropalin, nitratin, oryzalin, pendimethalin, prodiamine, profluralin
 and trifluralin; dinitrophenol herbicides such as dinofenat, dinoprop, dinosam, dinoseb,
 25 dinoterb, DNOC, etinofen and medinoterb; diphenyl ether herbicides such as ethoxyfen;
 nitrophenyl ether herbicides such as acifluorfen, aclonifen, bifenox, chlomethoxyfen,
 chlomitrofen, etnipro-mid, fluorodifen, fluoroglycofen, fluoronitrofen, fomesafen, furyloxyfen,
 halosafen, lactofen, nitrofen, nitrofluorfen and oxyfluorfen; dithiocarbamate herbicides such as
 dazomet and metam; halogenated aliphatic herbicides such as alorac, chloropon, dalapon,
 30 flupropanate, hexachloroacetone, iodomethane, methyl bromide, monochloroacetic acid, SMA
 and TCA; imidazolinone herbicides such as imazamethabenz, imazamox, imazapic, imazapyr,
 imazaquin and imazethapyr; inorganic herbicides such as ammonium sulfamate, borax, calcium
 chlorate, copper sulfate, ferrous sulfate, potassium azide, potassium cyanate, sodium azide,

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sodium chlorate and sulfuric acid; nitrile herbicides such as bromobonil, bromoxynil, chloroxynil, dichlobenil, iodobonil, ioxynil and pyraclonil; organophosphorus herbicides such as amiprofos-methyl, anilofos, bensulide, bilanafos, butamifos, 2,4-DEP, DMPA, EBEP, fosamine, glyphosate and piperophos; phenoxy herbicides such as bromofenoxim, clomeprop, 2,4-DEB, 2,4-DEP, 5 difenopenten, disul, erbon, etnipromid, fenteracol and trifopsime; phenoxyacetic herbicides such as 4-CPA, 2,4-D, 3,4-DA, MCPA, MCPA-thioethyl and 2,4,5-T; phenoxybutyric herbicides such as 4-CPB, 2,4-DB, 3,4-DB, MCPB and 2,4,5-TB; phenoxypropionic herbicides such as cloprop, 4-CPP, dichlorprop, dichlorprop-P, 3,4-DP, fenoprop, mecoprop and mecoprop-P; aryloxyphenoxypropionic herbicides such as chlorazifop, clodinafop, clofop, cyhalofop, diclofop, 10 fenoxaprop, fenoxaprop-P, fenthiaprop, fluazifop, fluazifop-P, haloxyfop, haloxyfop-P, isoxapyrifop, metamifop, propaquizafop, quizalofop, quizalofop-P and trifop; phenylenediamine herbicides such as dinitramine and prodiamine; pyrazolyl herbicides such as benzofenap, pyrazolynate, pyrasulfotole, pyrazoxyfen, pyroxasulfone and topramezone; pyrazolylphenyl herbicides such as fluazolate and pyraflufen; pyridazine herbicides such as credazine, pyridafol 15 and pyridate; pyridazinone herbicides such as brompyrazon, chloridazon, dimidazon, flufenpyr, metflurazon, norflurazon, oxapyrazon and pydanon; pyridine herbicides such as aminopyralid, clidinate, clopyralid, dithiopyr, fluroxypyr, haloxydine, picloram, picolinafen, pyriclor, thiazopyr and triclopyr; pyrimidinediamine herbicides such as iprymidam and tioclorim; quaternary ammonium herbicides such as cyperquat, diethamquat, difenzoquat, diquat, morfamquat and 20 paraquat; thiocarbamate herbicides such as butylate, cycloate, di-allate, EPTC, esprocarb, ethiolate, isopolinate, methiobencarb, molinate, orbencarb, pebulate, prosulfocarb, pyributicarb, sulfallate, thiobencarb, tiocarbamil, tri-allate and vemolate; thiocarbonate herbicides such as dimexano, EXD and proxan; thiourea herbicides such as methiuron; triazine herbicides such as dipropetryn, triaziflam and trihydroxytriazine; chlorotriazine herbicides such as atrazine, 25 chlorazine, cyanazine, cyprazine, eglinazine, ipazine, mesoprazine, procyazine, proglinazine, propazine, sebuthylazine, simazine, terbuthylazine and trietazine; methoxytriazine herbicides such as atraton, methometon, prometon, secbumeton, simeton and terbumeton; methylthiotriazine herbicides such as ametryn, aziprotryne, cyanatryn, desmetryn, dimethametryn, methoprotryne, prometryn, simetryn and terbutryn; triazinone herbicides such 30 as ametrifone, amibuzin, hexazinone, isomethiozin, metamitron and metribuzin; triazole herbicides such as amitrole, cafenstrole, epronaz and flupoxam; triazolone herbicides such as amicarbazone, bencarbazone, carfentrazone, flucarbazone, propoxycarbazone, sulfentrazone and thienicarbazone-methyl; triazolopyrimidine herbicides such as cloransulam, diclosulam,

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florasulam, flumetsulam, metosulam, penoxsulam and pyroxsulam; uracil herbicides such as butafenacil, bromacil, flupropacil, isocil, lenacil and terbacil; 3-phenyluracils; urea herbicides such as benzthiazuron, cumyluron, cycluron, dichloralurea, diflufenzopyr, isonoruron, isouron, methabenzthiazuron, monisouron and noruron; phenylurea herbicides such as anisuron,

5 buturon, chlorbromuron, chloreturon, chlorotoluron, chloroxuron, daimuron, difenoxuron, dimefuron, diuron, fenuron, fluometuron, fluothiuron, isoproturon, linuron, methiuron, methyldymron, metobenzuron, metobromuron, metoxuron, monolinuron, monuron, neburon, parafluron, phenobenzuron, siduron, tetrafluron and thidiazuron; pyrimidinylsulfonyleurea herbicides such as amidosulfuron, azimsulfuron, bensulfuron, chlorimuron, cyclosulfamuron,

10 ethoxysulfuron, flazasulfuron, flucetosulfuron, flupyrsulfuron, foramsulfuron, halosulfuron, imazosulfuron, mesosulfuron, nicosulfuron, orthosulfamuron, oxasulfuron, primisulfuron, pyrazosulfuron, rimsulfuron, sulfometuron, sulfosulfuron and trifloxysulfuron; triazinylsulfonyleurea herbicides such as chlorsulfuron, cinosulfuron, ethametsulfuron, iodosulfuron, metsulfuron, prosulfuron, thifensulfuron, triasulfuron, tribenuron, triflusulfuron and

15 tritosulfuron; thiadiazolyleurea herbicides such as buthiuron, ethidimuron, tebuthiuron, thiazafluron and thidiazuron; and unclassified herbicides such as acrolein, allyl alcohol, aminocyclopyrachlor, azafenidin, benazolin, bentazone, benzobicyclon, buthidazole, calcium cyanamide, cambendichlor, chlorfenac, chlorfenprop, chlorflurazole, chlorflurenol, cinmethylin, clomazone, CPMF, cresol, ortho-dichlorobenzene, dimepiperate, endothal, fluoromidine,

20 fluridone, flurochloridone, flurtamone, fluthiacet, indanofan, methazole, methyl isothiocyanate, nipyraclufen, OCH, oxadiargyl, oxadiazon, oxaziclomefone, pentachlorophenol, pentoxazone, phenylmercury acetate, pinoxaden, prosulfalin, pyribenzoxim, pyriftalid, quinoclamine, rhodethanil, sulglycapin, thidiazimin, tridiphane, trimeturon, tripropindan and tritac. The herbicidal compositions of the present invention can, further, be used in conjunction with

25 glyphosate or 2,4-D on glyphosate-tolerant or 2,4-D-tolerant crops. It is generally preferred to use the compositions of the invention in combination with herbicides that are selective for the crop being treated and which complement the spectrum of weeds controlled by these compositions at the application rate employed. It is further generally preferred to apply the compositions of the invention and other complementary herbicides at the same time, either as a

30 combination formulation or as a tank mix.

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The compositions described herein can be used in methods for selectively controlling weeds in a field or any other area, including, for example, a railway, lawn, golf course, and others where the control of weeds is desired. Optionally, the field or other area can contain a crop of planted seeds or crops that are resistant to glufosinate. The methods can include applying an effective amount of a composition comprising L-glufosinate as described herein to the field.

The compositions described herein are useful for application to a field of crop plants for the prevention or control of weeds. The composition may be formulated as a liquid for spraying on a field. The L-glufosinate is provided in the composition in effective amounts. As used herein, effective amount means from about 10 grams active ingredient per hectare to about 1,500 grams active ingredient per hectare, e.g., from about 50 grams to about 400 grams or from about 100 grams to about 350 grams. In some embodiments, the active ingredient is L-glufosinate. For example, the amount of L-glufosinate in the composition can be about 10 grams, about 50 grams, about 100 grams, about 150 grams, about 200 grams, about 250 grams, about 300 grams, about 350 grams, about 400 grams, about 500 grams, about 550 grams, about 600 grams, about 650 grams, about 700 grams, about 750 grams, about 800 grams, about 850 grams, about 900 grams, about 950 grams, about 1,000 grams, about 1,050 grams, about 1,100 grams, about 1,150 grams, about 1,200 grams, about 1,250 grams, about 1,300 grams, about 1,350 grams, about 1,400 grams, about 1,450 grams, or about 1,500 grams L-glufosinate per hectare.

IV. Exemplary Embodiments

Non-limiting embodiments include:

1. A method for making L-glufosinate, comprising:
 - reacting D-glufosinate with a D-amino acid oxidase (DAAO) enzyme to form PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid); and
 - aminating the PPO to L-glufosinate by a transaminase (TA) enzyme, using an amine group from one or more amine donors,
 - wherein at least 70% of the D-glufosinate is converted to L-glufosinate.

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2. The method of embodiment 1, wherein the amine donor is selected from the group consisting of glutamate, L-glutamate, alanine, sec-butylamine, phenylethylamine, glycine, lysine, valine, serine, glutamine, isopropylamine, ethanolamine, 2-aminobutyric acid, and diaminopropionic acid, or any secondary amine or amino acid.
- 5 3. The method of embodiment 1, wherein the D-glufosinate is originally present in a racemic mixture of D- and L-glufosinate or salts thereof.
4. The method of embodiment 1, wherein the DAAO enzyme is selected from the enzyme from *Rhodospiridium toruloides* or *Trigonopsis variabilis*, *Neolentinus lepideus*, *Trichoderma reesei*, or *Trichosporon oleaginosus*. In an embodiment, the *Rhodospiridium*
10 *toruloides* DAAO enzyme is UniProt P80324. In an embodiment, the *Trigonopsis variabilis* DAAO enzyme is UniProt Q99042. In an embodiment, the *Neolentinus lepideus* DAAO enzyme is KZT28066.1. In an embodiment, the *Trichoderma reesei* DAAO enzyme is XP_006968548.1. In an embodiment, the *Trichosporon oleaginosus* DAAO enzyme is KLT40252.1.
5. The method of embodiment 1, wherein the DAAO enzyme is a mutant DAAO.
- 15 6. The method of embodiment 5, wherein the mutant DAAO is a mutant DAAO based on the sequence from *Rhodospiridium toruloides*.
7. The method of embodiment claim 5, wherein the mutant DAAO comprises one or more mutations at positions 54, 56, 58, 213, and 238.
8. The method of embodiment 7, wherein the mutation at position 54 is selected
20 from the group consisting of N54C, N54L, N54T, and N54V.
9. The method of embodiment 7, wherein the mutation at position 56 is T56M.
10. The method of embodiment 7, wherein the mutation at position 58 is selected from the group consisting of F58A, F58G, F58H, F58K, F58N, F58Q, F58R, F58S, and F58T.
11. The method of embodiment 7, wherein the mutation at position 213 is M213S.
- 25 12. The method of embodiment 5, wherein the mutant DAAO comprises mutations F58K and M213S.

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13. The method of embodiment 5, wherein the mutant DAAO comprises mutations at positions 54 and 56.
14. The method of embodiment 5, wherein the mutant DAAO comprises mutations N54T and T56M.
- 5 15. The method of embodiment 5, wherein the mutant DAAO comprises mutations F58Q or F58H.
16. The method of embodiment 5, wherein the mutant DAAO comprises mutations N54V and F58Q.
- 10 17. The method of embodiment 5, wherein the mutant DAAO comprises mutations N54V, F58Q, and M213S.
18. The method of embodiment 1, wherein the TA enzyme is a gabT transaminase from *Escherichia coli*. In an embodiment, the *Escherichia coli* gabT transaminase is UniProt P22256.
- 15 19. The method of embodiment 1, wherein the TA enzyme is encoded by SEQ ID NO: 1.
20. The method of embodiment 1, wherein the reacting step and the aminating step are performed in a single container.
21. The method of embodiment 20, wherein all reagents are substantially added at the start of the reaction.
- 20 22. The method of embodiment 20, wherein the reagents for the reacting step and the reagents for the aminating step are added to the single container at different times.
23. The method of embodiment 1, wherein the reacting step and the aminating step are performed in separate containers.
24. A composition comprising D-glufosinate, PPO, and L-glufosinate.
- 25 25. The composition of embodiment 24, wherein the amount of L-glufosinate is 90% or greater based on the total amount of D-glufosinate, PPO, and L-glufosinate.

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26. The method of embodiment 1 wherein a solid is obtained having the composition of embodiment 24 or 25.

27. The method of embodiment 1 wherein a solution of L-glufosinate is obtained for use in a formulation which has herbicidal activity.

5 28. A formulation, comprising L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; one or more additional components selected from the group consisting of sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; dipropylene glycol in an amount from 4-18% by weight of the formulation; and alkyl polysaccharide in an
10 amount from 4-20% by weight of the formulation; and water as the balance of the formulation.

29. The composition of embodiment 28, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the
15 formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.

30. The composition of embodiment 28, wherein the formulation comprises: L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount
20 of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 24.5% by weight of the formulation.

31. The composition of embodiment 28, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount
25 of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation.

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32. A formulation, comprising L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; one or more additional components selected from the group consisting of sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; and alkyl polysaccharide in an amount from 3-10% by weight of the formulation; and water as the balance of the formulation.

33. The composition of embodiment 32, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 58.45% by weight of the formulation.

34. The composition of embodiment 32, wherein the formulation comprises: L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

35. The composition of embodiment 32, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 11.05% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; alkyl polysaccharide in an amount of 3.1% by weight of the formulation; and water in an amount of 73.1% by weight of the formulation.

36. A method for selectively controlling weeds in an area comprising:
applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate to the area.

37. The method of embodiment 36, wherein the amount of the composition is applied at less than 400 grams of the sum of L-glufosinate and D-glufosinate per hectare.

38. A method for selectively controlling weeds in an area comprising:

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applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate to the area and more than 0.01% but less than 10% PPO to the area.

39. The method of embodiment 38, wherein the amount of the composition is applied at less than 400 grams of the sum of L-glufosinate, D-glufosinate and PPO per hectare.

40. A method for selectively controlling weeds in an area containing a crop of planted seeds or crops that are resistant to glufosinate, comprising:
applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate and more than 0.01% but less than 10% PPO to the field.

The following examples are offered by way of illustration and not by way of limitation.

EXAMPLES**Example 1: DAAO enzyme purification**

The coding sequence of a mutant DAAO from *Rhodospiridium toruloides* (for example, consisting of a MMARIRL leader sequence and the F58K and M213S mutations) was cloned into the pET14b vector to allow for expression of an N terminally 6xHis tagged protein. This pET14b-RgDAAO plasmid was transformed into BL21 (BE3) trxB pLysS cells. The sequence of the wild type DAAO from *Rhodospiridium toruloides* to which all numbering described here corresponds, is:

MHSQKRVVVLGSGVIGLSSALILARKGYSVHILARDLPEDVSSQTFASPWAGANWTPF
MTLTDGPRQAKWEESTFKKWVELVPTGHAMWLKGTRRFAQNEDGLLGHWYKDITPNYRPLP
SSECPPGAIGVTYDTLSVHAPKYCQYLARELQKLGATFERRTVTSLEQAFDGADLVVNATGLG
AKSIAGIDDQAAEPIRGQTVLVKSPCKRCTMDSSDPASPAYIIPRPGGEVICGGTYGVGDWDL
VNPETVQRILKHCLRLDPTISSDGTIEGIEVLRHNVGLRPARRGGPRVEAERIVLPLDRTKSPLSL
GRGSARAAKEKEVTLVHAYGFSSAGYQQSWGAAEDVAQLVDEAFQRYHGAARESKL (SEQ
ID NO: 2).

To purify DAAO enzyme, cells were grown in 400 mL autoinducing medium (LB broth base with trace elements, Formedium) at 30°C for 20 to 24 hours. Cells were harvested in

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precooled centrifuges and buckets, washed with cold water, centrifuged again, and stored at -80 °C until purification.

Cell pellets were then thawed in lysis buffer (50 mM potassium phosphate, pH 8.0, 20 mM imidazole and 1% Sigma protease inhibitor cocktail (PIC) w/o EDTA) at a volume of 5 mL of lysis buffer per 1 g cell pellet. While on ice, cells were sonicated 4 times for 30 seconds at an amplitude of 10. The cell lysate was clarified by centrifugation and then added to cobalt resin (HisPur Cobalt, ThermoScientific) at 4 times the bed volume. The cell lysate was incubated for 1 hour, with gentle shaking, at room temperature. The resin was added to a column and washed twice with 5 bed volumes of wash buffer (50 mM Kpi, pH 8.0, 20 mM imidazole). The elution was performed 4 times with 1 bed volume of elution buffer (50 mM Kpi, 200 mM imidazole).

Example 2: Colorimetric determination of DAAO activity

DAAO activity was determined similarly to Berneman et al. In brief, 100 uL of substrate and HRP (0.1 mg/mL HRP, Sigma P8375, and the desired amount of D-glufosinate or racemic D/L-glufosinate in 50 mM potassium phosphate, pH 8) was added to a Brand UV micro cuvette. To that, 50 uL of dyes (60 ug/mL TBHBA, Sigma 439533, and 1 mg/mL 4-aminoantipyrine, Sigma A4382, in 50 mM potassium phosphate, pH 8) was added and then 50 uL of enzyme mix (DAAO concentration as desired in 100 mM potassium phosphate, pH 8). The reaction was monitored on a spectrophotometer at 510 nm over an appropriate time to determine the enzyme kinetics. Although no flavin adenine dinucleotide (FAD) was added to the purification of DAAO or the reaction, this reagent can optionally be included. Two exemplary mutant variants of *Rhodospiridium toruloides* DAAO, AC201 (containing F58K and M213S) and AC263 (containing N54T, T56M, F58K, and M213S), purified as in Example 1, were tested using this assay and shown to produce hydrogen peroxide, demonstrating their activity in oxidizing D-glufosinate. AC201 and AC263 have similar V_{max} , but AC263 has a lower K_M .

Example 3: Purification of transaminases

To purify, for example, the *Escherichia coli* gabT transaminase (UniProt P22256), the gene was amplified from E. coli K12 strain ER2925 and cloned into pET-14b to generate an N-terminal 6xHis tagged version. This plasmid was then transformed into BL21 (DE3) cells for

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induction. After induction in autoinducing media, cells were lysed by sonication and the 6xHis tagged enzyme purified as described in Example 1.

Example 4: Demonstration of transaminase activity

In a non-limiting example, the source of PPO for a transamination assay can be D-
5 glufosinate or racemic D/L-glufosinate that has been converted by a DAAO to PPO. In the first step, 39 mM racemic D/L-glufosinate was incubated with 0.5 mg/ml purified *Rhodospiridium toruloides* DAAO F58K M213S and 10 ug/mL catalase in 50 mM Potassium phosphate buffer, pH8 for 20 hours at 30 C. This resulted in conversion of the majority of the D-glufosinate to PPO. Subsequently, purified *E. coli* gabT was added at 20 ug/mL and L-glutamate was added at
10 50 mM as the amine donor. At relevant points, samples were stopped by boiling for 10 minutes followed by precipitation with an equal volume of acetonitrile. The individual chemical species were resolved on an HPLC with a Chirobiotic T2 column and quantified by comparison to authentic standards.

The combination of a mutant variant of DAAO and a transaminase resulted in an
15 improvement of an enantiomeric enrichment that started at 0% for L-glufosinate over D-glufosinate (i.e., equal representation of D-glufosinate and L-glufosinate) to an enantiomeric enrichment of 92%. These results demonstrate that *E. coli* gabT has transaminase activity, and this assay can be used to determine the activity of any number of wild type and/or mutant potential transaminases.

20 Example 5: De-racemization of racemic D/L-glufosinate in a single vessel

Reactions were set up similarly as in Example 4. The system (5.45 mL, 30 °C) was run in phosphate buffer at a pH of 7.3. It was noted that 50 mM of phosphate buffer at a pH of 8.0 was inadequate to buffer the amino acid additions and that the unadjusted pH after amino acid additions in this system was pH 6.4. The pH was adjusted using 1M of the base salt K₂HPO₄
25 to a volume of 5.45 mL, meaning that the actual initial substrate concentration by addition was 275 mM. The following reagents were added essentially simultaneously at the start of the reaction: 271 mg D,L-glufosinate, 420 mg glutamate, 15 mg AC263 DAAO, 50 µg catalase, and 1.0 mg *E. coli* gab T transaminase. Figure 2 shows that, when all reagents were added, the amount of D-PPT (D-glufosinate) diminished with only modest accumulation of PPO. This result

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indicates an efficient deracemisation of D/L-glufosinate into L-glufosinate by the RgDAAO/EcgabT enzyme couple.

Example 6: Demonstration of improved DAAO enzymes

5 Using protein mutagenesis strategies as outlined above, improved and variant DAAO enzymes were identified. The enzymes were assayed according to the procedures described below.

Stock Solutions:

The following dye stock solutions were prepared: a 20 mg/mL stock solution of 2,4,6-tribromo-3-hydroxybenzoic acid (TBHBA) in DMSO; and a 100 mg/mL stock solution of 4-aminoantipyrine (4-AAP) in water. The following enzyme stock solution was prepared: a 10 mg/mL stock solution of horseradish peroxidase (HRP) type 6 in a pH 8.0 potassium phosphate buffer. The following substrate stock solution was prepared: varying concentrations of D or DL amino acid in a pH 8.0 potassium phosphate buffer.

Reaction Mixes:

15 The following reaction mixtures were prepared:

Mix A is a combination of the substrate and HRP enzyme. Solutions were prepared for each substrate concentration to be assayed using reaction buffer. The solutions were two times the final substrate concentration and 0.2 mg/mL for the HRP solution.

20 Mix B is a dye mixture. To 5 mL of reaction buffer was added 120 μ L of TBHBA solution and 400 μ L of 4-AAP solution.

Mix C is an enzyme mixture. A 0.1 mg/mL solution of DAAO in reaction buffer was prepared. The final reaction concentration was 25 μ g/mL.

Protocol:

25 A spectrophotometer was used at a wavelength of 510 nm, which corresponds to the maximum absorbance for 4-AAP/TBHBA and is the point at which the extinction coefficient is 29400 M⁻¹ cm⁻¹. The temperature for performing the assays was 30 °C. The reaction kinetics

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were obtained by measuring every minute for 15 minutes. Between measurements, 20 seconds of orbital shaking at normal intensity was performed, followed by 10 seconds of settling time.

Using a 96-well plate, the following mixes (with replicates) were added in the following order using multi-channel: 100µl mix A, 50µl mix B, and 50µl mix C. The measurements were started immediately after the enzyme addition.

The enzyme kinetics were measured as described above, plotted on a Michaelis Menten graph, and used to calculate V_{max} and K_M . For the variant Ac302 (54V, 58Q, 213S), the V_{max} was 4.2 umol/min*mg.

This analysis was completed for a number of variant DAAO enzymes as above except that the Mix C stock was 0.2 mg/mL solution of DAAO and this final reaction concentration of DAAO was 50 ug/mL.

As shown below in Table 1, variant mutant DAAO enzymes showed a range of activities:

Table 1:

Variant	Mutations	Vmax (% of Ac302)
Ac263	54T, 56M, 58K, 213S	33
Ac302	54V, 58Q, 213S	100
Ac305	54C, 58H, 213S	88
Ac309	54T, 58T, 213S	71
Ac312	54T, 58G, 213S	74
Ac314	54T, 58Q, 213S	99
Ac316	54T, 58S, 213S	75
Ac318	54T, 58A, 213S	71
Ac319	54L, 58R, 213S	64
Ac320	54V, 58R, 213S	76
Ac322	54V, 58N, 213S	79

Example 7: De-racemization of racemic D/L-glufosinate at a 5 L reaction size

The scale of the de-racemization is increased using approaches familiar to those skilled in the art. Reagents and their relative ratios are substantially similar to Example 5, but the amounts are significantly greater. Rather than tubes in shakers, the reactions are performed in stirred jacketed reactors, including, optionally, air or oxygen sparging of the broth or headspace.

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These reactors vary in size, from less than 10 mL reaction to tens or hundreds of thousands of liters. Stirring rates are chosen to increase reaction mixing and rate while minimizing power consumption and shear.

- 5 In one example, the reaction was run at the 5 L scale. The system (5 L, 30 °C) was run in 200 mM phosphate buffer at a pH of 8.0 in a stirred, jacketed reactor. The following reagents were added essentially simultaneously at the start of the reaction: 300 mM D,L-glufosinate, 900 mM glutamate, 7.5 g AC302 DAAO, 0.2 g catalase, and 1.0 g *E. coli* gab T transaminase. In addition, 500 mL isopropanol was added to control foaming. During the course of the reaction, air was introduced at 0.3 VVM (volumes of air per volume of reaction mixture per minute).
- 10 HPLC analysis of the reaction demonstrated that equilibrium was reached within 8 hours, with the enantiomeric excess of L-glufosinate over D-glufosinate greater than 99% and the ratio of L-glufosinate to PPO 90% to 10%. This result indicates an efficient deracemisation of D/L-glufosinate into L-glufosinate by the RgDAAO/EcgabT enzyme couple at the larger scale.

Example 8: Impact of oxygen on reaction rate

- 15 Although stirred, jacketed reactors or immobilized columns typically allow for some oxygen transfer, the rate of oxygen uptake afforded by passive aeration is not sufficient for an efficient process. In one example, a reaction was run in the same vessel as Example 7 under substantially the same conditions, but under reduced (0.01 VVM), with twice the AC302 DAAO on a volumetric basis (3 g/L versus 1.5 g/L), and without the isopropanol. In this case, the
- 20 reaction took more than 60 hours to achieve equilibrium, demonstrating the critical importance of aeration for an efficient reaction.

Example 9: Co-immobilization of DAAO and TA

- DAAO and TA enzymes were co-immobilized on EziG controlled pore glass beads (EnginZyme). 100mg of EziG type 3 beads were shaken at room temperature with 3ml of
- 25 solution containing 16 mg of purified AC302 DAAO and 1.6mg of purified gabT in 50 mM potassium phosphate buffer pH 7.5, 0.5M NaCl, 20mM imidazole in a 50 ml Falcon tube. After 30 minutes, beads were spun down, immobilizing solution was removed, and beads were washed 3 times with 10ml of 100 mM potassium phosphate buffer pH 7.5.

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The reaction was started by adding all other components to the washed beads. The reaction mix contained 300 mM D/L-glufosinate, 900 mM L-glutamic acid, 50 ug catalase, 198 mM potassium phosphate in 2.5 mL. The reaction was incubated at 30C with shaking (250 rpm) in a 50 mL tube covered in parafilm with holes poked through for gas exchange.

5 After 1 hour, the depletion of D-glufosinate and formation of L-glufosinate was determined by HPLC and these rates calculated. After 6 hours, beads were spun down, reaction mixture was removed, and beads were washed 3 times with 10ml of 100 mM Potassium Phosphate buffer pH 7.5. Beads were then stored at 4 C for 18 to 72 hours before the reaction was repeated, for a total of 15 times, after which the retained activity was greater than 50% of
10 the initial activity.

Example 10: Effect of buffer on reaction

When soluble AC302 DAAO and *E. coli* gabT TA enzymes are used, phosphate buffer at >50 mM is required for full activity. A 100 mL reaction was incubated at 30C with shaking (250 rpm) in a 500 mL flask covered in parafilm. An air pump was used to bubble air through the
15 reaction for the first 5 hours. The air pump was removed for overnight incubation so the reaction would not bubble over and new parafilm with air holes was used for gas exchange. The reaction mix contained 300 mM D/L-glufosinate, 905 mM L-glutamic acid, 80 mg AC302 DAAO (0.8 mg/mL), 14.5 mg gabT (0.145 mg/mL), 2 mg catalase, and isopropanol as anti-foam reagent (10% initial concentration, isopropanol was additionally added at 2hr (2mL), 3hr (1mL), 3.5hr
20 (1mL), and at 4hr (2mL)). 500 uL of 1N NaOH (added before enzymes) was used to adjust pH from about 6 to about 7. pH remained at about 7 for the entire reaction without further adjustment. Due to the potassium phosphate in the stock enzyme buffer, the final mixture was 45 mM phosphate buffer. When compared to a similar reaction with 200 mM phosphate buffer, the reaction rate was 50-60% of that of the reaction with the 200 mM buffer.

25 When immobilized AC302 DOOA and *E. coli* gatT TA enzymes are used, phosphate buffer of less than 1 mM is sufficient for full activity. Immobilized proteins were prepared and reaction performed as in Example 9 for the "Buffered" reaction. In addition, immobilized proteins were prepared and reactions performed as in Example 9 for the "pH 7" reaction, except that sodium hydroxide was used to adjust the pH of the reaction to pH 7 and no phosphate buffer
30 was added (residual phosphate buffer from the enzyme storage buffer is less than 1 mM). This work demonstrated that the initial reaction rate for both the DAAO and combined DAAO and

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gabT reactions are very similar with and without the addition of phosphate buffer when immobilized enzymes are used.

Example 11: Isopropylamine as an amine donor

Isopropylamine can be used as an amine donor for conversion of PPO to L-glufosinate with the use of an appropriate TA. PPO was converted to L-glufosinate in a reaction with the following components:

- 0.25 mg/mL TA encoded by SEQ ID NO: 1
- 25 mM PPO
- 0.2 mM pyridoxal phosphate
- 250 mM isopropylamine (pHed to 8 w/ H₃PO₄)
- 100 mM Kphos buffer pH 8.0

The reaction was incubated at 25 to 30 °C for 30 hours with gentle shaking (250 rpm). At 0 hours, the amount of L-glufosinate as measured by HPLC was 0 mM, at 20 hours it was 14 mM, and at 30 hours it was 18 mM. This demonstrates that the enzyme encoded by SEQ ID NO: 1 can convert PPO into L-glufosinate.

Example 12: Lysine as an amine donor

Lysine can be used as an amine donor for conversion of PPO to L-glufosinate with the use of an appropriate TA. PPO was converted to L-glufosinate in a reaction with the following components:

- 0.4 mg/mL gabT (purified as in Example 3)
- 25 mM PPO (pHed to 8 w/ NaOH)
- 0.2 mM pyridoxal phosphate
- 75 mM L-Lysine dihydrochloride (pHed to 8 w/ NaOH)
- 100 mM Kphos buffer pH 8.0

The reaction was incubated at 30 °C for 20 hours with shaking (250 rpm). L-glufosinate was formed at a rate of 0.4 mM/hr over the 20 hours. This demonstrates that L-lysine can be used to convert PPO to L-glufosinate.

SUBSTITUTE SPECIFICATION - CLEAN**Example 13: Purification and isolation of L-glufosinate**

Several batches prepared following the procedure described in Example 9 but at larger scale were generated. After the beads were removed, each batch was heated to 90 °C for at least 10 minutes, and after cooling to 20-25 °C, filtered to remove a small amount of solids. To each individual batch was added 37% HCl, dropwise, to effect the precipitation of glutamic acid. The amount of 37% HCl added was approximately 10% of the volume of the batch. The resulting white solid was removed by filtration. The batches were combined and concentrated under vacuum to an oil; the oil contained approximately 153 grams of L-glufosinate. The oil was diluted with five volumes of water and 37% HCl was added to adjust the solution to pH 1. The solution was treated sequentially with two portions each of approximately 3.0 kg of prewashed DOWEX 50WX8 cation exchange resin. In each treatment, the solution was allowed to mix with the resin for 30 minutes after which the resin was isolated on a filter. Both portions of resins were combined and washed first with water and then eluted with 4M NH₄OH. The eluent was concentrated under vacuum to an oil; PPO and 2-oxoglutarate were not present in the oil. Approximately 100 grams of the oil was diluted with water and the aqueous ammonium hydroxide was added until the pH was approximately 9. To the batch was added 1.0 kg of prewashed DOWEX Monosphere (hydroxide form) anion exchange resin and the mixture was stirred for approximately 40 minutes. An equal amount of DOWEX Monosphere resin, prewashed, was charged to a glass column. The slurry of DOWEX resin in water was added to the column on top of the prewashed resin. 800 mL of water was charged to the column followed by 0.1 N acetic acid, which was kept flowing through the column until all of the glutamic acid had eluted as determined by HPLC. 4 N acetic acid was fed to the column until all of the L-glufosinate had eluted from the column as determined by HPLC. The solution of L-glufosinate was concentrated under vacuum. The resulting oil was diluted with water and concentrated under vacuum to minimum volume two times. Methanol was added until a clear solution was obtained and an equal volume of heptane was added. The mixture was concentrated under vacuum to minimum volume and the procedure was repeated. The remaining 168 grams of oil recovered from the cation exchange treatment was treated in a similar fashion to obtain an overall total of 108 grams of crude L-glufosinate. The ratio of L-glufosinate to glutamic acid was greater than 99:1 as determined by NMR. The resulting solid was mixed with aqueous ammonium hydroxide and concentrated to dryness to afford 111 grams of L-glufosinate ammonium. Neither methanol nor acetic acid was detected by NMR analysis of the product.

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It is understood that the terminology used herein is for the purpose of describing particular embodiments only, and the terminology is not intended to be limiting. The scope of the invention will be limited only by the appended claims. Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which this invention belongs. Where a range of values is provided, it is understood that each intervening value, to the tenth of the unit of the lower limit unless the context clearly dictates otherwise, between the upper and lower limit of that range and any other stated or intervening value in that stated range, is encompassed within the invention. The upper and lower limits of these smaller ranges may independently be included in the smaller ranges and are also encompassed within the invention, subject to any specifically excluded limit in the stated range. Where the stated range includes one or both of the limits, ranges excluding either or both of those included limits are also included in the invention. Certain ranges are presented herein with numerical values being preceded by the term "about." The term "about" is used herein to provide literal support for the exact number that it precedes, as well as a number that is near to or approximately the number that the term precedes. In determining whether a number is near to or approximately a specifically recited number, the near or approximating unrecited number may be a number, which, in the context in which it is presented, provides the substantial equivalent of the specifically recited number.

All publications, patents, and patent applications cited in this specification are incorporated herein by reference to the same extent as if each individual publication, patent, or patent application were specifically and individually indicated to be incorporated by reference. Furthermore, each cited publication, patent, or patent application is incorporated herein by reference to disclose and describe the subject matter in connection with which the publications are cited. The citation of any publication is for its disclosure prior to the filing date and should not be construed as an admission that the invention described herein is not entitled to antedate such publication by virtue of prior invention. Further, the dates of publication provided might be different from the actual publication dates, which may need to be independently confirmed.

It is noted that the claims may be drafted to exclude any optional element. As such, this statement is intended to serve as antecedent basis for use of such exclusive terminology as "solely," "only," and the like in connection with the recitation of claim elements, or use of a "negative" limitation. As will be apparent to those of skill in the art upon reading this disclosure, each of the individual embodiments described and illustrated herein has discrete components

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and features which may be readily separated from or combined with the features of any of the other several embodiments without departing from the scope or spirit of the invention. Any recited method may be carried out in the order of events recited or in any other order that is logically possible. Although any methods and materials similar or equivalent to those described

5 herein may also be used in the practice or testing of the invention, representative illustrative methods and materials are now described.

SEQUENCES

SEQ ID NO: 1:

MNIAAQSWERREATSFFHTFTDLP SLKTDGPVIIDHGEGPYIIDTVGRRYFEGNSGLWNMTLGF
 10 SERRLSDAALKQYQEFPGYHTFFGRNSKPTVELAERMLKLAPAPMSRVFFTN SGSEANESIVK
 LLWMMWAAEGRPERRKL LTRKNAYHGATVMASALTGKD YVKAFLPGPEIVTLDCPHAWRFA
 LPGEGDDEFAARLAANLETRILQEGPETIAGMFAEPVMGAGGVIVPPATYFAKIQPVLQRYGIPL
 IADEVICGFGRTGSLWGTLAVGQQPDII VASKSMSAGYFPMGAVMLSADIDKRATAASEVWEEF
 PHGFTTG GHPVGCAISLEAIRIITEEGVFENVKSVSETFQSGLRALADHPMIGEARGMGLMGAL
 15 ETVADKKTKQSFSGDLRIGERISKEARDRGFIIRPLGSSVVLAPPFISTHGQIEELLAVLKEVL DV
 VYGT VKGEVA

SEQ ID NO: 2:

MHSQKRVVVLGSGVIGLSSALILARKGYSVHILARDLPEDVSSQTFASPWAGANWTPFMTLTD
 GPRQAKWEESTFKKWVELVPTGHAMWLKGTRRFAQNEDG LLGHWYKDITPNYRPLPSSECP
 20 PGAIGVTYDTLSVHAPKYCQYLARELQKLGATFERRTVTSLEQAFDGADLVVNATGLGAKSIAG
 IDDQAAEPIRGQTVLVKSPCKRCTMDSSDPASPAYIIPRPGGEVICGGTYGVGDWDL SVNPETV
 QRILKHCLRLDPTISSDGTIEGIEVLRHNVGLRPARRGGPRVEAERIVLPLDRTKSPLSLGRGSA
 RAAKEKEVTLVHAYGFSSAGYQQSWGAAEDVAQLVDEAFQRYHGAARESKL

SEQ ID NO: 3:

MNSNKELMQRRSQAIPRGV GQIHPIFADRAENCRVWDVEGREYLDFAGGIAVLNTGHLHPKV V
 25 AAVEAQLKKLSHTCFQVLAYEPYLELCEIMNQKVP GDFAKKTLLVTTGSEAVENAVKIARAATK
 RSGTIAFSGAYHGRTHYTLALTGKVNPYSAGMGLMPGHVYRALYPCPLHGISEDDAIASIHRIFK
 NDAAPEDIAAIVIEPVQGE GGFYASSPAFMQRLRALCDEHGIMLIADEVQSGAGRTGTLFAMEQ
 MGVAPDLTTFAKSIAGGFPLAGVTGRAEVM DAVAPGGLGGTYAGNPIACVAALEVLKVFEQEN
 30 LLQKANDLGQKLKDGLLAIAEKHPEIGDVRGLGAMIAIELFEDGDHKNKPDAKLTAEIVARARDKG
 LILLSCGPYYNVLRILVPLTIEDAQIRQGLEIISQCFDEAKQ



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18/435,340

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Title of Invention

METHODS FOR MAKING L-GLUFOSINATE

Application Information

APPLICATION TYPE Utility - Nonprovisional Application
under 35 USC 111(a)

PATENT # -

CONFIRMATION # 4214

FILED BY Rebecca Walsh

PATENT CENTER # 65796481

FILING DATE 02/07/2024

CUSTOMER # 4743

FIRST NAMED INVENTOR Brian Michael Green

CORRESPONDENCE ADDRESS -

AUTHORIZED BY Joshua Elliott

Documents

TOTAL DOCUMENTS: 4

DOCUMENT	PAGES	DESCRIPTION	SIZE (KB)
Response to Notice to Comply with Requirements for Nucleotide or Amino Acid Sequence Discl.pdf	2	Response to Pre-Exam Sequence Notice	18 KB
Substitute Specification - Clean-SPEC.docx	44	Specification	70 KB

Warning: Bookmarks were found and have been removed.

Substitute Specification - Marked-SPEC.docx	44	Specification	73 KB
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Warning: One or more pages are missing page numbering. Page numbering will be

automatically applied after submission. Track changes containing insertions/deletions have been detected. These changes will be displayed in the document as underlines/strikethroughs. Bookmarks were found and have been removed.

M202647G_SubSeqListing.xml	-	Sequence Listing Supp (XML File)	5 KB
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Warning: Please note that ST.23 and ST.25 sequence listings are required for applications with a filing date before July 1, 2022.

Digest

DOCUMENT	MESSAGE DIGEST(SHA-512)
Response to Notice to Comply with Requirements for Nucleotide or Amino Acid Sequence Discl.pdf	7E519FB285D0598A0E2FABAEA6F0E05FAFBFC69AD20B754F2AFEC342C2E77BE89263AC9C39E0ACD9EC8B540FE42A3B883248B47462BA1566BF5588DEED6C4622
Substitute Specification - Clean-SPEC.docx	909B4B0379285E177EC96E862E91724FD5A7856BA7BDC6B470EF11D07A5BE7B221384F7705EA09973B6EC0BB4FBEACE6D98FD7A812756D9912BA184CCCF8E389
Substitute Specification - Marked-SPEC.docx	E1634748ADB41FC3C024176A15C6F45FB6A779F0620CA3C3245D6A28C3EEC069FFC3E9BF618AE7F67E3D567B2695C5A30D51937DB0868928378ABEF7C0136EF8
M202647G_SubSeqListing.xml	EE0B5365A966AC906988AB937B52E1ED40A39AC75CA4C5CD7E6216971B789B4FEB9BED619677804F0A6066AFD57B470419B41EBAFB719F998F5C1AEFB62F6732

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Docket No.: 32471/M202647G
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Brian Michael Green et al.

Application No.: 18/435,340

Confirmation No.: 4214

Filed: February 7, 2024

Art Unit: Not Yet Assigned

For: METHODS FOR MAKING L-GLUFOSINATE

Examiner: Not Yet Assigned

**RESPONSE TO NOTICE TO COMPLY WITH REQUIREMENTS
FOR PATENT APPLICATION CONTAINING NUCLEOTIDE
SEQUENCE AND/OR AMINO ACID SEQUENCE DISCLOSURES**

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Commissioner:

This is in response to the Notice To Comply With Requirements For Patent Applications Containing Nucleotide Sequence and/or Amino Acid Sequence Disclosures, dated April 3, 2024. Applicants are submitting their response herewith within the two-month response period set to expire on June 3, 2024.

The U.S. Patent and Trademark Office has taken the position that the previous "Sequence Listing" in computer readable form did not comply with the requirements of 37 C.F.R § 1.824 for the stated reason that it contained one raw sequence listing error. Submitted herewith in connection with the above-referenced patent application is a substitute, computer readable copy of the Sequence Listing that corrects the defects noted in the raw sequence listing. No new matter has been added to the substitute Sequence Listing.

In response to Comply with Requirements for Patent Applications Containing Nucleotide Sequence and/or Amino Acid Sequence Disclosures, Applicant submits the following:

1. M202647G_SubSeqListing.xml; Size: 5,116 bytes; Created: April 17, 2024

The replacement sequence listing in XML format has been updated to reflect ST.26 standards. Based on the Reviewer's comment, Seq ID No. 1 contained extra information "sp." in the organism qualifier value. Moreover, the Reviewer suggested that "Sinorhizobium arboris" is already a 'Genus species' name. Thus, the organism qualifier value has been correctly adjusted to "Sinorhizobium arboris" by removing the extra information "sp." as instructed by the reviewer.

Other than the mentioned changes, there has been no substantive addition of material within the sequence listing.

Applicant hereby submits a clean Substitute Specification and a marked Substitute Specification to update the information related to the sequence listing in the "Incorporation by Reference" section. Applicant respectfully requests that the Substitute Specification be entered. No new matter has been added.

Applicant believes that the stated grounds of objection and rejection set forth by the Examiner have been properly accommodated or addressed.

Dated: June 3, 2024

Respectfully submitted,

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METHODS FOR MAKING L-GLUFOSINATE

CROSS-REFERENCE TO PRIORITY APPLICATIONS

This application is a continuation of U.S. Application No. 18/179,136, filed March 6, 2023, which is a continuation of U.S. Application No. 17/530,018, filed November 18, 2021, which is a continuation of U.S. Application No. 16/997,133, filed August 19, 2020, now U.S. Patent No. 11,560,557, which is a continuation of U.S. Application No. 16/287,290, filed February 27, 2019, now U.S. Patent No. 10,781,465, which is a continuation of U.S. Application No. 15/787,448, filed October 18, 2017, now U.S. Patent No. 10,260,078, which is a division of U.S. Application No. 15/445,254, filed February 28, 2017, now U.S. Patent No. 9,834,802, which claims the benefit of U.S. Provisional Application No. 62/302,421, filed March 2, 2016; U.S. Provisional Application No. 62/336,989, filed May 16, 2016; and U.S. Provisional Application No. 62/413,240, filed October 26, 2016. The aforementioned applications are hereby incorporated herein by reference in their entireties.

INCORPORATION BY REFERENCE OF MATERIAL SUBMITTED ELECTRONICALLY

The Sequence Listing, which is a part of the present disclosure, is submitted concurrently with the specification as an .xml file. The name of the file containing the Sequence Listing is "M202647G_SubSeqListing.xml ~~M202647G_Seqlisting.xml~~", which was created on April 17, 2024 ~~December 20, 2023~~ and is 5,116 bytes ~~5,022 bytes~~ in size. The subject matter of the Sequence Listing is incorporated herein in its entirety by reference.

FIELD

Described herein are methods for producing a single stereoisomer of glufosinate, particularly for the production of L-glufosinate.

BACKGROUND

The herbicide glufosinate is a non-selective, foliarly-applied herbicide considered to be one of the safest herbicides from a toxicological or environmental standpoint. Current commercial chemical synthesis methods for glufosinate yield a racemic mixture of L- and D-glufosinate (Duke *et al.* 2010 *Toxins* 2:1943-1962). However, L-glufosinate (also known as phosphinothricin or (S)-2-amino-4-(hydroxy(methyl)phosphonoyl)butanoic acid) is much more potent than D-glufosinate (Ruhland *et al.* (2002) *Environ. Biosafety Res.* 1:29–37).

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Therefore, methods are needed to produce only or primarily the active, L-glufosinate form. Previously, cost effective methods to generate pure L-glufosinate, or a mixture of D- and L-glufosinate enriched for L-glufosinate, have not been available. Described herein are new and cost-effective methods for the production of L-glufosinate.

5

SUMMARY

Compositions and methods for making L-glufosinate are provided. The first step of the process involves the oxidative deamination of D-glufosinate to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). The second step involves the specific amination of PPO to L-glufosinate, using an amine group from one or more amine donors. In some
10 embodiments, the method involves reacting D-glufosinate with a D-amino acid oxidase (DAAO) enzyme to form PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid); followed by aminating the PPO to L-glufosinate by a transaminase (TA) enzyme, using an amine group from one or more amine donors, wherein at least 70% of the D-glufosinate is eliminated and/or the yield of L-glufosinate is at least 85% of the input racemic glufosinate or at least 70 to 85% of the D-
15 glufosinate is converted to L-glufosinate. In some embodiments, unreacted amine donor from one reaction can be reused in further rounds of reaction. Optionally, the D-glufosinate is originally present (i.e., in the reacting step) in a racemic mixture of D- and L-glufosinate.

The DAAO enzyme must have an increased activity of about 3 $\mu\text{mol}/\text{min} \cdot \text{mg}$ or greater to drive the reaction. DAAO enzymes are available in the art and can be modified or mutated to
20 have the necessary increased activity needed to drive the process. In this manner, mutant or modified enzymes from *Rhodospiridium toruloides* (UniProt P80324), *Trigonopsis variabilis* (UniProt Q99042), *Neolentinus lepideus* (GenBank KZT28066.1), *Trichoderma reesei* (GenBank XP_006968548.1), or *Trichosporon oleaginosus* (KLT40252.1) can be used. In some embodiments, the DAAO enzyme is a mutant DAAO based on the sequence from
25 *Rhodospiridium toruloides*. While a number of mutations can be made and tested for the effect on activity, the mutant DAAO in some embodiments may comprise mutations at positions 54, 56, 58, 213, and/or 238. For example, the mutant DAAO can comprise one or more of the following mutations at position 54: N54C, N54L, N54T, or N54V. The mutant DAAO can optionally comprise the following mutation at position 56: T56M. The mutant DAAO can
30 optionally comprise one or more of the following mutations at position 58: F58A, F58G, F58H, F58K, F58N, F58Q, F58R, F58S, or F58T. Optionally, the mutant DAAO can comprise the

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following mutation at position 213: M213S. In some embodiments, the mutant DAAO can comprise one or more of the following combinations of mutations: F58K and M213S; N54T and T56M; N54V and F58Q; and/or N54V, F58Q, and M213S. In each case, the enzyme needs to have an activity of equal to or greater than about 3 $\mu\text{mol}/\text{min}\cdot\text{mg}$, greater than about 4
5 $\mu\text{mol}/\text{min}\cdot\text{mg}$, or higher. A wild type enzyme can be used in the methods of the invention as long as the enzyme has an activity level as set forth above.

The TA enzyme may be a gabT transaminase from *Escherichia coli* (UniProt P22256). Alternatively, the TA enzyme may be a transaminase with the sequence identified as SEQ ID NO: 1. The TA enzyme may also be selected on the basis of sequence similarity to SEQ ID NO:
10 1 and/or mutated to improve its activity in the desired reaction. Thus, sequences having 80%, 85%, 90%, 95% or greater sequence identity to SEQ ID NO: 1, based on the BLASTP method of alignment, and retain transaminase activity are encompassed by the present invention. Any DNA sequence encoding the enzyme sequence of SEQ ID NO: 1 or variants thereof are encompassed herein as well.

15 The reacting step and the aminating step can be performed in a single container or in separate containers. In one embodiment, all reagents are substantially added at the start of the reaction. Alternatively, the reagents for the reacting step and the reagents for the aminating step are added to the single container at different times.

Also described herein is a method for selectively controlling weeds in a field containing a
20 crop of planted seeds or crops that may optionally be resistant to glufosinate, comprising applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate to the field. Also described herein is a method for selectively controlling weeds in a field, controlling weeds in non-field areas, defoliating plants or crops, and/or desiccating crops before harvest, comprising applying an effective amount of a
25 composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate and more than 0.01% but less than 15% PPO to the field.

The details of one or more embodiments are set forth in the drawings and the description below. Other features, objects, and advantages will be apparent from the description and drawings, and from the claims.

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Some compositions of the invention comprise D-glufosinate, PPO, and L-glufosinate. In such compositions, L-glufosinate is present at a concentration equal to or greater than 80%, greater than 90%, greater than 95%, greater than 96%, greater than 97%, greater than 98%, or greater than 99% by weight of the composition, based on the combined weight of D-glufosinate, PPO, and L-glufosinate. Other compositions comprise L-glufosinate at concentrations equal to 80% or greater after isolation of the L-glufosinate from the present reaction mixture.

DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic of an exemplary conversion of D-glufosinate to L-glufosinate. The amine donor and keto acid product are examples and are not intended to be limiting.

Figure 2 is a graph showing concentrations of L-glufosinate (circles), D-glufosinate (triangles), and PPO (squares) during a one-step de-racemization by a N54T, T56M, F58K, and M213S mutant variant of *Rhodospiridium toruloides* DAAO and the *E. coli* gabT transaminase.

DETAILED DESCRIPTION

Compositions and methods for the production of L-glufosinate (also known as phosphinothricin or (S)-2-amino-4-(hydroxy(methyl)phosphonoyl)butanoic acid) are provided. The methods comprise a two-step process, which may optionally occur in a single vessel and nearly simultaneously. The first step involves the oxidative deamination of D-glufosinate to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). The second step involves the specific amination of PPO to L-glufosinate, using an amine group from one or more amine donors. By combining these two reactions, the proportion of L-glufosinate can be substantially increased in a racemic glufosinate mixture. Thus, provided herein are methods to obtain a composition consisting substantially of L-glufosinate. Since L-glufosinate is more potent than D-glufosinate, smaller amounts of the composition are needed to be effective as a herbicide.

In one embodiment, described herein, is a composition comprising a mixture of L-glufosinate, PPO, and D-glufosinate, where L-glufosinate is the predominant compound among the mixture of L-glufosinate, PPO, and D-glufosinate. Such composition can be used directly as a herbicide as PPO can contribute herbicidal activity (EP0030424). In other embodiments, L-glufosinate can be purified or substantially purified and used as a herbicide.

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Compositions of L-glufosinate may comprise D-glufosinate, PPO, and L-glufosinate. Optionally, the amount of L-glufosinate is 80% or greater, 85% or greater, 90% or greater, or about 95% or greater, 97% or greater, 98% or greater based on the combined weight of D-glufosinate, PPO, and L-glufosinate. Optionally, the amount of D-glufosinate is 10% or less, 5% or less, 2.5% or less, or 1% or less based on the combined weight of D-glufosinate, PPO, and L-glufosinate. Optionally, the amount of PPO is more than 1% but less than 20%, less than 15%, less than 10%, or less than 5% based on the weight of D-glufosinate, PPO, and L-glufosinate. These compositions can optionally occur as dried powders or dissolved in aqueous or non-aqueous carrier and additional chemical species can optionally be present. Optionally, the composition is prepared and used in an ex vivo environment.

It is also recognized that the L-glufosinate can be further isolated and used in formulations as a herbicide.

Also described herein are formulations. The formulations comprise L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; one or more additional components selected from the group consisting of sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; dipropylene glycol in an amount from 4-18% by weight of the formulation; and alkyl polysaccharide in an amount from 4-20% by weight of the formulation; and water as the balance of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 24.5% by weight of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the

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formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation. Optionally, the formulation comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; alkylethersulfate, sodium salt in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1.0% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

While the methods can be used to produce a substantially purified L-glufosinate in a batch reaction, it is recognized that a continuous process can be used.

I. Methods of Synthesis

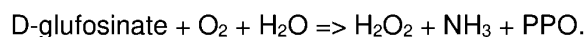
Methods for the conversion of D-glufosinate to L-glufosinate are provided. The methods described herein provide a means for converting a low cost feedstock of a racemic mixture of D- and L-glufosinate into a more valuable product that has been enriched for L-glufosinate. The methods for conversion includes two steps, which can occur in one or more separate containers. The first step is the oxidative deamination of D-glufosinate (which can be present in a racemic mixture of D- and L-glufosinate) to PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid). This step can be catalyzed by a D-amino acid oxidase (DAAO) enzyme, a D-amino acid dehydrogenase (DAAD) enzyme, or by chemical conversion. The second step is the specific amination of PPO to L-glufosinate, using an amine group from one or more amine donors. Such amine donors can be selected from glutamate, L-glutamate, lysine, alanine, isopropylamine, sec-butylamine, phenylethylamine and the like. This step can be catalyzed by a transaminase (TA) enzyme, an L-amino acid dehydrogenase (LAAD) enzyme, or by chemical conversion. Using the methods described herein, compositions of substantially purified L-glufosinate can be obtained.

Figure 1 sets forth an example of the conversion of D-glufosinate to L-glufosinate. As noted above, the method involves a two-step process. As illustrated, the first step is an oxidative deamination of D-glufosinate to PPO and the second step is an amination of PPO to L-glufosinate.

The first step, i.e., the oxidative deamination of D-glufosinate to PPO, can be catalyzed by several classes of enzymes or can occur non-enzymatically. Such enzymes include DAAO, DAAD, and D-amino acid dehydratase.

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In one embodiment a DAAO enzyme is used to catalyze the conversion of D-glufosinate to PPO. Such a reaction has the following stoichiometry:



Since the solubility of oxygen in aqueous reaction buffer is typically low compared to that
 5 of glufosinate, for an efficient process, oxygen must be introduced throughout the time period of the DAAO reaction. This is in contrast to, for example, the Hawkes reaction set forth in US Patent Nos: 7,723,576; 7,939,709; 8,642,836; and 8,946,507 in which the reaction was conducted in a sealed vessel. Initially, D-glufosinate is present at greater than 30 g/L up to as much as 140 g/L. The initial oxygen level is typically impacted by the reaction temperature, but
 10 is typically initially present at approximately 8 mg/L and is added throughout the reaction to allow for sufficient oxygen for the reaction to continue apace. Water is typically, but not obligately, present at greater than 500 g/L.

Several DAAO enzymes are known in the art and can be used in the methods described herein, as long as they are capable of accepting D-glufosinate as a substrate and provide an
 15 activity sufficient to level to drive the reaction. The DAAO enzymes useful in the methods of the invention have an activity of equal to or greater than about 3 umol/min*mg, greater than about 4 umol/min*mg, or higher. A wild type enzyme can be used in the methods of the invention as long as the enzyme has an activity level as set forth above. Such DAAO enzymes that can be used in the method include those from *Rhodospiridium toruloides*, *Trigonopsis variabilis*,
 20 *Fusarium sp*, *Candida sp*, *Schizosaccharomyces sp*, *Verticillium sp*, *Neolentinus lepideus*, *Trichoderma reesei*, *Trichosporon oleaginosus*, and the like that have been modified to increase activity. Any DAAO enzyme can be used as a starting enzyme including those having sequences corresponding to Swissprot accession numbers P80324, Q99042, P00371, and P24552 or SPTREMBL numbers Q9HGY3 and Q9Y7N4 or GenBank numbers KZT28066.1, XP_006968548.1, and KLT40252.1. The DNA sequences which encode the DAAO may be
 25 selected from sequences set forth in EMBL accessions A56901, RGU60066, Z50019, SSDA04, D00809, AB042032, RCDAAOX, A81420, and SPCC1450, or may be codon optimized from the protein sequences indicated above for optimal expression in the chosen expression host(s). U.S. Patent No. 8,227,228 describes DAAO enzymes from *Candida intermedia*. Such
 30 sequences are herein incorporated by reference. These enzymes can be modified for increased activity and used in the methods of the invention.

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Additional DAAO enzymes can be identified in a variety of ways, including sequence similarity and functional screens. The DAAO enzyme may be a mutant DAAO enzyme that is capable of accepting D-glufosinate as a substrate. In Hawkes *et al.*, *supra*, a mutant DAAO based on the sequence from *Rhodospiridium toruloides* (consisting of the F58K and M213S mutations) has been shown to accept D-glufosinate as a substrate (Hawkes *et al.* (2011) *Plant Biotechnol J.* 9(3):301-14). Other DAAO enzymes can be similarly modified to accept D-glufosinate and have greater activity. i.e., the activity needed to drive the method of the invention. In the same manner, known DAAO enzymes may be improved by mutagenesis, and/or novel DAAO enzymes could be identified.

In some embodiments, mutant enzymes can be made and tested in the methods described herein. Mutant DAAO enzymes (e.g., from *Rhodotorula gracilis*) can include one mutation, two mutations, three mutations, or more than three mutations (e.g., four mutations, five mutations, six mutations, seven mutations, eight mutations, nine mutations, or ten mutations or more) at positions in the mutant sequence as compared to the wild type sequence. The mutant DAAO can optionally comprise mutations at positions 54, 56, 58, 213, and/or 238. In some embodiments, such mutants can comprise amino acid substitutions at positions 54 and 56 when compared with the wild type sequence. In other embodiments, such mutants can comprise amino acid substitutions at positions 54 and 58 when compared to the wild type sequence. In other embodiments, such mutants can include amino acid substitutions at positions 54, 213, and 238 when compared with the wild type sequence.

Optionally, at position 54, the wild type asparagine may be replaced by Ala, Cys, Gly, Ile, Ser, Leu, or, more preferably, Thr or Val. For example, the mutant DAAO can comprise one of the following mutations at position 54: N54C, N54L, N54T, or N54V.

Optionally, at position 56, the wild type threonine can be replaced by Ala, Cys, Gly, Ile, Asn, Arg, Ser, Thr, Met, or Val. See, U.S. Patent No. 7,939,709, which is incorporated herein by reference. For example, the mutant DAAO can comprise the T56M mutation.

Additionally, at position 58, the wild type Phe can be replaced by Lys, Arg, Gln, Thr, Gly, Ser, Ala, Arg, Asn, or His. The mutant DAAO can optionally comprise one of the following mutations at position 58: F58A, F58G, F58H, F58K, F58N, F58Q, F58R, F58S, or F58T. In some embodiments, the mutant DAAO does not include a mutation at position 58.

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Optionally, at position 213, the wild type methionine is replaced by Arg, Lys, Ser, Cys, Asn, or Ala. In some examples, the mutant DAAO can comprise the mutation M213S.

Optionally, at position 238, the wild type tyrosine is replaced by His, Ser, Gys, Asn, or Ala.

5 In some embodiments, the mutant DAAO can comprise one or more of the following combinations of mutations: F58K and M213S; N54T and T56M; N54V and F58Q; N54C and F58H; N54T and F58T; N54T and F58G; N54T and F58Q; N54T and F58A; N54L and F58R; N54V and F58R; N54V and F58N; and/or N54V, F58Q, and M213S.

10 In one embodiment, the mutant DAAO comprises mutations in other DAAO enzymes in positions equivalent to positions 54, 56, 58, 213, and/or 238 of *Rhodospiridium toruloides* DAAO or *Trigonopsis variabilis* DAAO.

Other suitable D amino acid oxidases may be obtained preferably from fungal sources. Such DAAO enzymes can be identified and tested for use in the methods of the invention. To determine if the enzyme will accept D-glufosinate as a substrate, an oxygen electrode assay
15 (Hawkes, 2011, *supra*), colorimetric assay (Berneman A, Alves-Ferreira M, Coatnoan N, Chamond N, Minoprio P (2010) Medium/High Throughput D-Amino Acid Oxidase Colorimetric Method for Determination of D-Amino Acids. Application for Amino Acid Racemases. *J Microbial Biochem Technol* 2: 139-146), and/or direct measurement (via high performance liquid chromatography (HPLC), liquid chromatography mass spectrometry (LC-MS), or similar) of
20 product formation can be employed.

The reaction catalyzed by the DAAO enzyme requires oxygen. In some embodiments, oxygen, oxygen enriched air, an oxygen enriched gas stream, or air, is introduced to the reaction, either in the head space or by sparging gas through the reaction vessel, intermittently or continuously, to enhance the rate of reaction. Additionally, in other embodiments, optionally
25 combined with sparging gas through the reaction vessel, a pressurized reactor may be used. That is, the reactor may be sealed and allowed to consume O₂. Using a sealed chamber would limit vapor emissions.

When a DAAO enzyme catalyzes the conversion of D-glufosinate to PPO, hydrogen peroxide (H₂O₂) evolves. This may be damaging to enzymes and other components of the

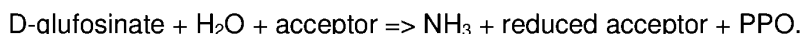
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biotransformation (e.g., products and/or substrates). Therefore, in one embodiment, an enzyme, such as catalase, can be used in addition to the DAAO enzyme to catalyze the elimination of hydrogen peroxide. Catalase catalyzes the decomposition of hydrogen peroxide with the following stoichiometry:



In some embodiments, hydrogen peroxide can be eliminated using catalyzed and non-catalyzed decomposition reactions. For example, hydrogen peroxide can be eliminated by a non-catalyzed decomposition reaction using increased heat and/or pH. Hydrogen peroxide can also be eliminated by a catalyzed decomposition reaction using, for example, transition metals
10 and other agents, such as potassium iodide. In addition to eliminating hydrogen peroxide, the use of catalase also produces oxygen (O_2). The production of oxygen by catalase can aid in facilitating the conversion of D-glufosinate to PPO using the DAAO enzyme, as DAAO requires oxygen to function.

Other enzymes can be used to catalyze the conversion of D-glufosinate to PPO. For
15 example, a DAAD enzyme that accepts D-glufosinate as a substrate can be used with the following stoichiometry:



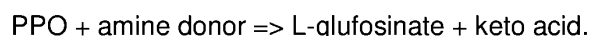
It is recognized that in methods where a DAAD is used, the DAAD catalyzed reaction can include redox cofactor recycling. This involves oxidizing the reduced acceptor so that it can
20 accept more electrons from D-glufosinate.

In one embodiment, chemical oxidative deamination, wherein an intermediate α -keto acid is produced from the parent amino acid, can be used in the methods described herein to convert D-glufosinate to L-glufosinate. Chemical oxidative deamination involves the conversion of an amine group to a keto group with concomitant release of ammonia typically using metal
25 ions such as those of copper or cobalt in an aqueous solution at temperatures between room temperature and the boiling point of the solution and at a pH in the range of about 4 – about 10. See, for example, Ikawa and Snell (1954) *J. Am. Chem. Soc.* 76 (19): 4900–4902, herein incorporated by reference.

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The substantially complete (greater than 70%, greater than 75%, greater than 80%, greater than 85%, greater than 90%, or greater than 95%) conversion of D-glufosinate to PPO can occur within 24 hours, within 18 hours, within 12 hours, within 8 hours or less.

The second step of the method described herein involves the conversion of PPO to L-glufosinate using a transaminase (TA) enzyme, an L-amino acid dehydrogenase (LAAD) enzyme, or by chemical conversion. In one embodiment, the method is a reaction catalyzed by a TA. A TA with the required stereospecificity that accepts PPO as a substrate catalyzes the amination of PPO to L-glufosinate with the following stoichiometry:



If the reaction is conducted as a two stage process where the D-glufosinate is substantially converted to PPO in the absence of amine donor and/or transaminase, starting amounts of PPO in the second stage typically range from 10g/L to 140g/L; 20g/L to 140g/L; or from 30g/L to 140g/L. If the reaction is conducted in a single stage process, the starting amounts of PPO are typically less than 1 g/L and the highest levels of PPO during the reaction are typically less than 25 g/L. The amine donor is initially present at between 1 and 50 fold molar excess over the starting amount of racemic glufosinate.

TAs useful in the methods described herein include the gabT transaminase from *Escherichia coli* (UniProt P22256; identified herein as SEQ ID NO: 3), which has been shown to catalyze the desired reaction with PPO as a substrate (Bartsch *et al.* (1990) *Appl Environ Microbiol.* 56(1):7-12). Another enzyme has been evolved to catalyze the desired reaction at a higher rate using isopropylamine as an amine donor (Bhatia *et al.* (2004) *Peptide Revolution: Genomics, Proteomics & Therapeutics*, Proceedings of the Eighteenth American Peptide Symposium, Ed. Michael Chorev and Tomi K. Sawyer, July 19-23, 2003, pp. 47-48). A transaminase with the amino acid sequence of SEQ ID NO: 1 also catalyzes the desired reaction with PPO and isopropylamine as the substrate (Example 11). Additionally, TA enzymes from numerous microorganisms, such as *Streptomyces hygroscopicus*, *Streptomyces viridochromogenes*, *Candida albicans*, and others can be used in the practice of the methods described herein. In particular, see, for example, EP0249188, and U.S. Patent No. 5,162,212, incorporated herein by reference. Where desired, the enzymes can be evolved by mutagenesis to increase their activities. Mutant TA enzymes can be selected for desired activity by the

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assays outlined in Schulz *et al.*, Appl Environ Microbiol. (1990) Jan. 56(1):1-6, and/or by direct measurement of the products by HPLC, LC-MS, or similar products.

Additional TA enzymes for use in the methods can be identified by screening collections of TAs, such as those sold by Prozomix Limited (Northumberland, United Kingdom),
5 SyncoZymes (Shanghai, China), Evocatal (Monheim am Rhein, Germany), Codexis (Redwood City, CA), or Abcam (Cambridge, United Kingdom) for the desired activity. Alternatively, sequence similarity can be used to identify novel TA enzymes. Finally, TA enzymes can also be identified from organisms capable of catalyzing the desired reaction.

The selection of an appropriate amine donor is important for an economical conversion
10 of D-glufosinate to L-glufosinate. A variety of issues may be considered, including the cost of the donor, equilibrium thermodynamics, potential recovery of the donor, separation of the keto acid product from the desired L-glufosinate, and others. Consequently, TA enzymes that accept several different amine donors can be used, including low cost amine donors such as L-aspartate or racemic aspartate, L-glutamate or racemic glutamate, L-alanine or racemic alanine,
15 L-phenylethylamine or racemic phenylalanine, L-glycine or racemic glycine, L-lysine or racemic lysine, L-valine or racemic valine, L-serine or racemic serine, L-glutamine or racemic glutamine, isopropylamine, sec-butylamine, ethanolamine, 2-aminobutyric acid, and diaminopropionic acid. In some embodiments, the amine donor is not aspartate or aspartic acid (e.g., L-aspartic acid, D-aspartic acid, or racemic D,L-aspartic acid).

20 In embodiments where the amino donor is glutamate, the keto acid co-product that results from the transamination reaction is α -ketoglutarate (which is also referred to as α -ketoglutaric acid or α -KG). The α -ketoglutarate can be isolated and/or purified using methods known to those of skill in art, such as in EP Patent No. 0073711, CN Patent No. 10519873, CN Patent No. 105177065, CN Patent No. 104529755, and Zhan et al., *Shipin Yu Shengwu Jishu Xuebao*, 32(10): 1043-1048 (2013), each of which are incorporated herein by reference in their
25 entireties. The produced and isolated α -ketoglutarate can be used in a variety of applications, including in synthesizing pharmaceutical agents, food additives, and biomaterials. Optionally, the α -ketoglutarate can be chemically converted to either racemic glutamate or L-glutamate, optionally for reuse in the reaction.

30 Chemical reductive amination involves the conversion of a keto group to an amine group via an imine compound typically by treatment of the keto compound with a suitable amine in an

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organic solution. Suitable amines include, for example, methylamine or ammonia. Suitable organic solvents for use in the organic solution include tetrahydrofuran, ethanol, or dichloromethane (DCM). The reductive amination can be performed at temperatures between room temperature and the boiling point of the solution. The produced imine can then be

5 reduced using a reducing agent in an organic solution. Suitable reducing agents include, for example, NaBH_4 , NaHB(OAc)_3 , or Na(CN)BH_3 . Suitable organic solvents for use in the organic solution include tetrahydrofuran, ethanol, or DCM. The reduction reaction can be performed at temperatures between 0°C and the boiling point of the solution. Those skilled in the art will know that the process may be done in "one-pot" or in multiple containers, i.e., in separate

10 transformations. Additionally, those skilled in the art will recognize that the described procedure will provide racemic amino material where possible. The use of a chiral reducing agent such as $\text{RuCl}_2[(S)\text{-BINAP}]$ and hydrogen gas or latent source of hydrogen gas, or achiral hydride based reducing agents in the presence chiral ligands such as $(S)\text{-}$ or $(R)\text{-VAPOL}$ in a ratio between 1:1 to 1:0.05 can produce enantiomerically pure and/or enriched amino material. See, for example,

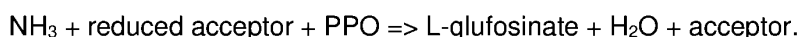
15 Mignonac (1921) *Compt. Rend.* 172:223 and G. Li, Y. Liang, J. C. Antilla (2007) *J. Am. Chem. Soc.*, 129:5830-5831.

A wild type TA that accepts a desired amine donor can be identified, or a TA that does not normally accept a desired amine donor can be evolved to accept the desired substrate. Optionally, the transaminase is not an aspartate transaminase. Optionally, the transaminase is

20 not 4-amino-butyrate: 2-ketoglutarate transaminase. In some embodiments, the transaminase is not a combination enzyme system that includes a PPT-specific transaminase and glutamate:oxaloacetate transaminase.

Other enzymes for use to catalyze the conversion of PPO to L-glufosinate include LAAD enzymes or imine reductases that accept PPO as a substrate. Such LAAD enzymes use the

25 following stoichiometry:



LAAD catalyzed reactions can include redox cofactor recycling, which involves reducing the oxidized acceptor so that it can donate more electrons to PPO.

Chemical reductive amination, wherein an amino group is produced from the parent

30 keto-compound, can also be used to produce glufosinate, in the case where no chiral reductant

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or ligand is used, or L-glufosinate, in the case where a chiral reductant or ligand is used.

Chemical reductive amination can be affected as described above.

The substantially complete conversion of PPO to L-glufosinate may occur within 24 hours, within 18 hours, within 12 hours, within 8 hours, or within 4 hours. Substantially
5 complete, in this context, means that the conversion of PPO to L-glufosinate is greater than about 70%, greater than about 75%, greater than about 80%, greater than about 85%, greater than about 90%, greater than about 95%, greater than about 98%, or greater than about 99%.

If the reaction occurs in a single container or vessel, the TA enzyme can be added with the DAAO enzyme or added at a later time, e.g., after the DAAO enzyme has been allowed to
10 catalyze some or substantially all of the oxidative deamination.

Enzymes can be added to the reaction by a number of methods. One approach is to express the enzyme(s) in microorganism(s) such as *E. coli*, *S. cerevisiae*, *P. pastoris*, and others, and to add the whole cells to the reactions as whole cell biocatalysts. Another approach is to express the enzyme(s), lyse the microorganisms, and add the cell lysate. Yet another
15 approach is to purify, or partially purify, the enzyme(s) from a lysate and add pure or partially pure enzyme(s) to the reaction. If multiple enzymes are required for a reaction, the enzymes can be expressed in one or several microorganisms, including expressing all enzymes within a single microorganism.

A further approach, which can be combined with the above approaches, is to immobilize
20 enzyme(s) to a support (exemplary strategies are outlined in Datta *et al.* (2013) 3 Biotech. Feb; 3(1): 1–9). As outlined in Datta *et al.*, and not intending to be limiting, enzymes, either singly or in combination, can, for example, be adsorbed to, or covalently or non-covalently attached to, or entrapped within, natural or synthetic polymers or inorganic supports, including aggregates of the enzyme(s) themselves. Once immobilized, the enzyme(s) and support can be dispersed into
25 bulk solution or packed into beds, columns, or any number of similar approaches to interacting reaction solution with the enzymes. Since aeration is important for the DAAO reaction envisioned here, bubble columns or similar may be used for enzyme immobilization. As examples, reaction mixture can be flowed through a column of immobilized enzymes (flow reaction), added to a fixed bed or column of immobilized enzymes, allowed to react, and either
30 removed from the bottom or top of the reaction vessel (plug flow), or added to dispersed immobilized enzymes and allowed to react then the immobilized enzymes removed by filtration,

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centrifugation, or similar (batch). Thus, any method for immobilization of the enzymes may be employed in the methods of the invention.

The DAAO, TA, and/or other reactions can occur in a buffer. Exemplary buffers commonly used in biotransformation reactions include Tris, phosphate, or any of Good's buffers, such as 2-(N-morpholino)ethanesulfonic acid (MES); N-(2-Acetamido)iminodiacetic acid (ADA); piperazine-N,N'-bis(2-ethanesulfonic acid) (PIPES); N-(2-Acetamido)-2-aminoethanesulfonic acid (ACES); β -Hydroxy-4-morpholinepropanesulfonic acid (MOPSO); choline chloride; 3-(N-morpholino)propanesulfonic acid (MOPS); N,N-Bis(2-hydroxyethyl)-2-aminoethanesulfonic acid (BES); 2-[[1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl]amino]ethanesulfonic acid (TES); 4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid (HEPES); 3-(Bis(2-hydroxyethyl)amino)-2-hydroxypropane-1-sulfonic acid (DIPSO); acetamidoglycine, 3-(N-Tris(hydroxymethyl)methylamino)-2-hydroxypropanesulfonic acid (TAPSO); Piperazine-N,N'-bis(2-hydroxypropanesulfonic acid) (POPSO); 4-(2-Hydroxyethyl)piperazine-1-(2-hydroxypropanesulfonic acid) (HEPPSO); 3-[4-(2-Hydroxyethyl)-1-piperazinyl]propanesulfonic acid (HEPPS); tricine; glycine; bicine; or 3-[[1,3-dihydroxy-2-(hydroxymethyl)propan-2-yl]amino]propane-1-sulfonic acid (TAPS). Additional exemplary buffer recipes can be found in Whittall, J. and Sutton, P. W. (eds) (2012) Front Matter, in Practical Methods for Biocatalysis and Biotransformations 2, John Wiley & Sons, Ltd, Chichester, UK. In some embodiments, ammonium can act as a buffer. One or more organic solvents can also be added to the reaction.

Surprisingly, the DAAO, TA, and/or other reactions can occur with no or low levels (less than 1 mM) of buffer added (other than ammonium that may optionally be present due to addition of racemic glufosinate ammonium). In particular, immobilized DAAO and TA may be stable and active in the presence of less than 1 mM phosphate buffer and with no other buffer except any ammonium present due to the addition of racemic glufosinate ammonium.

The racemic glufosinate starting material can be provided in a number of forms. Various salts of racemic glufosinate, such as ammonium and hydrochloride, or the zwitterion, can be used. The racemic glufosinate may be in the form of a solid powder (such as a powder of greater than 80%, 85%, 90%, or 95% purity) or an aqueous solution (such as a roughly 50% solution of racemic glufosinate).

In some embodiments, the reaction occurs within a defined pH range, which can be between pH 4 to pH 10 (e.g., between pH 6 and pH 9, such as approximately pH 7.5 to pH 8).

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In some embodiments, the reaction occurs at a defined temperature. The temperature can be kept at a point between room temperature and the boiling point of the solvent, most typically between room temperature and 50 °C.

As indicated, the methods described herein provide a composition of substantially pure
5 L-glufosinate (rather than a racemic mixture of L-glufosinate and D-glufosinate). Substantially
pure L-glufosinate means that greater than about 70%, greater than about 75%, greater than
about 80%, greater than about 85%, greater than about 90%, greater than about 95%, greater
than about 96%, greater than about 97%, greater than about 98%, or greater than about 99% of
the D-glufosinate has been converted to L-glufosinate resulting in a composition having greater
10 than about 80%, greater than about 85%, greater than about 90%, greater than about 95%,
greater than about 96%, greater than about 97%, greater than about 98%, or greater than about
99% L-glufosinate compared to the sum of the D-glufosinate and the L-glufosinate present in
the composition.

In one embodiment, the L-glufosinate is not isolated from the biotransformation mixture
15 and a composition comprising D-glufosinate, PPO, and L-glufosinate is obtained. This
composition will contain at least 80% L-glufosinate by weight of the sum of L-glufosinate, D-
glufosinate, and PPO, at least 90% L-glufosinate by weight of the sum of the components. This
composition may be used directly as a herbicidal composition or as an ingredient in a
formulated herbicidal product.

20 Alternatively, some or all of the components other than L-glufosinate can be removed
from the biotransformation mixture, the mixture optionally concentrated, and then the mixture
can be used directly (and/or with the addition of various adjuvants) for the prevention or control
of weeds. The biotransformation mixture, in some instances, can be used directly (and/or with
the addition of various adjuvants) for the prevention or control of weeds.

25 Additional steps to further purify the L-glufosinate can be added. Such further
purification and isolation methods include ion exchange, extraction, salt formation, crystallization
and filtration; each may be used multiple times or in suitable combination. Enzymes can be
removed by simple filtration if supported, or if free in solution by the use of ultrafiltration, the use
of absorbants like celite, cellulose or carbon, or denaturation via various techniques known to
30 those skilled in the art.

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Ion exchange processes effect separation by selective adsorption of solutes onto resins chosen for this purpose. Because products and impurities must be dissolved in a single solution prior to adsorption, concentration of the purified product stream by evaporation or distillation prior to isolation is usually required. Examples of the use of ion exchange for purification are
5 described by Schultz et al., and in EP0249188(A2).

Purification may be achieved by the formation of an insoluble salt of L-glufosinate by the addition of a suitable acid, including hydrochloric acid, sulfuric acid, phosphoric acid, nitric acid, acetic acid and the like. Similarly, the purification may be achieved by the addition of a suitable base to form an insoluble salt. Useful bases include hydroxides, carbonates, sulfates and
10 phosphates of alkali metals or hydroxides, carbonates, sulfates and phosphates of alkali earth metals. Other bases which contain nitrogen may be used, including ammonia, hydroxylamine, isopropylamine, triethylamine, tributylamine, pyridine, 2-picoline, 3-picoline, 4-picoline, 2,4-lutidine, 2,6-lutidine, morpholine, N-methymorpholine, 1,8-diazabicyclo[5.4.0]undec-7-ene, and dimethylethanolamine. It may be advantageous to concentrate the mixture or to add a solvent
15 (or both) to maximize yield and optimize purity of the desired salt. Solvents suitable for this purpose include those in which the solubility of the desired salt is very low (such solvents are often called "anti-solvents"). Salts of L-glufosinate can be transformed into forms of glufosinate suitable for formulation by standard methods known to those skilled in the art. Alternatively, the L-glufosinate can be isolated as a zwitterion.

US 9,255,115 B2 describes how the hydrochloric acid salt of L-glufosinate can be
20 converted to the zwitterionic form with a base such as sodium hydroxide or sodium methoxide and then crystallized from aqueous alcohol solvent to afford L-glufosinate in relatively high purity. This method has the advantage of producing crystalline L-glufosinate that is not hygroscopic and therefore maintains a higher purity compared to amorphous L-glufosinate when
25 exposed to humidity over time.

Other salts of L-glufosinate are known in the art. US 5,767,309 and US 5,869,668 teach the use of chiral alkaloid bases to form diastereomeric salts with racemic glufosinate. Purification is achieved because the salt of L-glufosinate precipitates from solution in much larger quantity than the corresponding salt of D-glufosinate. Therefore this method could be
30 used with the present invention to obtain L-glufosinate with high enantiomeric excess, if desired.

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Optionally, purification may be achieved by first crystallizing one or more impurities, removing the impurities by filtration and then further purifying L-glufosinate from the resulting filtrate by forming a salt as previously described. This is advantageous if unreacted amine donor can be partially or completely isolated and used in subsequent reactions. Similarly, unreacted PPO that is partially or completely isolated may be recycled for use in subsequent reactions.

Extraction may be used to purify the product. DE 3920570 C2 describes a process in which excess glutamic acid (used as the amine donor) is precipitated by adjusting the solution pH to 3.7 to 4.2 with sulfuric acid. After filtering the glutamic acid, the filtrate pH is lowered to 1-2 whereupon other impurities are extracted into a solvent. After extraction and concentration, ammonia is added to the aqueous solution to a pH of 5-7 whereupon ammonium sulfate precipitates. The ammonium sulfate is removed by filtration and the resulting filtrate is concentrated to afford the ammonium salt of L-glufosinate.

Isolation of L-glufosinate or its salts may be desirable, for example, for the purpose of shipping solids to the location of formulation or use. Typical industrial methods of isolation may be used, for example, a filtration, centrifugation, etc. Isolated product often requires the removal of water, volatile impurities and solvents (if present) and typical industrial drying equipment may be used for this purpose. Examples of such equipment include ovens, rotating drum dryers, agitated dryers, etc. In some cases, it may be advantageous to use a spray dryer.

It is not necessary to produce a solid product after purification. This may be advantageous if the formulation of L-glufosinate is to occur at the same site used for L-glufosinate production. L-glufosinate and many of its salts are readily soluble in water, and water is a convenient liquid to use for formulating products. For example, the amine donor is isolated by filtration and the resulting filtrate is concentrated by distillation. The pH of the filtrate may be adjusted to a desirable value and the resulting solution may be used as is or blended with formulation ingredients. In another example, a slurry of L-glufosinate or one of its salts may be prepared as described above and isolated by filtration. The solid could be dissolved directly on the filter by adding water or a suitable solvent to obtain a solution of L-glufosinate.

SUBSTITUTE SPECIFICATION - MARKED**II. Compositions**

Also described herein are compositions comprising the reaction products described above. In some embodiments, the composition substantially includes L-glufosinate and acceptable cationic or anionic salt forms such as the hydrochloride, ammonium, or isopropylammonium salts. In some embodiments, the composition comprises a mixture of L-glufosinate, PPO, and D-glufosinate.

Optionally, L-glufosinate is the predominant compound among L-glufosinate, PPO, and D-glufosinate. For example, L-glufosinate can be present in the composition in an amount of at least 80% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 85 % by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 90% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 95% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 96% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 97% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, at least 98% by weight of the sum of L-glufosinate, PPO, and D-glufosinate, or at least 99% by weight of the sum of L-glufosinate, PPO, and D-glufosinate.

The composition can include PPO in an amount up to 20% by weight of the sum of L-glufosinate, PPO, and D-glufosinate. Optionally, the composition includes from 0.001% to 20% PPO (e.g., from 0.05% to 15% or from more than 0.01% to less than 5% PPO). For example, the composition can include PPO in an amount of less than 20%, less than 19%, less than 18%, less than 17%, less than 16%, less than 15%, less than 14%, less than 13%, less than 12%, less than 11%, less than 10%, less than 9%, less than 8%, less than 7%, less than 6%, less than 5%, less than 4%, less than 3%, less than 2%, less than 1%, less than 0.5%, less than 0.1%, or less than 0.01% by weight of the sum of the masses of L-glufosinate, PPO, and D-glufosinate.

D-Glufosinate can be present in the composition in an amount of 15% or less by weight of the sum of L-glufosinate, PPO, and D-glufosinate. For example, D-glufosinate can be present in an amount of 14 % or less, 13 % or less, 12 % or less, 11 % or less, 10 % or less, 9 % or less, 8 % or less, 7 % or less, 6 % or less, 5 % or less, 4 % or less, 3 % or less, 2 % or less, 1 % or less, or 0.5 % or less by weight of the sum of L-glufosinate, PPO, and D-glufosinate.

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In some embodiments, the composition can contain small amounts (e.g., about 10% or less, about 8% or less, about 5% or less, about 2% or less, or about 1% or less by weight of the composition) of D-glufosinate. In some embodiments, the composition can contain small amounts (e.g., about 15% or less, about 10% or less, about 8% or less, about 5% or less, about 2% or less, or about 1% or less by weight of the composition) of PPO.

The compositions described herein are useful for application to a field of crop plants for the prevention or control of weeds. The composition may be formulated as a liquid for spraying on a field. The L-glufosinate is provided in the composition in effective amounts. As used herein, effective amount means from about 10 grams active ingredient per hectare to about 1,500 grams active ingredient per hectare, e.g., from about 50 grams to about 400 grams or from about 100 grams to about 350 grams. In some embodiments, the active ingredient is L-glufosinate. For example, the amount of L-glufosinate in the composition can be about 10 grams, about 50 grams, about 100 grams, about 150 grams, about 200 grams, about 250 grams, about 300 grams, about 350 grams, about 400 grams, about 500 grams, about 550 grams, about 600 grams, about 650 grams, about 700 grams, about 750 grams, about 800 grams, about 850 grams, about 900 grams, about 950 grams, about 1,000 grams, about 1,050 grams, about 1,100 grams, about 1,150 grams, about 1,200 grams, about 1,250 grams, about 1,300 grams, about 1,350 grams, about 1,400 grams, about 1,450 grams, or about 1,500 grams L-glufosinate per hectare.

The herbicidal compositions (including concentrates which require dilution prior to application to the plants) described herein contain L-glufosinate (i.e., the active ingredient), optionally some residual D-glufosinate and/or PPO, and one or more adjuvant components in liquid or solid form.

The compositions are prepared by admixing the active ingredient with one or more adjuvants, such as diluents, extenders, carriers, surfactants, organic solvents, humectants, or conditioning agents, to provide a composition in the form of a finely-divided particulate solid, pellet, solution, dispersion, or emulsion. Thus, the active ingredient can be used with an adjuvant, such as a finely-divided solid, a liquid of organic origin, water, a wetting agent, a dispersing agent, an emulsifying agent, or any suitable combination of these. From the viewpoint of economy and convenience, water is the preferred diluent. However, not all the

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compounds are resistant to hydrolysis and in some cases this may dictate the use of non-aqueous solvent media, as understood by those of skill in the art.

Optionally, one or more additional components can be added to the composition to produce a formulated herbicidal composition. Such formulated compositions can include L-glufosinate, carriers (e.g., diluents and/or solvents), and other components. The formulated composition includes an effective amount of L-glufosinate. Optionally, the L-glufosinate can be present in the form of L-glufosinate ammonium. The L-glufosinate ammonium can be present in an amount ranging from 10 % to 30 % by weight of the formulated composition. For example, the L-glufosinate ammonium can be present in an amount of 10 %, 12 %, 14 %, 16 %, 18 %, 20 %, 22 %, 24 %, 26 %, 28 %, or 30 % by weight of the formulated composition. Optionally, the L-glufosinate ammonium is present in an amount of 12.25 % or of 24.5%

In some examples, the formulated composition can include one or more surfactants. A suitable surfactant for use in the formulated composition includes sodium alkyl ether sulfate. The surfactant can be present in an amount from 10 % to 40 % by weight of the formulated composition. For example, the surfactant can be present in an amount of 10 %, 12 %, 14 %, 16 %, 18 %, 20 %, 22 %, 24 %, 26 %, 28 %, 30 %, 32 %, 34 %, 36 %, 38 %, or 40 % by weight of the formulated composition. Optionally, the sodium alkyl ether sulfate is present in an amount of 11.05%, 15.8 %, 22.1%, or 31.6 %.

The formulated composition can optionally include one or more solvents (e.g., organic solvents). Optionally, the solvent can be 1-methoxy-2-propanol, dipropylene glycol, ethylene glycol, and mixtures thereof. The one or more solvents can be present in an amount ranging from 0.5 % to 20 % by weight of the formulated composition. For example, the total amount of solvents in the composition can be present in an amount of 0.5% to 18 %, 5 % to 15 %, or 7.5 % to 10 % by weight of the formulated composition.

Optionally, the solvent includes a combination of two solvents. For example, the solvents in the formulation can include 1-methoxy-2-propanol and dipropylene glycol. The 1-methoxy-2-propanol can be present, for example, in an amount of 0.5 % to 2 % by weight of the formulated composition. For example, the 1-methoxy-2-propanol can be present in the amount of 0.5 %, 0.6 %, 0.7 %, 0.8 %, 0.9 %, 1.0 %, 1.1 %, 1.2 %, 1.3 %, 1.4 %, 1.5 %, 1.6 %, 1.7 %, 1.8 %, 1.9 %, or 2.0 % by weight of the formulated composition. Optionally, the 1-methoxy-2-propanol is present in an amount of 0.5 % or 1.0 % by weight of the formulated composition.

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The dipropylene glycol can be present in an amount of from 4 % to 18 % by weight of the formulated composition. For example, the dipropylene glycol can be present in an amount of 4 %, 6 %, 8 %, 10 %, 12 %, 14 %, 16 %, or 18 % by weight of the formulated composition. Optionally, the dipropylene glycol is present in an amount of 4.3 % or 8.6 % by weight of the formulated composition.

The formulated composition can also include one or more polysaccharide humectants. Examples of suitable polysaccharide humectants include, for example, alkyl polysaccharides, pentoses, high fructose corn syrup, sorbitol, and molasses. The polysaccharide humectant, such as alkyl polysaccharide, can be present in the formulated composition in an amount ranging from 4 % to 20 % by weight of the formulated composition. For example, the total amount of polysaccharide humectant in the composition can be present in an amount of 4 % to 18 %, 4.5 % to 15 %, or 5 % to 10 % by weight of the formulated composition. In some examples, the total amount of polysaccharide humectant, such as the alkyl polysaccharide, present in the formulated composition can be 4 %, 5 %, 6 %, 7 %, 8 %, 9 %, 10 %, 11 %, 12 %, 13 %, 14 %, 15 %, 16 %, 17 %, or 18 %. Optionally, the alkyl polysaccharide can be present in an amount of 3.2%, 4.9 %, 6.2%, or 9.8 %.

A diluent can also be included in the formulated composition. Suitable diluents include water and other aqueous components. Optionally, the diluents are present in an amount necessary to produce compositions ready for packaging or for use.

In one example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.

In another example, the formulated composition includes L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.

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5 In another example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation.

10 In another example, the formulated composition includes L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

15 In another example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 58.45% by weight of the formulation.

20 In another example, the formulated composition includes L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 11.05% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; alkyl polysaccharide in an amount of 3.1% by weight of the formulation; and water in an amount of 73.1% by weight of the formulation.

Further components suitable for use in the formulated compositions provided herein are described in U.S. Patent Nos. 4,692,181 and 5,258,358, both of which are incorporated by reference herein in their entireties.

25 The herbicidal compositions described herein, particularly liquids and soluble powders, can contain as further adjuvant components one or more surface-active agents in amounts sufficient to render a given composition readily dispersible in water or in oil. The incorporation of a surface-active agent into the compositions greatly enhances their efficacy. Surface-active agent, as used herein, includes wetting agents, dispersing agents, suspending agents, and

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emulsifying agents are included therein. Anionic, cationic, and non-ionic agents can be used with equal facility.

Suitable wetting agents include alkyl benzene and alkyl naphthalene sulfonates, sulfated fatty alcohols, amines or acid amides, long chain acid esters of sodium isothionate, esters of sodium sulfosuccinate, sulfated or sulfonated fatty acid esters petroleum sulfonates, sulfonated vegetable oils, ditertiary acetylenic glycols, polyoxyethylene derivatives of alkylphenols (particularly isooctylphenol and nonylphenol), and polyoxethylene derivatives of the mono-higher fatty acid esters of hexitol anhydrides (e.g. sorbitan). Exemplary dispersants include methyl cellulose, polyvinyl alcohol, sodium lignin sulfonates, polymeric alkyl naphthalene sulfonates, sodium naphthalene sulfonate, polymethylene bisnaphthalenesulfonate, and sodium N-methyl-N- (long chain acid) laurates.

Water-dispersible powder compositions can be made containing one or more active ingredients, an inert solid extender, and one or more wetting and dispersing agents. The inert solid extenders are usually of mineral origin, such as the natural clays, diatomaceous earth, and synthetic minerals derived from silica and the like. Examples of such extenders include kaolin, attapulgite clay, and synthetic magnesium silicate. Water-dispersible powders described herein can optionally contain from about 5 to about 95 parts by weight of active ingredient (e.g., from about 15 to 30 parts by weight of active ingredient), from about 0.25 to 25 parts by weight of wetting agent, from about 0.25 to 25 parts by weight of dispersant, and from 4.5 to about 94.5 parts by weight of inert solid extender, all parts being by weight of the total composition. Where required, from about 0.1 to 2.0 parts by weight of the solid inert extender can be replaced by a corrosion inhibitor or anti-foaming agent or both.

Aqueous suspensions can be prepared by dissolution or by mixing together and grinding an aqueous slurry of a water-insoluble active ingredient in the presence of a dispersing agent to obtain a concentrated slurry of very finely-divided particles. The resulting concentrated aqueous suspension is characterized by its extremely small particle size, so that when diluted and sprayed, coverage is very uniform.

Emulsifiable oils are usually solutions of active ingredient in water-immiscible or partially water-immiscible solvents together with a surface active agent. Suitable solvents for the active ingredient described herein include hydrocarbons and water-immiscible ethers, esters, or ketones. The emulsifiable oil compositions generally contain from about 5 to 95 parts active

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ingredient, about 1 to 50 parts surface active agent, and about 4 to 94 parts solvent, all parts being by weight based on the total weight of emulsifiable oil.

Compositions described herein can also contain other additaments, for example, fertilizers, phytotoxicants and plant growth regulants, pesticides, and the like used as adjuvants or in combination with any of the above-described adjuvants. The compositions described herein can also be admixed with the other materials, e.g., fertilizers, other phytotoxicants, etc., and applied in a single application.

In each of the formulation types described herein, e.g., liquid and solid formulations, the concentration of the active ingredients are the same.

In some embodiments, the composition can include α -ketoglutarate as the major component. α -ketoglutarate is an important dicarboxylic acid and one of the key intermediates in the tricarboxylic acid cycle and amino acid metabolism. α -ketoglutarate can be isolated from the reaction mixture by methods such as that set forth in French Patent No. 07199, herein incorporated by reference. The α -ketoglutarate composition can be formulated with pharmaceutical excipients and carriers, food additives, or components used to form biomaterials. The α -ketoglutarate composition can be used in a variety of applications, including in synthesizing pharmaceutical agents, food additives, and biomaterials, as described in Li et al., *Bioprocess Biosyst Eng*, 39:967-976 (2016).

It is recognized that the herbicidal compositions can be used in combination with other herbicides. The herbicidal compositions of the present invention are often applied in conjunction with one or more other herbicides to control a wider variety of undesirable vegetation. When used in conjunction with other herbicides, the presently claimed compounds can be formulated with the other herbicide or herbicides, tank mixed with the other herbicide or herbicides or applied sequentially with the other herbicide or herbicides. Some of the herbicides that can be employed in conjunction with the compounds of the present invention include: amide herbicides such as allidochlor, beflubutamid, benzadox, benzipram, bromobutide, cafenstrole, CDEA, chlorthiamid, cyprazole, dimethenamid, dimethenamid-P, diphenamid, epronaz, etnipromid, fentrazamide, flupoxam, fomesafen, halosafen, isocarbamid, isoxaben, napropamide, naptalam, pethoxamid, propyzamide, quinonamid and tebutam; anilide herbicides such as chloranocryl, cisanilide, clomeprop, cypromid, diflufenican, etobenzanid, fenasulam, flufenacet, flufenican, mefenacet, mefluidide, metamifop, monalide, naproanilide, pentanochlor, picolinafen and

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- propanil; arylalanine herbicides such as benzoylprop, flamprop and flamprop-M;
 chloroacetanilide herbicides such as acetochlor, alachlor, butachlor, butenachlor, delachlor,
 diethatyl, dimethachlor, metazachlor, metolachlor, S-metolachlor, pretilachlor, propachlor,
 propisochlor, prynachlor, terbuchlor, thenylchlor and xylachlor; sulfonanilide herbicides such as
 5 benzo-fluor, perfluidone, pyrimisulfan and profluazol; sulfonamide herbicides such as asulam,
 carbasulam, fenasulam and oryzalin; antibiotic herbicides such as bilanafos; benzoic acid
 herbicides such as chloramben, dicamba, 2,3,6-TBA and tricamba; pyrimidinyloxybenzoic acid
 herbicides such as bispyribac and pyriminobac; pyrimidinylthiobenzoic acid herbicides such as
 pyri-thiobac; phthalic acid herbicides such as chlorthal; picolinic acid herbicides such as
 10 aminopyralid, clopyralid and picloram; quinolinecarboxylic acid herbicides such as quinclorac
 and quinmerac; arsenical herbicides such as cacodylic acid, CMA, DSMA, hexaflurate, MAA,
 MAMA, MSMA, potassium arsenite and sodium arsenite; benzoylcyclohexanedione herbicides
 such as mesotrione, sulcotrione, tefuryltrione and tembotrione; benzofuranyl alkylsulfonate
 herbicides such as benfuresate and ethofumesate; carbamate herbicides such as asulam,
 15 carboxazole chlorprocarb, dichlormate, fenasulam, karbutilate and terbucarb; carbanilate
 herbicides such as barban, BCPC, carbasulam, carbetamide, CEPC, chlorbufam,
 chlorpropham, CPPC, desmedipham, phenisopham, phenmedipham, phenmedipham-ethyl,
 propham and swep; cyclohexene oxime herbicides such as alloxymid, butoxydim, clethodim,
 cloproxydim, cycloxydim, profoxydim, sethoxydim, tepraloxymid and tralkoxydim;
 20 cyclopropylisoxazole herbicides such as isoxachlortole and isoxaflutole; dicarboximide
 herbicides such as benzfendazole, cinidon-ethyl, flumezin, flumiclorac, flumioxazin and
 flumipropyn; dinitroaniline herbicides such as benfluralin, butralin, dinitramine, ethalfluralin,
 fluchloralin, isopropalin, methalpropalin, nitratin, oryzalin, pendimethalin, prodiamine, profluralin
 and trifluralin; dinitrophenol herbicides such as dinofenat, dinoprop, dinosam, dinoseb,
 25 dinoterb, DNOC, etinofen and medinoterb; diphenyl ether herbicides such as ethoxyfen;
 nitrophenyl ether herbicides such as acifluorfen, aclonifen, bifenox, chlomethoxyfen,
 chlomitrofen, etnipro-mid, fluorodifen, fluoroglycofen, fluoronitrofen, fomesafen, furyloxyfen,
 halosafen, lactofen, nitrofen, nitrofluorfen and oxyfluorfen; dithiocarbamate herbicides such as
 dazomet and metam; halogenated aliphatic herbicides such as alorac, chloropon, dalapon,
 30 flupropanate, hexachloroacetone, iodomethane, methyl bromide, monochloroacetic acid, SMA
 and TCA; imidazolinone herbicides such as imazamethabenz, imazamox, imazapic, imazapyr,
 imazaquin and imazethapyr; inorganic herbicides such as ammonium sulfamate, borax, calcium
 chlorate, copper sulfate, ferrous sulfate, potassium azide, potassium cyanate, sodium azide,

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sodium chlorate and sulfuric acid; nitrile herbicides such as bromobonil, bromoxynil, chloroxynil, dichlobenil, iodobonil, ioxynil and pyraclostrobin; organophosphorus herbicides such as amiprofos-methyl, anilofos, bensulide, bilanafos, butamifos, 2,4-DEP, DMPA, EBEP, fosamine, glyphosate and piperophos; phenoxy herbicides such as bromofenoxim, clomeprop, 2,4-DEB, 2,4-DEP, 5 difenopenten, disul, erbon, etniproamid, fenteracol and trifluralin; phenoxyacetic herbicides such as 4-CPA, 2,4-D, 3,4-DA, MCPA, MCPA-thioethyl and 2,4,5-T; phenoxybutyric herbicides such as 4-CPB, 2,4-DB, 3,4-DB, MCPB and 2,4,5-TB; phenoxypropionic herbicides such as cloprop, 4-CPP, dichlorprop, dichlorprop-P, 3,4-DP, fenoprop, mecoprop and mecoprop-P; aryloxyphenoxypropionic herbicides such as chlorazifop, clodinafop, clofop, cyhalofop, diclofop, 10 fenoxaprop, fenoxaprop-P, fenthiaprop, fluazifop, fluazifop-P, haloxyfop, haloxyfop-P, isoxapyrifop, metamifop, propaquizafop, quizalofop, quizalofop-P and trifop; phenylenediamine herbicides such as dinitramine and prodiamine; pyrazolyl herbicides such as benzofenap, pyrazolynate, pyrasulfotole, pyrazoxyfen, pyroxasulfone and topramezone; pyrazolylphenyl herbicides such as fluazolate and pyraflufen; pyridazine herbicides such as credazine, pyridafol 15 and pyridate; pyridazinone herbicides such as brompyrazon, chloridazon, dimidazon, flufenpyr, metflurazon, norflurazon, oxapyrazon and pydanon; pyridine herbicides such as aminopyralid, clidinate, clopyralid, dithiopyr, fluroxypyr, haloxydine, picloram, picolinafen, pyriclor, thiazopyr and triclopyr; pyrimidinediamine herbicides such as iprymidam and tioclorim; quaternary ammonium herbicides such as cyperquat, diethamquat, difenzoquat, diquat, morfamquat and 20 paraquat; thiocarbamate herbicides such as butylate, cycloate, di-allate, EPTC, esprocarb, ethiolate, isopollinate, methiobencarb, molinate, orbencarb, pebulate, prosulfocarb, pyributicarb, sulfallate, thiobencarb, tiocarbamil, tri-allate and vemolate; thiocarbonate herbicides such as dimexano, EXD and proxan; thiourea herbicides such as methiuron; triazine herbicides such as dipropetryn, triaziflam and trihydroxytriazine; chlorotriazine herbicides such as atrazine, 25 chlorazine, cyanazine, cyprazine, eglazine, ipazine, mesoprazine, procyzazine, proglazine, propazine, sebutylazine, simazine, terbutylazine and trietazine; methoxytriazine herbicides such as atraton, methometon, prometon, secbumeton, simeton and terbumeton; methylthiotriazine herbicides such as ametryn, aziprotryne, cyanatryne, desmetryn, dimethametryn, methoprotetryn, prometryn, simetryn and terbutryn; triazinone herbicides such 30 as ametrifone, amibuzin, hexazinone, isomethiozin, metamitron and metribuzin; triazole herbicides such as amitrole, cafenstrole, epronaz and flupoxam; triazolone herbicides such as amicarbazone, bencarbazone, carfentrazone, flucarbazone, propoxycarbazone, sulfentrazone and thienicarbazone-methyl; triazopyrimidine herbicides such as cloransulam, diclosulam,

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florasulam, flumetsulam, metosulam, penoxsulam and pyroxsulam; uracil herbicides such as butafenacil, bromacil, flupropacil, isocil, lenacil and terbacil; 3-phenyluracils; urea herbicides such as benzthiazuron, cumyluron, cycluron, dichloralurea, diflufenzopyr, isonoruron, isouron, methabenzthiazuron, monisouron and noruron; phenylurea herbicides such as anisuron,

5 buturon, chlorbromuron, chloreturon, chlorotoluron, chloroxuron, daimuron, difenoxuron, dimefuron, diuron, fenuron, fluometuron, fluothiuron, isoproturon, linuron, methiuron, methyldymron, metobenzuron, metobromuron, metoxuron, monolinuron, monuron, neburon, parafluron, phenobenzuron, siduron, tetrafluron and thidiazuron; pyrimidinylsulfonyleurea herbicides such as amidosulfuron, azimsulfuron, bensulfuron, chlorimuron, cyclosulfamuron,

10 ethoxysulfuron, flazasulfuron, flucetosulfuron, flupyrsulfuron, foramsulfuron, halosulfuron, imazosulfuron, mesosulfuron, nicosulfuron, orthosulfamuron, oxasulfuron, primisulfuron, pyrazosulfuron, rimsulfuron, sulfometuron, sulfosulfuron and trifloxysulfuron; triazinylsulfonyleurea herbicides such as chlorsulfuron, cinosulfuron, ethametsulfuron, iodosulfuron, metsulfuron, prosulfuron, thifensulfuron, triasulfuron, tribenuron, triflusulfuron and

15 tritosulfuron; thiadiazolyleurea herbicides such as buthiuron, ethidimuron, tebuthiuron, thiazafluron and thidiazuron; and unclassified herbicides such as acrolein, allyl alcohol, aminocyclopyrachlor, azafenidin, benazolin, bentazone, benzobicyclon, buthidazole, calcium cyanamide, cambendichlor, chlorfenac, chlorfenprop, chlorflurazole, chlorflurenol, cinmethylin, clomazone, CPMF, cresol, ortho-dichlorobenzene, dimepiperate, endothal, fluoromidine,

20 fluridone, flurochloridone, flurtamone, fluthiacet, indanofan, methazole, methyl isothiocyanate, nipyraclufen, OCH, oxadiargyl, oxadiazon, oxaziclomefone, pentachlorophenol, pentoxazone, phenylmercury acetate, pinoxaden, prosulfalin, pyribenzoxim, pyriftalid, quinoclamine, rhodethanil, sulglycapin, thidiazimin, tridiphane, trimeturon, tripropindan and tritac. The herbicidal compositions of the present invention can, further, be used in conjunction with

25 glyphosate or 2,4-D on glyphosate-tolerant or 2,4-D-tolerant crops. It is generally preferred to use the compositions of the invention in combination with herbicides that are selective for the crop being treated and which complement the spectrum of weeds controlled by these compositions at the application rate employed. It is further generally preferred to apply the compositions of the invention and other complementary herbicides at the same time, either as a

30 combination formulation or as a tank mix.

SUBSTITUTE SPECIFICATION - MARKED**III. Methods of Use of L-Glufosinate Compositions**

The compositions described herein can be used in methods for selectively controlling weeds in a field or any other area, including, for example, a railway, lawn, golf course, and others where the control of weeds is desired. Optionally, the field or other area can contain a crop of planted seeds or crops that are resistant to glufosinate. The methods can include applying an effective amount of a composition comprising L-glufosinate as described herein to the field.

The compositions described herein are useful for application to a field of crop plants for the prevention or control of weeds. The composition may be formulated as a liquid for spraying on a field. The L-glufosinate is provided in the composition in effective amounts. As used herein, effective amount means from about 10 grams active ingredient per hectare to about 1,500 grams active ingredient per hectare, e.g., from about 50 grams to about 400 grams or from about 100 grams to about 350 grams. In some embodiments, the active ingredient is L-glufosinate. For example, the amount of L-glufosinate in the composition can be about 10 grams, about 50 grams, about 100 grams, about 150 grams, about 200 grams, about 250 grams, about 300 grams, about 350 grams, about 400 grams, about 500 grams, about 550 grams, about 600 grams, about 650 grams, about 700 grams, about 750 grams, about 800 grams, about 850 grams, about 900 grams, about 950 grams, about 1,000 grams, about 1,050 grams, about 1,100 grams, about 1,150 grams, about 1,200 grams, about 1,250 grams, about 1,300 grams, about 1,350 grams, about 1,400 grams, about 1,450 grams, or about 1,500 grams L-glufosinate per hectare.

IV. Exemplary Embodiments

Non-limiting embodiments include:

1. A method for making L-glufosinate, comprising:
 - reacting D-glufosinate with a D-amino acid oxidase (DAAO) enzyme to form PPO (2-oxo-4-(hydroxy(methyl)phosphinoyl)butyric acid); and
 - aminating the PPO to L-glufosinate by a transaminase (TA) enzyme, using an amine group from one or more amine donors,
 - wherein at least 70% of the D-glufosinate is converted to L-glufosinate.

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2. The method of embodiment 1, wherein the amine donor is selected from the group consisting of glutamate, L-glutamate, alanine, sec-butylamine, phenylethylamine, glycine, lysine, valine, serine, glutamine, isopropylamine, ethanolamine, 2-aminobutyric acid, and diaminopropionic acid, or any secondary amine or amino acid.
- 5 3. The method of embodiment 1, wherein the D-glufosinate is originally present in a racemic mixture of D- and L-glufosinate or salts thereof.
4. The method of embodiment 1, wherein the DAAO enzyme is selected from the enzyme from *Rhodospiridium toruloides* or *Trigonopsis variabilis*, *Neolentinus lepideus*, *Trichoderma reesei*, or *Trichosporon oleaginosus*. In an embodiment, the *Rhodospiridium*
10 *toruloides* DAAO enzyme is UniProt P80324. In an embodiment, the *Trigonopsis variabilis* DAAO enzyme is UniProt Q99042. In an embodiment, the *Neolentinus lepideus* DAAO enzyme is KZT28066.1. In an embodiment, the *Trichoderma reesei* DAAO enzyme is XP_006968548.1. In an embodiment, the *Trichosporon oleaginosus* DAAO enzyme is KLT40252.1.
5. The method of embodiment 1, wherein the DAAO enzyme is a mutant DAAO.
- 15 6. The method of embodiment 5, wherein the mutant DAAO is a mutant DAAO based on the sequence from *Rhodospiridium toruloides*.
7. The method of embodiment claim 5, wherein the mutant DAAO comprises one or more mutations at positions 54, 56, 58, 213, and 238.
8. The method of embodiment 7, wherein the mutation at position 54 is selected
20 from the group consisting of N54C, N54L, N54T, and N54V.
9. The method of embodiment 7, wherein the mutation at position 56 is T56M.
10. The method of embodiment 7, wherein the mutation at position 58 is selected from the group consisting of F58A, F58G, F58H, F58K, F58N, F58Q, F58R, F58S, and F58T.
11. The method of embodiment 7, wherein the mutation at position 213 is M213S.
- 25 12. The method of embodiment 5, wherein the mutant DAAO comprises mutations F58K and M213S.

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13. The method of embodiment 5, wherein the mutant DAAO comprises mutations at positions 54 and 56.
14. The method of embodiment 5, wherein the mutant DAAO comprises mutations N54T and T56M.
- 5 15. The method of embodiment 5, wherein the mutant DAAO comprises mutations F58Q or F58H.
16. The method of embodiment 5, wherein the mutant DAAO comprises mutations N54V and F58Q.
17. The method of embodiment 5, wherein the mutant DAAO comprises mutations
10 N54V, F58Q, and M213S.
18. The method of embodiment 1, wherein the TA enzyme is a gabT transaminase from *Escherichia coli*. In an embodiment, the *Escherichia coli* gabT transaminase is UniProt P22256.
19. The method of embodiment 1, wherein the TA enzyme is encoded by SEQ ID
15 NO: 1.
20. The method of embodiment 1, wherein the reacting step and the aminating step are performed in a single container.
21. The method of embodiment 20, wherein all reagents are substantially added at the start of the reaction.
- 20 22. The method of embodiment 20, wherein the reagents for the reacting step and the reagents for the aminating step are added to the single container at different times.
23. The method of embodiment 1, wherein the reacting step and the aminating step are performed in separate containers.
24. A composition comprising D-glufosinate, PPO, and L-glufosinate.
- 25 25. The composition of embodiment 24, wherein the amount of L-glufosinate is 90% or greater based on the total amount of D-glufosinate, PPO, and L-glufosinate.

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26. The method of embodiment 1 wherein a solid is obtained having the composition of embodiment 24 or 25.

27. The method of embodiment 1 wherein a solution of L-glufosinate is obtained for use in a formulation which has herbicidal activity.

5 28. A formulation, comprising L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; one or more additional components selected from the group consisting of sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; dipropylene glycol in an amount from 4-18% by weight of the formulation; and alkyl polysaccharide in an
10 amount from 4-20% by weight of the formulation; and water as the balance of the formulation.

29. The composition of embodiment 28, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the
15 formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.

30. The composition of embodiment 28, wherein the formulation comprises: L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount
20 of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 24.5% by weight of the formulation.

31. The composition of embodiment 28, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount
25 of 0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation.

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32. A formulation, comprising L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; one or more additional components selected from the group consisting of sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; and alkyl polysaccharide in an amount from 3-10% by weight of the formulation; and water as the balance of the formulation.

33. The composition of embodiment 32, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 58.45% by weight of the formulation.

34. The composition of embodiment 32, wherein the formulation comprises: L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

35. The composition of embodiment 32, wherein the formulation comprises: L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 11.05% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5% by weight of the formulation; alkyl polysaccharide in an amount of 3.1% by weight of the formulation; and water in an amount of 73.1% by weight of the formulation.

36. A method for selectively controlling weeds in an area comprising:
applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate to the area.

37. The method of embodiment 36, wherein the amount of the composition is applied at less than 400 grams of the sum of L-glufosinate and D-glufosinate per hectare.

38. A method for selectively controlling weeds in an area comprising:

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applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate to the area and more than 0.01% but less than 10% PPO to the area.

39. The method of embodiment 38, wherein the amount of the composition is applied at less than 400 grams of the sum of L-glufosinate, D-glufosinate and PPO per hectare.

40. A method for selectively controlling weeds in an area containing a crop of planted seeds or crops that are resistant to glufosinate, comprising:
applying an effective amount of a composition comprising L-glufosinate at an enantiomeric excess of greater than 90% over D-glufosinate and more than 0.01% but less than 10% PPO to the field.

The following examples are offered by way of illustration and not by way of limitation.

EXAMPLES**Example 1: DAAO enzyme purification**

The coding sequence of a mutant DAAO from *Rhodospiridium toruloides* (for example, consisting of a MMARIRL leader sequence and the F58K and M213S mutations) was cloned into the pET14b vector to allow for expression of an N terminally 6xHis tagged protein. This pET14b-RgDAAO plasmid was transformed into BL21 (BE3) trxB pLysS cells. The sequence of the wild type DAAO from *Rhodospiridium toruloides* to which all numbering described here corresponds, is:

MHSQKRVVVLGSGVIGLSSALILARKGYSVHILARDLPEDVSSQTFASPWAGANWTPF
MTLTDGPRQAKWEESTFKKWVELVPTGHAMWLKGTRRFAQNEDGLLGHWYKDITPNYRPLP
SSECPPGAIGVTYDTLSVHAPKYCQYLARELQKLGATFERRTVTSLEQAFDGADLVVNATGLG
AKSIAGIDDQAAEPIRGQTVLVKSPCKRCTMDSSDPASPAYIIPRPGGEVICGGTYGVGDWDL
VNPETVQRILKHCLRLDPTISSDGTIEGIEVLRHNVGLRPARRGGPRVEAERIVLPLDRTKSPLSL
GRGSARAAKEKEVTLVHAYGFSSAGYQQSWGAAEDVAQLVDEAFQRYHGAARESKL (SEQ
ID NO: 2).

To purify DAAO enzyme, cells were grown in 400 mL autoinducing medium (LB broth base with trace elements, Formedium) at 30°C for 20 to 24 hours. Cells were harvested in

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precooled centrifuges and buckets, washed with cold water, centrifuged again, and stored at -80 °C until purification.

Cell pellets were then thawed in lysis buffer (50 mM potassium phosphate, pH 8.0, 20 mM imidazole and 1% Sigma protease inhibitor cocktail (PIC) w/o EDTA) at a volume of 5 mL of lysis buffer per 1 g cell pellet. While on ice, cells were sonicated 4 times for 30 seconds at an amplitude of 10. The cell lysate was clarified by centrifugation and then added to cobalt resin (HisPur Cobalt, ThermoScientific) at 4 times the bed volume. The cell lysate was incubated for 1 hour, with gentle shaking, at room temperature. The resin was added to a column and washed twice with 5 bed volumes of wash buffer (50 mM Kpi, pH 8.0, 20 mM imidazole). The elution was performed 4 times with 1 bed volume of elution buffer (50 mM Kpi, 200 mM imidazole).

Example 2: Colorimetric determination of DAAO activity

DAAO activity was determined similarly to Berneman et al. In brief, 100 uL of substrate and HRP (0.1 mg/mL HRP, Sigma P8375, and the desired amount of D-glufosinate or racemic D/L-glufosinate in 50 mM potassium phosphate, pH 8) was added to a Brand UV micro cuvette. To that, 50 uL of dyes (60 ug/mL TBHBA, Sigma 439533, and 1 mg/mL 4-aminoantipyrine, Sigma A4382, in 50 mM potassium phosphate, pH 8) was added and then 50 uL of enzyme mix (DAAO concentration as desired in 100 mM potassium phosphate, pH 8). The reaction was monitored on a spectrophotometer at 510 nm over an appropriate time to determine the enzyme kinetics. Although no flavin adenine dinucleotide (FAD) was added to the purification of DAAO or the reaction, this reagent can optionally be included. Two exemplary mutant variants of *Rhodospiridium toruloides* DAAO, AC201 (containing F58K and M213S) and AC263 (containing N54T, T56M, F58K, and M213S), purified as in Example 1, were tested using this assay and shown to produce hydrogen peroxide, demonstrating their activity in oxidizing D-glufosinate. AC201 and AC263 have similar V_{max} , but AC263 has a lower K_M .

Example 3: Purification of transaminases

To purify, for example, the *Escherichia coli* gabT transaminase (UniProt P22256), the gene was amplified from E. coli K12 strain ER2925 and cloned into pET-14b to generate an N-terminal 6xHis tagged version. This plasmid was then transformed into BL21 (DE3) cells for

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induction. After induction in autoinducing media, cells were lysed by sonication and the 6xHis tagged enzyme purified as described in Example 1.

Example 4: Demonstration of transaminase activity

In a non-limiting example, the source of PPO for a transamination assay can be D-glufosinate or racemic D/L-glufosinate that has been converted by a DAAO to PPO. In the first
5 step, 39 mM racemic D/L-glufosinate was incubated with 0.5 mg/ml purified *Rhodospiridium toruloides* DAAO F58K M213S and 10 ug/mL catalase in 50 mM Potassium phosphate buffer, pH8 for 20 hours at 30 C. This resulted in conversion of the majority of the D-glufosinate to PPO. Subsequently, purified *E. coli* gabT was added at 20 ug/mL and L-glutamate was added at
10 50 mM as the amine donor. At relevant points, samples were stopped by boiling for 10 minutes followed by precipitation with an equal volume of acetonitrile. The individual chemical species were resolved on an HPLC with a Chirobiotic T2 column and quantified by comparison to authentic standards.

The combination of a mutant variant of DAAO and a transaminase resulted in an
15 improvement of an enantiomeric enrichment that started at 0% for L-glufosinate over D-glufosinate (i.e., equal representation of D-glufosinate and L-glufosinate) to an enantiomeric enrichment of 92%. These results demonstrate that *E. coli* gabT has transaminase activity, and this assay can be used to determine the activity of any number of wild type and/or mutant potential transaminases.

20 Example 5: De-racemization of racemic D/L-glufosinate in a single vessel

Reactions were set up similarly as in Example 4. The system (5.45 mL, 30 °C) was run in phosphate buffer at a pH of 7.3. It was noted that 50 mM of phosphate buffer at a pH of 8.0 was inadequate to buffer the amino acid additions and that the unadjusted pH after amino acid additions in this system was pH 6.4. The pH was adjusted using 1M of the base salt K₂HPO₄
25 to a volume of 5.45 mL, meaning that the actual initial substrate concentration by addition was 275 mM. The following reagents were added essentially simultaneously at the start of the reaction: 271 mg D,L-glufosinate, 420 mg glutamate, 15 mg AC263 DAAO, 50 µg catalase, and 1.0 mg *E. coli* gab T transaminase. Figure 2 shows that, when all reagents were added, the amount of D-PPT (D-glufosinate) diminished with only modest accumulation of PPO. This result

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indicates an efficient deracemisation of D/L-glufosinate into L-glufosinate by the RgDAAO/EcgabT enzyme couple.

Example 6: Demonstration of improved DAAO enzymes

5 Using protein mutagenesis strategies as outlined above, improved and variant DAAO enzymes were identified. The enzymes were assayed according to the procedures described below.

Stock Solutions:

The following dye stock solutions were prepared: a 20 mg/mL stock solution of 2,4,6-tribromo-3-hydroxybenzoic acid (TBHBA) in DMSO; and a 100 mg/mL stock solution of 4-aminoantipyrine (4-AAP) in water. The following enzyme stock solution was prepared: a 10 mg/mL stock solution of horseradish peroxidase (HRP) type 6 in a pH 8.0 potassium phosphate buffer. The following substrate stock solution was prepared: varying concentrations of D or DL amino acid in a pH 8.0 potassium phosphate buffer.

Reaction Mixes:

15 The following reaction mixtures were prepared:

Mix A is a combination of the substrate and HRP enzyme. Solutions were prepared for each substrate concentration to be assayed using reaction buffer. The solutions were two times the final substrate concentration and 0.2 mg/mL for the HRP solution.

20 Mix B is a dye mixture. To 5 mL of reaction buffer was added 120 μ L of TBHBA solution and 400 μ L of 4-AAP solution.

Mix C is an enzyme mixture. A 0.1 mg/mL solution of DAAO in reaction buffer was prepared. The final reaction concentration was 25 μ g/mL.

Protocol:

25 A spectrophotometer was used at a wavelength of 510 nm, which corresponds to the maximum absorbance for 4-AAP/TBHBA and is the point at which the extinction coefficient is 29400 M⁻¹ cm⁻¹. The temperature for performing the assays was 30 °C. The reaction kinetics

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were obtained by measuring every minute for 15 minutes. Between measurements, 20 seconds of orbital shaking at normal intensity was performed, followed by 10 seconds of settling time.

Using a 96-well plate, the following mixes (with replicates) were added in the following order using multi-channel: 100µl mix A, 50µl mix B, and 50µl mix C. The measurements were started immediately after the enzyme addition.

The enzyme kinetics were measured as described above, plotted on a Michaelis Menten graph, and used to calculate V_{max} and K_M . For the variant Ac302 (54V, 58Q, 213S), the V_{max} was 4.2 umol/min*mg.

This analysis was completed for a number of variant DAAO enzymes as above except that the Mix C stock was 0.2 mg/mL solution of DAAO and this final reaction concentration of DAAO was 50 ug/mL.

As shown below in Table 1, variant mutant DAAO enzymes showed a range of activities:

Table 1:

Variant	Mutations	Vmax (% of Ac302)
Ac263	54T, 56M, 58K, 213S	33
Ac302	54V, 58Q, 213S	100
Ac305	54C, 58H, 213S	88
Ac309	54T, 58T, 213S	71
Ac312	54T, 58G, 213S	74
Ac314	54T, 58Q, 213S	99
Ac316	54T, 58S, 213S	75
Ac318	54T, 58A, 213S	71
Ac319	54L, 58R, 213S	64
Ac320	54V, 58R, 213S	76
Ac322	54V, 58N, 213S	79

Example 7: De-racemization of racemic D/L-glufosinate at a 5 L reaction size

The scale of the de-racemization is increased using approaches familiar to those skilled in the art. Reagents and their relative ratios are substantially similar to Example 5, but the amounts are significantly greater. Rather than tubes in shakers, the reactions are performed in stirred jacketed reactors, including, optionally, air or oxygen sparging of the broth or headspace.

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These reactors vary in size, from less than 10 mL reaction to tens or hundreds of thousands of liters. Stirring rates are chosen to increase reaction mixing and rate while minimizing power consumption and shear.

- 5 In one example, the reaction was run at the 5 L scale. The system (5 L, 30 °C) was run in 200 mM phosphate buffer at a pH of 8.0 in a stirred, jacketed reactor. The following reagents were added essentially simultaneously at the start of the reaction: 300 mM D,L-glufosinate, 900 mM glutamate, 7.5 g AC302 DAAO, 0.2 g catalase, and 1.0 g *E. coli* gab T transaminase. In addition, 500 mL isopropanol was added to control foaming. During the course of the reaction, air was introduced at 0.3 VVM (volumes of air per volume of reaction mixture per minute).
- 10 HPLC analysis of the reaction demonstrated that equilibrium was reached within 8 hours, with the enantiomeric excess of L-glufosinate over D-glufosinate greater than 99% and the ratio of L-glufosinate to PPO 90% to 10%. This result indicates an efficient deracemisation of D/L-glufosinate into L-glufosinate by the RgDAAO/EcgabT enzyme couple at the larger scale.

Example 8: Impact of oxygen on reaction rate

- 15 Although stirred, jacketed reactors or immobilized columns typically allow for some oxygen transfer, the rate of oxygen uptake afforded by passive aeration is not sufficient for an efficient process. In one example, a reaction was run in the same vessel as Example 7 under substantially the same conditions, but under reduced (0.01 VVM), with twice the AC302 DAAO on a volumetric basis (3 g/L versus 1.5 g/L), and without the isopropanol. In this case, the
- 20 reaction took more than 60 hours to achieve equilibrium, demonstrating the critical importance of aeration for an efficient reaction.

Example 9: Co-immobilization of DAAO and TA

- 25 DAAO and TA enzymes were co-immobilized on EziG controlled pore glass beads (EnginZyme). 100mg of EziG type 3 beads were shaken at room temperature with 3ml of solution containing 16 mg of purified AC302 DAAO and 1.6mg of purified gabT in 50 mM potassium phosphate buffer pH 7.5, 0.5M NaCl, 20mM imidazole in a 50 ml Falcon tube. After 30 minutes, beads were spun down, immobilizing solution was removed, and beads were washed 3 times with 10ml of 100 mM potassium phosphate buffer pH 7.5.

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The reaction was started by adding all other components to the washed beads. The reaction mix contained 300 mM D/L-glufosinate, 900 mM L-glutamic acid, 50 ug catalase, 198 mM potassium phosphate in 2.5 mL. The reaction was incubated at 30C with shaking (250 rpm) in a 50 mL tube covered in parafilm with holes poked through for gas exchange.

5 After 1 hour, the depletion of D-glufosinate and formation of L-glufosinate was determined by HPLC and these rates calculated. After 6 hours, beads were spun down, reaction mixture was removed, and beads were washed 3 times with 10ml of 100 mM Potassium Phosphate buffer pH 7.5. Beads were then stored at 4 C for 18 to 72 hours before the reaction was repeated, for a total of 15 times, after which the retained activity was greater than 50% of
10 the initial activity.

Example 10: Effect of buffer on reaction

When soluble AC302 DAAO and *E. coli* gabT TA enzymes are used, phosphate buffer at >50 mM is required for full activity. A 100 mL reaction was incubated at 30C with shaking (250 rpm) in a 500 mL flask covered in parafilm. An air pump was used to bubble air through the
15 reaction for the first 5 hours. The air pump was removed for overnight incubation so the reaction would not bubble over and new parafilm with air holes was used for gas exchange. The reaction mix contained 300 mM D/L-glufosinate, 905 mM L-glutamic acid, 80 mg AC302 DAAO (0.8 mg/mL), 14.5 mg gabT (0.145 mg/mL), 2 mg catalase, and isopropanol as anti-foam reagent (10% initial concentration, isopropanol was additionally added at 2hr (2mL), 3hr (1mL), 3.5hr
20 (1mL), and at 4hr (2mL)). 500 uL of 1N NaOH (added before enzymes) was used to adjust pH from about 6 to about 7. pH remained at about 7 for the entire reaction without further adjustment. Due to the potassium phosphate in the stock enzyme buffer, the final mixture was 45 mM phosphate buffer. When compared to a similar reaction with 200 mM phosphate buffer, the reaction rate was 50-60% of that of the reaction with the 200 mM buffer.

25 When immobilized AC302 DOOA and *E. coli* gatT TA enzymes are used, phosphate buffer of less than 1 mM is sufficient for full activity. Immobilized proteins were prepared and reaction performed as in Example 9 for the "Buffered" reaction. In addition, immobilized proteins were prepared and reactions performed as in Example 9 for the "pH 7" reaction, except that sodium hydroxide was used to adjust the pH of the reaction to pH 7 and no phosphate buffer
30 was added (residual phosphate buffer from the enzyme storage buffer is less than 1 mM). This work demonstrated that the initial reaction rate for both the DAAO and combined DAAO and

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gabT reactions are very similar with and without the addition of phosphate buffer when immobilized enzymes are used.

Example 11: Isopropylamine as an amine donor

Isopropylamine can be used as an amine donor for conversion of PPO to L-glufosinate with the use of an appropriate TA. PPO was converted to L-glufosinate in a reaction with the following components:

- 0.25 mg/mL TA encoded by SEQ ID NO: 1
- 25 mM PPO
- 0.2 mM pyridoxal phosphate
- 250 mM isopropylamine (pHed to 8 w/ H₃PO₄)
- 100 mM Kphos buffer pH 8.0

The reaction was incubated at 25 to 30 °C for 30 hours with gentle shaking (250 rpm). At 0 hours, the amount of L-glufosinate as measured by HPLC was 0 mM, at 20 hours it was 14 mM, and at 30 hours it was 18 mM. This demonstrates that the enzyme encoded by SEQ ID NO: 1 can convert PPO into L-glufosinate.

Example 12: Lysine as an amine donor

Lysine can be used as an amine donor for conversion of PPO to L-glufosinate with the use of an appropriate TA. PPO was converted to L-glufosinate in a reaction with the following components:

- 0.4 mg/mL gabT (purified as in Example 3)
- 25 mM PPO (pHed to 8 w/ NaOH)
- 0.2 mM pyridoxal phosphate
- 75 mM L-Lysine dihydrochloride (pHed to 8 w/ NaOH)
- 100 mM Kphos buffer pH 8.0

The reaction was incubated at 30 °C for 20 hours with shaking (250 rpm). L-glufosinate was formed at a rate of 0.4 mM/hr over the 20 hours. This demonstrates that L-lysine can be used to convert PPO to L-glufosinate.

SUBSTITUTE SPECIFICATION - MARKED**Example 13: Purification and isolation of L-glufosinate**

Several batches prepared following the procedure described in Example 9 but at larger scale were generated. After the beads were removed, each batch was heated to 90 °C for at least 10 minutes, and after cooling to 20-25 °C, filtered to remove a small amount of solids. To each individual batch was added 37% HCl, dropwise, to effect the precipitation of glutamic acid. The amount of 37% HCl added was approximately 10% of the volume of the batch. The resulting white solid was removed by filtration. The batches were combined and concentrated under vacuum to an oil; the oil contained approximately 153 grams of L-glufosinate. The oil was diluted with five volumes of water and 37% HCl was added to adjust the solution to pH 1. The solution was treated sequentially with two portions each of approximately 3.0 kg of prewashed DOWEX 50WX8 cation exchange resin. In each treatment, the solution was allowed to mix with the resin for 30 minutes after which the resin was isolated on a filter. Both portions of resins were combined and washed first with water and then eluted with 4M NH₄OH. The eluent was concentrated under vacuum to an oil; PPO and 2-oxoglutarate were not present in the oil. Approximately 100 grams of the oil was diluted with water and the aqueous ammonium hydroxide was added until the pH was approximately 9. To the batch was added 1.0 kg of prewashed DOWEX Monosphere (hydroxide form) anion exchange resin and the mixture was stirred for approximately 40 minutes. An equal amount of DOWEX Monosphere resin, prewashed, was charged to a glass column. The slurry of DOWEX resin in water was added to the column on top of the prewashed resin. 800 mL of water was charged to the column followed by 0.1 N acetic acid, which was kept flowing through the column until all of the glutamic acid had eluted as determined by HPLC. 4 N acetic acid was fed to the column until all of the L-glufosinate had eluted from the column as determined by HPLC. The solution of L-glufosinate was concentrated under vacuum. The resulting oil was diluted with water and concentrated under vacuum to minimum volume two times. Methanol was added until a clear solution was obtained and an equal volume of heptane was added. The mixture was concentrated under vacuum to minimum volume and the procedure was repeated. The remaining 168 grams of oil recovered from the cation exchange treatment was treated in a similar fashion to obtain an overall total of 108 grams of crude L-glufosinate. The ratio of L-glufosinate to glutamic acid was greater than 99:1 as determined by NMR. The resulting solid was mixed with aqueous ammonium hydroxide and concentrated to dryness to afford 111 grams of L-glufosinate ammonium. Neither methanol nor acetic acid was detected by NMR analysis of the product.

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It is understood that the terminology used herein is for the purpose of describing particular embodiments only, and the terminology is not intended to be limiting. The scope of the invention will be limited only by the appended claims. Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which this invention belongs. Where a range of values is provided, it is understood that each intervening value, to the tenth of the unit of the lower limit unless the context clearly dictates otherwise, between the upper and lower limit of that range and any other stated or intervening value in that stated range, is encompassed within the invention. The upper and lower limits of these smaller ranges may independently be included in the smaller ranges and are also encompassed within the invention, subject to any specifically excluded limit in the stated range. Where the stated range includes one or both of the limits, ranges excluding either or both of those included limits are also included in the invention. Certain ranges are presented herein with numerical values being preceded by the term "about." The term "about" is used herein to provide literal support for the exact number that it precedes, as well as a number that is near to or approximately the number that the term precedes. In determining whether a number is near to or approximately a specifically recited number, the near or approximating unrecited number may be a number, which, in the context in which it is presented, provides the substantial equivalent of the specifically recited number.

All publications, patents, and patent applications cited in this specification are incorporated herein by reference to the same extent as if each individual publication, patent, or patent application were specifically and individually indicated to be incorporated by reference. Furthermore, each cited publication, patent, or patent application is incorporated herein by reference to disclose and describe the subject matter in connection with which the publications are cited. The citation of any publication is for its disclosure prior to the filing date and should not be construed as an admission that the invention described herein is not entitled to antedate such publication by virtue of prior invention. Further, the dates of publication provided might be different from the actual publication dates, which may need to be independently confirmed.

It is noted that the claims may be drafted to exclude any optional element. As such, this statement is intended to serve as antecedent basis for use of such exclusive terminology as "solely," "only," and the like in connection with the recitation of claim elements, or use of a "negative" limitation. As will be apparent to those of skill in the art upon reading this disclosure, each of the individual embodiments described and illustrated herein has discrete components

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and features which may be readily separated from or combined with the features of any of the other several embodiments without departing from the scope or spirit of the invention. Any recited method may be carried out in the order of events recited or in any other order that is logically possible. Although any methods and materials similar or equivalent to those described

5 herein may also be used in the practice or testing of the invention, representative illustrative methods and materials are now described.

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Application Number: 18435340

Reviewed by: 97ClN/eHe4jhmn5GMimwpAP70alPRU2kNiDwvOWHBrv8bqgC4kdzMg==

Note: This validation report concerns the sequence listing filed 06-03-2024 17:45:42 and indicates that NO errors were found by the USPTO during validation of the sequence listing. This validation report should not be construed as meaning that the sequence listing complies with all of the requirements of 37 CFR §§ 1.821-1.825, 37 CFR §§ 1.831-1.835 and 1.839, WIPO Standard ST.25, or WIPO Standard ST.26.

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APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY. DOCKET NO.	TOT CLAIMS	IND CLAIMS
18/435,340	02/07/2024		1820	32471/M202647G	6	1

4743

MARSHALL, GERSTEIN & BORUN LLP
233 SOUTH WACKER DRIVE
6300 WILLIS TOWER
CHICAGO, IL 60606-6357

CONFIRMATION NO. 4214
UPDATED FILING RECEIPT



00000000/3546421

Date Mailed: 06/06/2024

Receipt is acknowledged of this non-provisional utility patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF FIRST INVENTOR, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection.

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Inventor(s)

Brian Michael Green, Lutherville, MD;
Michelle Lorraine Gradley, Canterbury, UNITED KINGDOM;

Applicant(s)

BASF SE, Ludwigshafen, GERMANY;

Power of Attorney: The patent practitioners associated with Customer Number 04743

Domestic Priority data as claimed by applicant

This application is a CON of 18/179,136 03/06/2023 PAT 11,913,048
which is a CON of 17/530,018 11/18/2021 PAT 11,905,538
which is a CON of 16/997,133 08/19/2020 PAT 11,560,577
which is a CON of 16/287,290 02/27/2019 PAT 10,781,465
which is a CON of 15/787,448 10/18/2017 PAT 10,260,078
which is a DIV of 15/445,254 02/28/2017 PAT 9,834,802
which claims benefit of 62/413,240 10/26/2016 ABN
and claims benefit of 62/336,989 05/16/2016 ABN
and claims benefit of 62/302,421 03/02/2016 ABN

Foreign Applications for which priority is claimed (You may be eligible to benefit from the Patent Prosecution Highway program at the USPTO. Please see <http://www.uspto.gov> for more information.) - None.
Foreign application information must be provided in an Application Data Sheet in order to constitute a claim to foreign priority. See 37 CFR 1.55 and 1.76.

Permission to Access Application via Priority Document Exchange: Yes

Permission to Access Search Results: Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

If Required, Foreign Filing License Granted: 02/21/2024

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 18/435,340**

Projected Publication Date: 09/12/2024

Non-Publication Request: No

Early Publication Request: No

Title

METHODS FOR MAKING L-GLUFOSINATE

Preliminary Class

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications: No

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Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

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For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, <http://www.stopfakes.gov>. Part of a Department of Commerce initiative,

page 2 of 4

this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

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technology, manufacture products, deliver services, and grow your business, visit <http://www.SelectUSA.gov> or call +1-202-482-6800.

PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875						Application or Docket Number 18/435,340				
APPLICATION AS FILED - PART I										
(Column 1)		(Column 2)		SMALL ENTITY		OTHER THAN SMALL ENTITY				
FOR	NUMBER FILED	NUMBER EXTRA	RATE(\$)	FEE(\$)		RATE(\$)	FEE(\$)			
BASIC FEE <small>(37 CFR 1.16(a), (b), or (c))</small>	N/A	N/A	N/A			N/A	320			
SEARCH FEE <small>(37 CFR 1.16(k), (l), or (m))</small>	N/A	N/A	N/A			N/A	700			
EXAMINATION FEE <small>(37 CFR 1.16(o), (p), or (q))</small>	N/A	N/A	N/A			N/A	800			
TOTAL CLAIMS <small>(37 CFR 1.16(i))</small>	6 minus 20 =	*				x =	0			
INDEPENDENT CLAIMS <small>(37 CFR 1.16(h))</small>	1 minus 3 =	*				x =	0			
APPLICATION SIZE FEE <small>(37 CFR 1.16(s))</small>	If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$310 (\$155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).						0			
MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))							0			
			TOTAL			TOTAL	1820			
* If the difference in column 1 is less than zero, enter "0" in column 2.										
APPLICATION AS AMENDED - PART II										
(Column 1)		(Column 2)		(Column 3)		SMALL ENTITY		OTHER THAN SMALL ENTITY		
AMENDMENT A		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)		RATE(\$)	ADDITIONAL FEE(\$)
	Total <small>(37 CFR 1.16(i))</small>	*	Minus	**	=	x =			x =	
	Independent <small>(37 CFR 1.16(h))</small>	*	Minus	***	=	x =			x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))									
						TOTAL ADD'L FEE			TOTAL ADD'L FEE	
AMENDMENT B		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE(\$)	ADDITIONAL FEE(\$)		RATE(\$)	ADDITIONAL FEE(\$)
	Total <small>(37 CFR 1.16(i))</small>	*	Minus	**	=	x =			x =	
	Independent <small>(37 CFR 1.16(h))</small>	*	Minus	***	=	x =			x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))									
						TOTAL ADD'L FEE			TOTAL ADD'L FEE	
* If the entry in column 1 is less than the entry in column 2, write "0" in column 3. ** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20". *** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3". The "Highest Number Previously Paid For" (Total or Independent) is the highest found in the appropriate box in column 1.										



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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
18/435,340	02/07/2024	Brian Michael Green	32471/M202647G

CONFIRMATION NO. 4214

4743

MARSHALL, GERSTEIN & BORUN LLP
233 SOUTH WACKER DRIVE
6300 WILLIS TOWER
CHICAGO, IL 60606-6357

PUBLICATION NOTICE



0000000078143975

Date Mailed: 09/19/2024

Title:METHODS FOR MAKING L-GLUFOSINATE

Publication No.US-2024-0309409-A1

Publication Date:09/19/2024

NOTICE OF PUBLICATION OF APPLICATION

The above-identified application will be electronically published as a patent application publication pursuant to 37 CFR 1.211, et seq. The patent application publication number and publication date are set forth above.

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Office of Data Management, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

<i>Notice of References Cited</i>	Application/Control No. 18/435,340		Applicant(s)/Patent Under Reexamination Green et al.	
	Examiner GANAPATHIRAMA RAGHU		Art Unit 1652	Page 1 of 1

U.S. PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date YYYY-MM-DD	Name	CPC Classification	US Classification
*	A	US-10888095-B2	2021-01-12	Bhoge; Satish Ekanath	A01N25/02	1/1
*	B	US-11905538-B2	2024-02-20	Green; Brian Michael	A01N57/20	1/1
	C					
	D					
	E					
	F					
	G					
	H					
	I					
	J					
	K					
	L					
	M					

FOREIGN PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date YYYY-MM-DD	Country	Name	CPC Classification
	N					
	O					
	P					
	Q					
	R					
	S					
	T					

NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
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	V	
	W	
	X	

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in YYYY-MM-DD format are publication dates. Classifications may be US or foreign.

U.S. Patent and Trademark Office
PTO-892 (Rev. 11-2023)

Notice of References Cited

Part of Paper No. 20240927

<i>Search Notes</i> 	Application/Control No. 18/435,340	Applicant(s)/Patent Under Reexamination Green et al.
	Examiner GANAPATHIRAMA RAGHU	Art Unit 1652

CPC - Searched*		
Symbol	Date	Examiner
C12P13/02;A01N25/30;A01N57/20;C12N1/20;C12N9/10;C12P9/00; C 12P13/04;C12P41/00;C12Y104/03003;C12Y206/01019;C12N9/ 0024	09/27/2024	GR

CPC Combination Sets - Searched*		
Symbol	Date	Examiner

US Classification - Searched*			
Class	Subclass	Date	Examiner

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

Search Notes		
Search Notes	Date	Examiner
PE2E Search: US-PGPUB, USPAT, USCOR, FIT, FPRS, EPO, JPO, DERWENT, IBM_TDB, Inventors search (see attachment)	09/27/2024	GR
Google Scholar (see attachment)	09/27/2024	GR

Interference Search			
US Class/CPC Symbol	US Subclass/CPC Group	Date	Examiner

/GANAPATHIRAMA RAGHU/ Primary Examiner, Art Unit 1652	09/27/2024
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BIB DATA SHEET

CONFIRMATION NO. 4214

SERIAL NUMBER	FILING or 371(c) DATE	CLASS	GROUP ART UNIT	ATTORNEY DOCKET NO.		
18/435,340	02/07/2024	504	1652	32471/M202647G		
APPLICANTS BASF SE, Ludwigshafen, GERMANY; INVENTORS Brian Michael Green, Lutherville, MD; Michelle Lorraine Gradley, Canterbury, UNITED KINGDOM;						
** CONTINUING DATA ***** This application is a CON of 18/179,136 03/06/2023 PAT 11913048 which is a CON of 17/530,018 11/18/2021 PAT 11905538 which is a CON of 16/997,133 08/19/2020 PAT 11560577 which is a CON of 16/287,290 02/27/2019 PAT 10781465 which is a CON of 15/787,448 10/18/2017 PAT 10260078 which is a DIV of 15/445,254 02/28/2017 PAT 9834802 which claims benefit of 62/413,240 10/26/2016 ABN and claims benefit of 62/336,989 05/16/2016 ABN and claims benefit of 62/302,421 03/02/2016 ABN ** FOREIGN APPLICATIONS ***** ** IF REQUIRED, FOREIGN FILING LICENSE GRANTED ** 02/21/2024						
Foreign Priority claimed ☐ Yes ☒ No 35 USC 119(a-d) conditions met ☐ Yes ☒ No Verified and /GANAPATHIRAMA RAGHU/ Acknowledged Examiner's Signature		☐ Met after Allowance GR Initials	STATE OR COUNTRY MD	SHEETS DRAWINGS 2	TOTAL CLAIMS 6	INDEPENDENT CLAIMS 1
ADDRESS MARSHALL, GERSTEIN & BORUN LLP 233 SOUTH WACKER DRIVE 6300 WILLIS TOWER CHICAGO, IL 60606-6357 UNITED STATES						
TITLE METHODS FOR MAKING L-GLUFOSINATE						
FILING FEE RECEIVED 0.00	FEES: Authority has been given in Paper No. _____ to charge/credit DEPOSIT ACCOUNT No. _____ for following:			☐ All Fees ☐ 1.16 Fees (Filing) ☐ 1.17 Fees (Processing Ext. of time) ☐ 1.18 Fees (Issue) ☐ Other _____		

BIB (Rev. 05/07).

	☐ Credit
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Methods for making L-glufosinate

BM Green, ML Gradley - US Patent 10,260,078, 2019 - Google Patents

... the formulations comprise **L-glufosinate** ammonium in an ... the group consisting of **sodium alkyl ether sulfate** in an amount from ... the formulation comprises **L-glufosinate** ammonium in an ...

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Methods for making L-glufosinate

BM Green, ML Gradley - US Patent 11,560,577, 2023 - Google Patents

... the formulations comprise **L-glufosinate** ammonium in an ... the group consisting of **sodium alkyl ether sulfate** in an amount from ... the formulation comprises **L-glufosinate** ammonium in an ...

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Methods for making L-glufosinate

BM Green, ML Gradley - US Patent 11,732,281, 2023 - Google Patents

... The formulations comprise **L-glufosinate** ammonium in an amount ... the group consisting of **sodium alkyl ether sulfate** in an amount ... Optionally, the formulation comprises **L-glufosinate** ...

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METHODS FOR MAKING L-GLUFOSINATE

BM Green, ML Gradley - US Patent App. 18/435,544, 2024 - freepatentsonline.com

... The formulations comprise **L-glufosinate** ammonium in an amount ... the group consisting of **sodium alkyl ether sulfate** in an amount ... Optionally, the formulation comprises **L-glufosinate** ...

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Methods for improving yields of l-glufosinate

BM Green, DM Estridge - US Patent App. 17/272,803, 2021 - Google Patents

... A suitable surfactant for use in the formulated composition includes **sodium alkyl ether sulfate**. The surfactant can be present in an amount from 10% to 40% by weight of the formulated ...

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Methods for the purification of L-glufosinate

SC Fields, MR Oberholzer, BM Green, . - US Patent ... , 2023 - Google Patents

... ammonium in an amount of 12.25% by weight of the formulation; **sodium alkyl ether sulfate** in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of 0.5...

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Related searches

"l-glufosinate" "sodium alkyl ether **sulphate**"

"glufosinate" "sodium alkyl ether **sulphate**"

Novel Oil-Based Adjuvant Composition

R Vermeer, M Eberhard, R Fischer... - US Patent App. 12 ..., 2010 - Google Patents

The present invention provides compositions comprising oil, surfactants and ammonium salts suspended therein which may be added to agrochemical compositions, improving their ...

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Stable, concentrated herbicidal compositions

DA Long - US Patent 8,445,406, 2013 - Google Patents

... Examples 10 to 12 illustrate the effects of **dipropylene glycol** with various co-solvents. Example 10 is a composition of the present invention while Examples 11 and 12 are comparative. ...

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Stable, concentrated herbicidal compositions

DA Long - US Patent 7,842,647, 2010 - Google Patents

... Examples 10 to 12 illustrate the effects of **dipropylene glycol** with various co-solvents. Example 10 is a composition of the present invention while Examples 11 and 12 are comparative. ...

☆ Save  Cite Cited by 17 Related articles All 4 versions 

Stable, concentrated herbicidal compositions

DA Long - US Patent 9,078,432, 2015 - Google Patents

... Examples 10 to 12 illustrate the effects of **dipropylene glycol** with various co-solvents. Example 10 is a composition of the present invention while Examples 11 and 12 are comparative. ...

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Concentrate herbicidal composition

HE Agbaje, RJ Brinker, DJ Carter - US Patent 6,165,939, 2000 - Google Patents

... For example, antifreeze agents or pour-point modifying agents such as glycols, eg, diethylene glycol, propylene glycol, **dipropylene glycol**, or polyethylene glycol of molecular weight in ...

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Synergistic herbicidal compositions of S-metolachlor

M Hudetz, DW Kidder, RF Milliken, N Neigen - US Patent 5,981,432, 1999 - Google Patents

... as paraffins, cyclohexane or tetrahydronaphthalene; alcohols such as ethanol, propanol or butanol; glycols and their ethers and esters such as propylene glycol or **dipropylene glycol** ...

☆ Save  Cite Cited by 29 Related articles All 2 versions 

composição de microemulsão herbicida concentrada líquida e processo para aniquilar ou controlar plantas indesejadas.

GA Jimoh - 2012 - Google Patents

A01N25/00—Biocides, pest repellants or attractants, or plant growth regulators, characterised by their forms, or by their non-active ingredients or by their methods of application, eg ...

☆ Save  Cite Related articles 

Herbicidal composition and method of weed control

W Ruegg - US Patent 5,965,486, 1999 - Google Patents

... as paraffins, cyclohexane or tetrahydronaphthalene; alcohols such as ethanol, propanol or butanol; glycols and their ethers and esters such as propylene glycol or **dipropylene glycol** ...

☆ Save  Cite Cited by 2 Related articles All 2 versions 

Concentrate herbicidal composition

HE Agbaje, RJ Brinker, DJ Carter - 1999 - Google Patents

... antifreeze agents or pour-point modifying agents such as glycols, eg, diethylene glycol, propylene glycol, **dipropylene glycol**, or polyethylene glycol of molecular weight in the range ...

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Stable, concentrated herbicidal compositions

DA Long - US Patent App. 13/691,411, 2013 - Google Patents

... Examples 10 to 12 illustrate the effects of **dipropylene glycol** with various co-solvents. Example 10 is a composition of the present invention while Examples 11 and 12 are comparative. ...

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Adjuvant composition

R Killick, P Jones, J Morrison, A Killick... - US Patent App. 10 ..., 2006 - Google Patents

According to the invention, there is provided a composition having pH adjusting capabilities for use with water to be used for spraying an agrochemical, said composition comprising:(a) ...

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PTO/SB/08a (07-09)

Approved for use through 05/31/2024. OMB 0651-0031

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				Filing Date	February 7, 2024
				First Named Inventor	Brian Michael Green
				Art Unit	
				Examiner Name	
Sheet	1	of	10	Attorney Docket Number	32471/M202647G

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	**	Takahashi, S. et al.; "Bacterial d-amino acid oxidases: Recent findings and future perspectives; <i>Bioengineered</i> : July/August 2015; pages 237-241; Volume 6, No. 4.		

Examiner Signature	/GANAPATHIRAMA RAGHU/	Date Considered	09/27/2024
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*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ Applicant's unique citation designation number (optional). ² Applicant is to place a check mark here if English language Translation is attached.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /G.R/

PTO/SB/08b (07-09)

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<div>Substitute for form 1449/PTO</div> <div>INFORMATION DISCLOSURE STATEMENT BY APPLICANT</div> <div>(Use as many sheets as necessary)</div>				Complete if Known	
				Application Number	18/435,340-Conf. #4214
				Filing Date	February 7, 2024
				First Named Inventor	Brian Michael Green
				Art Unit	
				Examiner Name	
Sheet	10	of	10	Attorney Docket Number	32471/M202647G

NON PATENT LITERATURE DOCUMENTS				
Examiner Initials*	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.		T ²
	**	Trost et al., Minimization of by-product formation during d-amino acid oxidase catalyzed racemate resolution of D/L-amino acids, J. Mol. Catalysis B: Enzymatic, 19-20:189-95 (2002).		
	**	UniProtKB/Swiss-prot: P22256.1: 10 pages downloaded from https://www.ncbi.nlm.nih.gov/protein/P22256 on 06/12/17.		
	**	Whisstock et al., Prediction of protein function from protein sequence. Q. Rev. Biophysics., 2003, Vol. 36 (3): 307-340.		
	**	Wikipedia, Glufosinate, 5 pages, downloaded from the Internet at: < https://en.wikipedia.org/ > on May 25, 2022.		
		Wikipedia, Transaminase, 2 pages, downloaded from the Internet at: < https://en.wikipedia.org/wiki/Transaminase/ > on April 19, 2024.		
	**	Witkowski et al., Conversion of b-ketoacyl synthase to a Malonyl Decarboxylase by replacement of the active cysteine with glutamine. Biochemistry, 1999, Vol. 38: 11643-11650.		
	**	ZA2018/05680, Notice of Allowance, September 3, 2019, 1 page.		
	**	ZEISS, "Enantioselective synthesis of L-phosphinothricin from L-methionine and L-glutamic acid via L-vinylglycine," Tetrahedron, 1992, 48:8263-8270.		

Examiner Signature	/GANAPATHIRAMA RAGHU/	Date Considered	09/27/2024
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*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ Applicant's unique citation designation number (optional). ² Applicant is to place a check mark here if English language Translation is attached.

Methods for making L-glufosinate

BM Green, ML Gradley - US Patent 10,260,078, 2019 - Google Patents

... of the formulation; **1-methoxy-2-propanol** in an amount from 0.5-2... the formulation comprises **L-glufosinate** ammonium in an ... of the formulation; **1-methoxy-2-propanol** in an amount of 1% ...

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Methods for making L-glufosinate

BM Green, ML Gradley - US Patent 11,560,577, 2023 - Google Patents

... of the formulation; **1-methoxy-2-propanol** in an amount from 0.5-2... the formulation comprises **L-glufosinate** ammonium in an ... of the formulation; **1-methoxy-2-propanol** in an amount of 1% ...

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Methods for making L-glufosinate

BM Green, ML Gradley - US Patent 11,732,281, 2023 - Google Patents

... weight of the formulation; **1-methoxy-2-propanol** in an amount from ... Optionally, the formulation comprises **L-glufosinate** ... weight of the formulation; **1-methoxy-2-propanol** in an amount of 1...

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METHODS FOR MAKING L-GLUFOSINATE

BM Green, ML Gradley - US Patent App. 18/435,544, 2024 - freepatentsonline.com

... weight of the formulation; **1-methoxy-2-propanol** in an amount from ... Optionally, the formulation comprises **L-glufosinate** ... weight of the formulation; **1-methoxy-2-propanol** in an amount of 1...

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Process for producing a liquid herbicidal composition

A MONDAL, SE Bhoge - 2022 - Google Patents

WO2022185243A1 - Process for producing a liquid herbicidal composition - Google Patents

WO2022185243A1 - Process for producing a liquid herbicidal composition - Google Patents ...

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Herbicidal glufosinate composition

P Pentland, M Nernaric, TN LE... - US Patent App. 17/770,136, 2022 - Google Patents

... of alkylether sulfate sodium salt, 1-15% **1-methoxy-2-propanol**. Other components are not listed in the ... composition fully disclosed is about 200 g/L **glufosinate**-ammonium. US Pat. No. ...

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Methods for improving yields of l-glufosinate

BM Green, DM Estridge - US Patent App. 17/272,803, 2021 - Google Patents

... can include **1-methoxy-2-propanol** and dipropylene glycol. The **1-methoxy-2-propanol** can ... For example, the **1-methoxy-2-propanol** can be present in the amount of 0.5%, 0.6%, 0.7%, ...

☆ Save  Cite Related articles All 2 versions 

Methods for the purification of L-glufosinate

SC Fields, MR Oberholzer, BM Green... - US Patent ..., 2023 - Google Patents

... ammonium in an amount of 12.25% by weight of the formulation; sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; **1-methoxy-2-propanol** in an amount of 0.5...

☆ Save  Cite Cited by 3 Related articles All 4 versions 

Stable herbicidal compositions

SE Bhoge, PV Talati, JR Shroff, VR Shroff - US Patent 10,888,065, 2021 - Google Patents

... Glufosinate ammonium was mixed in a formulation vessel with water, propylene glycol followed by the addition of **1-methoxy-2-propanol**, Myristyldimethyl amine N-oxide, ammonium ...

☆ Save  Cite Cited by 1 Related articles All 4 versions 

A stable agrochemical composition and process for preparation thereof

FML Silva, G Lenz, G Rao, A Mondal... - US Patent App. 17 ... , 2023 - Google Patents

... According to most preferred embodiment of the present invention, herbicide in aqueous phase is glufosinate ammonium salt, or **L-glufosinate** ammonium salt, or their mixtures and salts ...

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Related searches

"l-glufosinate" "1-methoxy-2-propanol" "**toxicity**"

"l-glufosinate" " 1-methoxy-2-propanol" "**soil**"

PE2E SEARCH - Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	British Equivalents	Time Stamp
L1	50	<p>(Artificial Intelligence) Similar to: 18/435,340 with 0 CPC Selections and 0 Text Selections</p> <p>Text: (US-11905538-B2 OR US-11732281-B2 OR US-20170253897-A1 OR US-20220064680- A1 OR US- 20200385767-A1 OR US-11913048-B2 OR US-20180030487-A1 OR US-20230265472- A1 OR US-10781465- B2 OR US- 20210395790-A1 OR US-11560577-B2 OR CN-116121315-A OR US-20240301452-A1 OR US-9834802-B2 OR US-10260078-B2 OR CN-109072261-B OR US-20190185889-A1 OR JP-2023061954-A OR JP-2021078506-A OR JP-7041066-B2 OR JP-2024513190-A OR US-20240182502-A1 OR WO-2022207753- A1 OR CN-117120625- A OR CA-3213964-A1 OR KR-20230164054-A OR AU-2015326965-A1 OR EP-0930823-B2 OR DE-69718027-T3 OR US-10888095-B2 OR WO-2024130353-A1 OR AU-2022405733-A1 OR KR-20240121807-A OR AU-2012347377-B2 OR EP-0930823-B1 OR CA-2937009-C OR CA- 2868134-A1 OR AU- 2012347377-A1 OR AU-2022407771-A1 OR IL-262789-A OR US- 20140371069-A1 OR US-20080045415-A1 OR US-20170265477- A1 OR JP-2000517334-</p>	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN))	ADJ	ON	ON	2024/09/27 03:00 PM

L2	5	A OR US-9468213-B2 OR CA-3089399-A1 OR US-10729129-B2 OR CA-3240064-A1 OR US-20190110469-A1 OR CA-2715672- A1).did. ("5130246").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:19 PM
L3	5	("5162212").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:20 PM
L4	5	("5587319").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:21 PM
L5	5	("5753470").pn.	(US-PGPUB; USPAT;	ADJ	ON	ON	2024/09/27

			USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)				03:22 PM
L6	5	("5767309").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:22 PM
L7	5	("5877013").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:23 PM
L8	5	("5919669").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:29 PM

L9	5	("5962281").pn.	NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:30 PM
L10	6	("6051408").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:30 PM
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L12	6	("6936444").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:35 PM

L13	6	("7217544").pn.	AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:36 PM
L14	2	("7939702").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:37 PM
L15	2	("7939709").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:38 PM

L16	2	("8383137").pn.	RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:39 PM
L17	2	("8445406").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:39 PM
L18	3	("8716184").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:41 PM
L19	3	("8946507").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY,	ADJ	ON	ON	2024/09/27 03:41 PM

L20	2	("8981142").pn.	CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:42 PM
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L22	2	("8637432").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:45 PM

L23	5	("6165939").pn.	TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:46 PM
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L25	4	("10260078").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:51 PM
L26	2	("10781465").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:52 PM

L27	2	("10888095").pn.	CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:53 PM
L28	5	("9834802").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:56 PM
L29	2	("11560577").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:56 PM

L30	2	("11634442").pn.	VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:57 PM
L31	2	("11905538").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:57 PM
L32	2	("11913048").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:58 PM
L33	3239	(L-glufosinate OR L- phosphinothricin OR 2- amino-4- [hydroxy(methyl)phosph oryl]butanoic acid)	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA,	ADJ	ON	ON	2024/09/27 04:04 PM

			EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)				
L34	103	L33 AND (Sodium alkyl ether OR Sodium lauryl ether sulfate OR Sodium laureth sulphate OR Sodium lauryl ether sulphate)	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:05 PM
L35	72	L34 AND (Propylene glycol methyl ether OR PGME OR 1-methoxy-2-propanol OR Methoxypropanol OR α -Propylene glycol monomethyl ether)	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:05 PM
L36	71	L35 AND (Dipropylene glycol OR 1,1'-Oxybis(1-propanol) OR 1,1'-Oxybis(2-propanol))	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO;	ADJ	ON	ON	2024/09/27 04:06 PM

L37	70	L36 AND (Alkyl polysaccharide OR Alkyl polyglucoside OR Alkyl polyglycoside)	JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:06 PM
L38	0	L37 AND @ad<="20160516"	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:07 PM
L39	262658	(C12P13/02;A01N25/30;A01N57/20;C12N1/20;C12N9/10;C12P9/00;C12P13/04;C12P41/00;C12Y104/03003;C12Y206/01019;C12N9/0024). CPC.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:09 PM
L40	66	L39 AND L37	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB,	ADJ	ON	ON	2024/09/27 04:09 PM

L41	200	(green brian michael OR gradley\$).in.	HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:10 PM
L42	44	L41 AND L37	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:11 PM

PE2E SEARCH - Search History (Interference)

There are no Interference searches to show.



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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
18/435,340	02/07/2024	Brian Michael Green	32471/M202647G	4214
4743	7590	10/10/2024		
MARSHALL, GERSTEIN & BORUN LLP 233 SOUTH WACKER DRIVE 6300 WILLIS TOWER CHICAGO, IL 60606-6357			EXAMINER RAGHU, GANAPATHIRAM	
			ART UNIT 1652	PAPER NUMBER
			NOTIFICATION DATE 10/10/2024	DELIVERY MODE ELECTRONIC

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

mgbdoCKET@marshallip.com

Notice of Pre-AIA or AIA Status

The present application, filed on or after March 16, 2013, is being examined under the first inventor to file provisions of the AIA.

Detailed Action

Claims 1-6 are pending in this application and are now under consideration.

Priority

This application is a CON of 18/179,136 filed on 03/06/2023 now US Patent 11,913,048, which is a CON of 16/997,133 filed on 08/19/2020 now US Patent 11,560,577, which is a CON of 16/287,290 filed on 02/27/2019 now US Patent 10,781,465, which is a CON of 15/787,448 filed on 10/18/2017 now US Patent 10,260,078, which is a DIV of 15/445,254 filed on 02/28/2017 now US Patent 9,834,802 and applicants' claim for the benefit of priority under 35 U.S.C. 119(e) to the Provisional Applications: 62/302,421 filed on 03/02/2016; 62/336,989 filed on 05/16/2016; and 62/413,240 filed on 10/26/2016 is acknowledged.

Information disclosure statement

The information disclosure statements (IDS) submitted on 05/06/2024 is in compliance with the provisions of 37 CFR 1.97. Accordingly, the IDS statement is considered and initialed by the examiner.

Double Patenting rejection

The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory obviousness-type double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct from the reference claim(s) because the examined application claim is either anticipated by, or would have been obvious over, the reference claim(s). See, e.g., *In re Berg*, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); *In re Goodman*, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); *In re Longi*, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); *In re Van Ornum*, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); *In re Vogel*, 422 F.2d 438, 164

USPQ 619 (CCPA 1970); and *In re Thorington*, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on a nonstatutory double patenting ground provided the conflicting application or patent either is shown to be commonly owned with this application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement.

Effective January 1, 1994, a registered attorney or agent of record may sign a terminal disclaimer. A terminal disclaimer signed by the assignee must fully comply with 37 CFR 3.73(b).

Claims 1-6 of the instant application are rejected under the judicially created doctrine of obviousness-type double patenting as being unpatentable over claims 1-15 of US 11,905,538 B2. An obviousness-type double patenting rejection is appropriate where the conflicting claims are not identical, but an examined application claim is not patentably distinct from the reference claim, because the examined claim is either anticipated by, or would have been obvious over reference claim. See, e.g., *In re Berg*, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); *In re Goodman*, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); *In re Longi* 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985). Although the conflicting claims are not identical, they are not patentably distinct from each other.

Claims 1-6 of the instant application cannot be considered patentably distinct over claims 1-15 of US 11,905,538 B2 when there is specifically disclosed embodiments and falls within the scope of the instant claims 1-6 herein. In the instant case, the allowed patent specification and the allowed patent i.e., claims 1-15 of US 11,905,538 B2 discloses as a preferred embodiment and are directed to "A formulation comprising: L-glufosinate ...". Regarding rejection of instant claims 1-6 over allowed patent 1-15 of US 11,905,538 B2, examiner takes the position that the product claims of US 11,905,538 B2 and the product claims 1-6 of the instant application is a different form/version of the

allowed patent claims; examiner also notes in said allowed patent there was no restriction in place for “a method of making L-glufosinate ...”. Therefore, the product and claims 1-6 of the instant invention are deemed as an obvious variation of the invention of allowed patents claims -15 of US 11,905,538 B2.

Claim Rejections: 35 USC § 102 (AIA)

The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless –

(a)(1) the claimed invention was patented, described in a printed publication, or in public use, on sale or otherwise available to the public before the effective filing date of the claimed invention.

(a)(2) the claimed invention was described in a patent issued under section 151, or in an application for patent published or deemed published under section 122(b), in which the patent or application, as the case may be, names another inventor and was effectively filed before the effective filing date of the claimed invention.

In the event the determination of the status of the application as subject to AIA 35 U.S.C. 102 and 103 (or as subject to pre-AIA 35 U.S.C. 102 and 103) is incorrect, any correction of the statutory basis for the rejection **will not be considered** a new ground of rejection if the prior art relied upon, and the rationale supporting the rejection, would be the same under either status.

Claims 1-6 are rejected under 35 U.S.C. 102(a)(1) and 35 U.S.C. 102(a)(2) as being anticipated by Long DA., (US 8,445,406 B2, in IDS), when given the broadest reasonable interpretation.

Claims 1-6 as interpreted are directed to product claims i.e., a formulation comprising: L-glufosinate ammonium in an amount from 10-30% by weight of the formulation; **one or** more additional components selected from the group consisting of: sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation; 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; dipropylene glycol in an amount from 4-18% by weight of the formulation; alkyl polysaccharide in an

amount from 4-20% by weight of the formulation; and water as the balance of the formulation... and claims use open-ended transitional term “comprising” which is inclusive of additional, unrecited elements.

Long DA., (US 8,445, 406 B2, in IDS) disclose stable concentrated herbicidal composition comprising surfactant alkyl ether sulfate (Abstract; and entire document); L-enantiomer glufosinate and its salt in an amount 20%-35% (Abstract; col. 1, Summary of the invention; col. 2, lines 39-67; col. 3, lines 10-34); surfactant alkyl ether sulfate in an amount 3-35% by weight (col. 3, lines 35-54); said composition further comprising organic solvent such dipropylene glycol in an amount 1-20% by weight (col. 3, lines 55-67 to col. 4, lines 1-20); alkyl polysaccharide/alkyl polyglucoside in an amount 1-15% by weight (Abstract; col. 4, lines 21-67 to col. 5, lines 1-19). Also see Examples 1 to 9, Table, col. 6; Examples 10 to 12, col. 7-8; Table Examples 13-14, col. 9-10; claims and entire document.

Therefore, the reference of Long DA., (US 8,445, 406 B2, in IDS) is deemed to anticipate claims 1-6 as written and when given the broadest reasonable interpretation.

Since the Office does not have the facilities for examining and comparing applicants' composition with the composition of the prior art, the burden is on the applicant to show a novel or unobvious difference between the claimed product and the product of the prior art (i.e., that the composition of the prior art does not possess the same material structural and functional characteristics of the composition of the instant invention). See *In re Best*, 562 F.2d 1252, 195 USPQ 430 (CCPA 1977) and *In re Fitzgerald et al.*, 205 USPQ 594.

Claim Rejections: 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.
3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

In the event the determination of the status of the application as subject to AIA 35 U.S.C. 102 and 103 (or as subject to pre-AIA 35 U.S.C. 102 and 103) is incorrect, any correction of the statutory basis for the rejection will not be considered a new ground of rejection if the prior art relied upon, and the rationale supporting the rejection, would be the same under either status.

Claims 1-6 are rejected under 35 U.S.C. 103(a) as being unpatentable over Long DA., (US 8,445, 406 B2, in IDS) as applied to claims 1-6 (see 35 U.S.C. 102(a)(1) and 35 U.S.C. 102(a)(2) rejection above) and in view of Bhoge et al., (US 10,888,095 B2).

The disclosure of Long DA., (US 8,445, 406 B2, in IDS) as applied to claims 1-6 is described above in 35 U.S.C. 102(a)(1) and 35 U.S.C. 102(a)(2) rejection above. However, Long DA., is silent regarding wherein one or more additional components selected from the group consisting of: ... 1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation; ... (as in claims 1-6).

Regarding claims 1-6, analogous art Bhoge et al., (US 10,888,095 B2) teach stable herbicidal compositions comprising glufosinate, and said herbicidal composition further comprising 1-methoxy-2-propanol including the structural and functional information of

herbicidal compositions comprising 1-methoxy-2-propanol in an amount 1-2% by weight (see Abstract; Example 1, TABLE 1; claims 5-6; and entire document).

Therefore, it would have been obvious to a person of ordinary skill in the art to combine and modify the teachings of Long DA., and employ the compositions comprising herbicide glufosinate and 1-methoxy-2-propanol of Bhoge et al., that teach structural and functional elements of the instant invention to prepare stable herbicidal compositions, and the expectation of success is high, because the combined teachings provide teaching, suggestion and motivation.

Examiner also finds support in: MPEP 2144.05 [R-5]: Optimization Within Prior Art Conditions or Through Routine Experimentation Generally, differences in concentration or temperature will not support the patentability of subject matter encompassed by the prior art unless there is evidence indicating such concentration or temperature is critical. "[W]here the general conditions of a claim are disclosed in the prior art, it is not inventive to discover the optimum or workable ranges by routine experimentation". As to optimization results, a patent will not be granted based upon the optimization of result effective variables when the optimization is obtained through routine experimentation unless there is a showing of unexpected results which properly rebuts the *prima facie* case of obviousness. See *In re Boesch*, 617 F.2d 272,276,205 USPQ 215,219 (CCPA 1980). See also *In re Woodruff*, 919 F.2d 1575, 1578, 16 USPQ2d 1934, 1936-37 (Fed. Cir. 1990), and *In re Aller*, 220 F2d 454,456,105 USPQ 233,235 (CCPA 1955).

Therefore, claims 1-6 are rejected under 35 U.S.C. 103(a) as being unpatentable over Long DA., (US 8,445, 406 B2, in IDS) as applied to claims 1-6 (see 35 U.S.C.

102(a)(1) and 35 U.S.C. 102(a)(2) rejection above) and in view of Bhoge et al., (US 10,888,095 B2).

Allowable Subject Matter/Conclusion

None of the claims are allowable.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to GANAPATHIRAMA RAGHU whose telephone number is (571)272-4533. The examiner can normally be reached on M-F 8:30am-5pm EST.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at <http://www.uspto.gov/interviewpractice>.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Robert Mondesi can be reached on 408-918-7584. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/GANAPATHIRAMA RAGHU/
Primary Examiner, Art Unit 1652

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<p>Substitute for form 1449/PTO</p> <p>INFORMATION DISCLOSURE STATEMENT BY APPLICANT</p> <p><i>(Use as many sheets as necessary)</i></p>				Complete if Known	
				Application Number	18/435,340-Conf. #4214
				Filing Date	February 7, 2024
				First Named Inventor	Brian Michael Green
				Art Unit	1652
				Examiner Name	G. Raghu
				Attorney Docket Number	32471/M202647G

[illegible][illegible]

Examiner Signature		Date Considered	
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<div>Substitute for form 1449/PTO</div> <div>INFORMATION DISCLOSURE STATEMENT BY APPLICANT</div> <div>(Use as many sheets as necessary)</div>				Complete if Known	
				Application Number	18/435,340-Conf. #4214
				Filing Date	February 7, 2024
				First Named Inventor	Brian Michael Green
				Art Unit	1652
				Examiner Name	G. Raghu
Sheet	2	of	2	Attorney Docket Number	32471/M202647G

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials*	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T ²
		How do I link to a specific version of a UniProt entry, downloaded from the Internet at: < https://www.uniprot.org/help/link_old_versions > retrieved September 25, 2024.	
		P22256 GABT_ECOLI, downloaded from the Internet: < https://www.uniprot.org/uniprotkb/P22256/history >, retrieved September 25, 2024.	
		Ricca et al., Multi-enzymatic cascade reactions: overview and perspectives, Adv. Synth. Catal., 353:2239-62 (2011).	

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ELECTRONIC PAYMENT RECEIPT

APPLICATION # 18/435,340	RECEIPT DATE / TIME 11/07/2024 10:42:45 AM Z ET	ATTORNEY DOCKET # 32471/M202647G
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Title of Invention

METHODS FOR MAKING L-GLUFOSINATE

Application Information

APPLICATION TYPE	Utility - Nonprovisional Application under 35 USC 111(a)	PATENT #	-
CONFIRMATION #	4214	FILED BY	Audrey Nagelberg
PATENT CENTER #	67875401	AUTHORIZED BY	Joshua Elliott
CUSTOMER #	4743	FILING DATE	02/07/2024
CORRESPONDENCE ADDRESS	-	FIRST NAMED INVENTOR	Brian Michael Green

Payment Information

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Docket No.: 32471/M202647G
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Brian Michael Green et al.

Application No.: 18/435,340

Confirmation No.: 4214

Filed: February 7, 2024

Art Unit: 1652

For: METHODS FOR MAKING L-GLUFOSINATE

Examiner: G. Raghu

INFORMATION DISCLOSURE STATEMENT (IDS)

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Commissioner:

Pursuant to 37 C.F.R. §§ 1.56, 1.97, and 1.98, the attention of the United States Patent and Trademark Office is hereby directed to the references listed on the attached PTO/SB/08. It is respectfully requested that the information be expressly considered during the prosecution of the above-identified application, and that the references be made of record therein and appear among the "References Cited" on any patent to issue therefrom.

This Information Disclosure Statement is filed after the mailing date of a first Office Action on the merits, but before the mailing date of any of a Final Action under 37 C.F.R. § 1.113, a Notice of Allowance under 37 C.F.R. § 1.311 or an action that otherwise closes prosecution in this application (37 C.F.R. § 1.97(c)).

In accordance with 37 C.F.R. § 1.98(a)(2)(ii), copies of U.S. patents and U.S. patent application publications are not submitted. Submitted herewith are copies of foreign patents and non-patent literature in accordance with 37 C.F.R. § 1.98(a)(2).

In accordance with 37 C.F.R. § 1.97(g), the filing of this Information Disclosure Statement shall not be construed as a representation that a search has been made. In accordance with 37 C.F.R. § 1.97(h), the filing of this Information Disclosure Statement shall not be construed to be an admission that the information cited in this

Application No.: 18/435,340

Docket No.: 32471/M202647G

Information Disclosure Statement is, or is considered to be, material to patentability as defined in 37 C.F.R. § 1.56(b).

It is submitted that the Information Disclosure Statement is in compliance with 37 C.F.R. § 1.98, and the Examiner is respectfully requested to consider the listed references.

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Dated: November 7, 2024

Respectfully submitted,

Electronic signature: /Joshua T. Elliott/
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DESCRIPTION CN105567780A

10 A kind of enzyme-chemical catalytic deracemization preparation method of L-glufosinate-ammonium

[0001]

14 Technical field

[0002]

18 The invention relates to an enzyme-chemical catalytic deracemization preparation method of L-glufosinate and belongs to the technical field of enzymatic preparation of chiral pesticides.

[0003]

23 Background technique

[0004]

27 Glufosinate developed by the German company Hoechst is a glutamine synthetase (GS) inhibitor that can inhibit all known forms of GS, leading to nitrogen metabolism disorders and excessive accumulation of ammonia in plants, chloroplasts disintegrate, thus inhibiting photosynthesis and eventually leading to plant death.

31 Glufosinate-ammonium has strong herbicidal activity and can effectively control almost all tested weeds. It is safe for crops, has high activity, broad herbicidal spectrum and little harm. It is currently an ideal herbicide for genetically modified resistant crops.

34 Glufosinate is also an excellent fungicide.

[0005]

38 Glufosinate-am monium contains a chiral center, and its L-enantiomer has twice the herbicidal activity of the
racemate. It is less likely to develop resistance and has a longer product life cycle than the racemate.
40 If glufosinate-am monium products can be used in the form of L-configuration pure optical isomers, the usage
of glufosinate-am monium can be reduced by 50%, which is very important for improving atom economy,
reducing use costs, and reducing environmental pressure. meaning.

[0006]

46 The preparation methods of L-glufosinate mainly include chemical methods and biological methods.

[0007]

50 (1) Chemical method.

51 Hoechst Company (US5767309) adopts an asymmetric conversion method and uses quinine as a chiral
resolving agent to resolve DL-glufosinate to prepare L-glufosinate.

53 This method uses expensive chiral resolving agents and undergoes complex steps such as salt formation,
induced crystallization, salt solution, and recrystallization.

55 A large number of chemical asymmetric synthesis methods of L-glufosinate have been reported in the literature
(Mao Mingzhen et al., Pesticides, 2014, 53(6): 391-393). Chemical asymmetric synthesis methods use
expensive chiral sources, chiral auxiliaries or chiral catalysts. They usually have lengthy steps, complex
synthesis routes, harsh reaction conditions, low product yield and optical purity, and high production costs, so
they are not suitable for L-Industrial production of glufosinate-am monium.

[0008]

63 (2) Biological law.

64 Biological methods include biological asymmetric synthesis and biological resolution (Lou Yiyuan et al.,
Modern Pesticides, 2009, 8(3): 1-10). Bioasymmetric synthesis methods mainly include amino acid
dehydrogenase method and transaminase method. The amino acid dehydrogenase method uses 2-carbonyl-4-
(hydroxymethylphosphono)butyric acid as raw material and uses a glutamate dehydrogenase/glucose
dehydrogenase system to prepare L-glufosinate-am monium, with a yield of only 25%, ee value is 89.2%
(Fang JM et al. JChemSoc, PerkinTransI, 1995: 967-978.). In this method, not only the utilization efficiency of
glutamate dehydrogenase is very low and the product yield is low, but also the coenzyme NADH needs to be
regenerated, making it difficult to apply in industrial production. The transaminase method uses 2-carbonyl-4-
(hydroxymethylphosphono)butyric acid as the ammonia acceptor, L-glutamic acid or L-aspartic acid as the
ammonia donor, and prepares L-glufosinate through a transamination reaction. Phosphine, the conversion
rate reaches 75%, ee>99% (US5153355). Since transamination is a reversible reaction and there is a reaction
equilibrium between two L-amino acids and two α -keto acids, L-glufosinate not only has a low yield, but also
increases the difficulty of separation. The biological resolution method mainly includes the L-amidase
resolution method using DL-2-amino-4-(hydroxymethylphosphono)butanamide as raw material, and N-
acetyl (phenylacetyl)-DL-glufosinate as the raw material. The acylase separation method of raw materials, the
nitrilase separation method using 2-amino-4-(hydroxymethylphosphoryl)butyronitrile as raw material

(CN103275896A), etc. The theoretical yield of bioresolution is only 50%. Typically, bioresolution methods require derivatized DL-glufosinate as raw material, involving chemical racemization and cyclic resolution of invalid enantiomers.

[0009]

- 86 Compared with chemical methods, biological methods have the advantages of strict stereoselectivity, mild reaction conditions, high yields, and easy separation and purification of products. They are the most promising method for realizing the industrial production of L-glufosinate.
- 89 Developing a simple, efficient and green biological preparation method will be an important breakthrough and development direction for the production of L-glufosinate in the future.

[0010]

94 Contents of the invention

[0011]

- 98 In view of this, the object of the present invention is to provide an enzyme-chemical catalytic deracemization preparation method of L-glufosinate.
- 100 Using a "one-pot cooking" reaction method, using underivatized DL-glufosinate as raw material, immobilized D-amino acid oxidase is used to enantioselectively catalyze the oxidative dehydrogenation of the D-enantiomer in DL-glufosinate to 2-imino-4-(hydroxymethylphosphono)butyric acid, L-glufosinate is completely retained.
- 104 The palladium carbon-ammonium formate chemical catalytic hydrogen transfer system is used to reduce 2-imino-4-(hydroxymethylphosphono)butyric acid to DL-glufosinate in situ, and DL-glufosinate is realized through a biological oxidation-chemical reduction cycle. During the deracemization of phosphine, the hydrogen peroxide formed during the reaction is decomposed and removed in situ by catalase, thereby preparing L-glufosinate-ammonium with high yield and high optical purity.

[0012]

112 The technical route is as follows:

[0013]

116

[0014]

121 The present invention includes a deracemization step and an L-glufosinate-ammonium separation step. The

specific method steps are as follows:

[0015]

- 126 1) For deracemization reaction, add DL-glufosinate ammonium to 1 mol/L ammonium formate solution (pH 6.5), stir and dissolve, the concentration of DL-glufosinate ammonium in the prepared solution is 10~20% (w/v).
- 129 Then add immobilized D-amino acid oxidase, catalase and palladium carbon, continuously introduce oxygen, and stir the reaction at 30° C until the D-enantiomer reaction is complete;

[0016]

- 134 Among them, the mass ratio of immobilized D-amino acid oxidase (enzyme activity is 50u/g) and DL-glufosinate is 0.1~0.2:1, and the mass ratio of catalase (enzyme activity is 50000u/ml) and immobilized D-amino acid oxidase is 100~200:1, the mass ratio of palladium carbon (10%) and immobilized D-amino acid oxidase is 1~2:1, and the reaction time of the stirring reaction is 12~24h.

[0017]

- 141 The reaction process was monitored and the optical purity of L-glufosinate was determined using chiral liquid chromatography.
- 143 Chiral liquid chromatography column: CrownpakCR (+) (4.0×150mm, 5 μm), mobile phase: perchloric acid solution (pH1.5), flow rate: 0.5ml/min, detection: UV210nm.
- 145 Enantiomeric chromatographic peaks were positioned using standard controls.
- 146 The optical purity of L-glufosinate is expressed as enantiomeric excess (ee).

[0018]

- 150 2) Separate L-glufosinate, filter the reaction solution after the reaction in step 1) to remove the immobilized D-amino acid oxidase and palladium carbon, then adjust the pH of the filtrate to 8.0 with 28% ammonia water, and concentrate under reduced pressure at 80° C. The volume is 1/5 of the filtrate, cooled in an ice bath, the solid is precipitated, and dried to obtain L-glufosinate.

[0019]

- 157 Compared with the existing L-glufosinate preparation method, the enzyme-chemical catalytic deracemization preparation method proposed by the present invention has the following advantages: 1) The process is simple.
- 160 The dual enzyme reaction and the chemical catalytic hydrogen transfer reaction are carried out in the same reaction system.
- 162 High-concentration DL-glufosinate is directly used as the reaction substrate of immobilized D-amino acid oxidase without derivatization.

164 The intermediate product 2-imino-4-(hydroxymethylphosphono)butyric acid is directly converted into DL-glufosinate in situ without isolation for continuous conversion.

166 The hydrogen peroxide produced during the reaction is decomposed in situ by catalase, which minimizes the inhibitory effect of hydrogen peroxide on D-amino acid oxidase activity and the inhibitory effect on 2-imino-4-(hydroxymethylphosphine) Decomposition of acyl)butyric acid.

2)

172 2) The separation process is simple. The product stream contains only L-glufosinate and a small amount of by-products 4-(hydroxymethylphosphono)-2-butyric acid and 3-(hydroxymethylphosphono)-propionic acid, requiring only simple concentration, the crystallization method can realize the separation of L-glufosinate-ammonium with high yield and high optical purity.

3)

179 3) Low cost. Both immobilized D-amino acid oxidase and Pd-C catalyst are heterogeneous catalysts and can be recycled and reused multiple times, effectively reducing process costs.

[0020]

184 Detailed ways

[0021]

188 The present invention will be further described below with reference to the examples, but it should not be understood that the above subject scope of the present invention is limited to the following examples.

190 Without departing from the above-mentioned technical ideas of the present invention, various substitutions and changes can be made based on common technical knowledge and common means in the field, and all of them should be included in the scope of the present invention.

[0022]

196 Example 1:

[0023]

200 Add 20g DL-glufosinate ammonium to 200mL ammonium formate (1mol/L, pH6.5) solution, stir to dissolve, add 2g immobilized D-amino acid oxidase (50u/g), 0.2mL catalase (50000u/mL) and 2g of palladium on carbon (10%), continuously add oxygen, and stir for 24 hours at 30° C. Chiral HPLC checks that the D-enantiomer reaction is complete.

204 The reaction solution was filtered, and the filtrate was adjusted to pH 8.0 with 28% ammonia water, concentrated under reduced pressure at 80° C to about 40 mL, cooled in an ice bath, and the solid was

precipitated, dried to obtain 18g L-glufosinate-ammonium, yield 90%, purity 98.4%, ee 99.3%.

[0024]

210 Example 2:

[0025]

- 214 Add 20g DL-glufosinate ammonium to 200 mL 1 mol/L ammonium formate (pH 6.5), stir and dissolve, add 4g immobilized D-amino acid oxidase (50u/g), 0.4 mL catalase (50000u/mL) and 4g palladium Carbon (10%), continuously add oxygen, stir and react at 30° C for 12 hours, and check by chiral HPLC that the D-enantiomer reaction is complete.
- 218 The reaction liquid was filtered, and the filtrate was adjusted to pH 8.0 with 28% ammonia water, concentrated under reduced pressure at 80° C to about 40 mL, cooled in an ice bath, and the solid was precipitated, and dried to obtain 19.6g L-glufosinate-ammonium, with a yield of 98% and a purity of 99.5%. ee 99.8%.

[0026]

225 Example 3:

[0027]

- 229 Add 40g DL-glufosinate ammonium to 200 mL 1 mol/L ammonium formate (pH 6.5), stir to dissolve, add 4g immobilized D-amino acid oxidase (50u/g), 0.4 mL catalase (50000u/mL) and 4g palladium Carbon (10%), continuously add oxygen, stir and react at 30° C for 24 hours, and check by chiral HPLC that the D-enantiomer reaction is complete.
- 233 The reaction solution was filtered, and the filtrate was adjusted to pH 8.0 with 28% ammonia water, concentrated under reduced pressure at 80° C to about 40 mL, cooled in an ice bath, and the solid was precipitated, dried to obtain 34g L-glufosinate-ammonium, yield 85%, purity 96.3%, ee 98.6%.

[0028]

239 Example 4:

[0029]

- 243 Add 40g DL-glufosinate ammonium to 200 mL 1 mol/L ammonium formate (pH 6.5), stir and dissolve, add 8g immobilized D-amino acid oxidase (50u/g), 1.6 mL catalase (50000u/mL) and 8g palladium Carbon (10%), continuously add oxygen, stir and react at 30° C for 12 hours, and check by chiral HPLC that the D-enantiomer reaction is complete.
- 247 The reaction solution was filtered, and the filtrate was adjusted to pH 8.0 with 28% ammonia water,

concentrated under reduced pressure at 80° C to about 40 mL, cooled in an ice bath, and the solid was precipitated, dried to obtain 36gL-glufosinate, with a yield of 90 %, a purity of 98.3 %, and ee99.1 %.

[0030]

253 Example 5:

[0031]

257 Add 40g DL-glufosinate ammonium to 200mL 1mol/L ammonium formate (pH 6.5), stir to dissolve, add 8g immobilized D-amino acid oxidase (50u/g), 1.6mL catalase (50000u/mL) and 16g palladium Carbon (10 %), continuously add oxygen, stir and react at 30° C for 12 hours, and check by chiral HPLC that the D-enantiomer reaction is complete.

261 The reaction solution was filtered, and the filtrate was adjusted to pH 8.0 with 28 % ammonia water, concentrated under reduced pressure at 80° C to about 40 mL, cooled in an ice bath, and the solid was precipitated, dried to obtain 38gL-glufosinate, with a yield of 95 %, a purity of 99.8 %, and ee99.6 %.

[0032]

267 Example 6:

[0033]

271 Add 40g DL-glufosinate ammonium to 200mL of 1mol/L ammonium formate (pH 6.5), stir and dissolve, add the immobilized D-amino acid oxidase and palladium carbon obtained by filtration in Example 5 and 1.6mL catalase (50000u/mL), continuously introduce oxygen, and stir the reaction for 12 hours at 30° C. Chiral HPLC checks that the D-enantiomer reaction is complete.

275 The reaction solution was filtered, and the filtrate was adjusted to pH 8.0 with 28 % ammonia water, concentrated under reduced pressure at 80° C to about 40 mL, cooled in an ice bath, and the solid was precipitated, dried to obtain 36gL-glufosinate, with a yield of 99 %, a purity of 99.4 %, and ee99.5 %.



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CONFIRMATION #	4214	FILED BY	Audrey Nagelberg
PATENT CENTER #	67875401	FILING DATE	02/07/2024
CUSTOMER #	4743	FIRST NAMED INVENTOR	Brian Michael Green
CORRESPONDENCE ADDRESS	-	AUTHORIZED BY	Joshua Elliott

Documents

TOTAL DOCUMENTS: 6

DOCUMENT	PAGES	DESCRIPTION	SIZE (KB)
NPL_Howdollink.pdf	2	Non Patent Literature	258 KB
NPL_P22256GABTecoli.pdf	8	Non Patent Literature	504 KB
NPL_Ricca.pdf	24	Non Patent Literature	3659 KB
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PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875				Application or Docket Number 18/435,340		Filing Date 02/07/2024		☐ To be Mailed	
ENTITY: ☒ LARGE ☐ SMALL ☐ MICRO									
APPLICATION AS FILED - PART I									
	(Column 1)	(Column 2)							
FOR	NUMBER FILED	NUMBER EXTRA			RATE (\$)			FEE (\$)	
☐ BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A			N/A				
☐ SEARCH FEE (37 CFR 1.16(k), (l), or (m))	N/A	N/A			N/A				
☐ EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A			N/A				
TOTAL CLAIMS (37 CFR 1.16(j))	minus 20 =	*			x \$100 =				
INDEPENDENT CLAIMS (37 CFR 1.16(h))	minus 3 =	*			x \$480 =				
☐ APPLICATION SIZE FEE (37 CFR 1.16(s))	If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$310 (\$155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).								
☐ MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))									
* If the difference in column 1 is less than zero, enter "0" in column 2.					TOTAL				
APPLICATION AS AMENDED - PART II									
	(Column 1)		(Column 2)	(Column 3)					
AMENDMENT	03/10/2025	CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA			RATE (\$)	ADDITIONAL FEE (\$)
	Total (37 CFR 1.16(j))	* 6	Minus	** 20	= 0			x \$200 =	0
	Independent (37 CFR 1.16(h))	* 1	Minus	*** 3	= 0			x \$600 =	0
	☐ Application Size Fee (37 CFR 1.16(s))								
	☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))								
								TOTAL ADD'L FEE	0
AMENDMENT		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA			RATE (\$)	ADDITIONAL FEE (\$)
	Total (37 CFR 1.16(j))	*	Minus	**	=			x \$0 =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=			x \$0 =	
	☐ Application Size Fee (37 CFR 1.16(s))								
	☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))								
								TOTAL ADD'L FEE	
* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.								HSLIE	
** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".								/TIFFANY Y VINES/	
*** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".									
The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1.									

This collection of information is required by 37 CFR 1.16. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.



UNITED STATES
PATENT AND TRADEMARK OFFICE

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ELECTRONIC PAYMENT RECEIPT

APPLICATION # 18/435,340	RECEIPT DATE / TIME 03/10/2025 06:17:09 PM Z ET	ATTORNEY DOCKET # 32471/M202647G
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Title of Invention

METHODS FOR MAKING L-GLUFOSINATE

Application Information

APPLICATION TYPE	Utility - Nonprovisional Application under 35 USC 111(a)	PATENT #	-
CONFIRMATION #	4214	FILED BY	Rebecca Walsh
PATENT CENTER #	69518980	AUTHORIZED BY	Joshua Elliott
CUSTOMER #	4743	FILING DATE	02/07/2024
CORRESPONDENCE ADDRESS	-	FIRST NAMED INVENTOR	Brian Michael Green

Payment Information

PAYMENT METHOD CARD / 0428	PAYMENT TRANSACTION ID E202530118028002	PAYMENT AUTHORIZED BY Rebecca Walsh
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FEE CODE	DESCRIPTION	ITEM PRICE(\$)	QUANTITY	ITEM TOTAL(\$)
1252	EXTENSION FOR RESPONSE WITHIN SECOND MONTH, EXCEPT PROVISIONAL APPLICATIONS	690.00	1	690.00
1814	STATUTORY DISCLAIMER, INCLUDING TERMINAL DISCLAIMER	183.00	1	183.00
TOTAL AMOUNT:				\$873.00

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for filing date (see 37 CFR 1.53(b)-(d))

and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

PETITION FOR EXTENSION OF TIME UNDER 37 CFR 1.136(a) (NOT for Provisional Applications)		Docket Number (Optional) 32471/M202647G																														
Application Number 18/435,340-Conf. #4214	Filed February 7, 2024																															
For METHODS FOR MAKING L-GLUFOSINATE																																
Art Unit 1652	Examiner G. Raghu																															
<p>This is a request under the provisions of 37 CFR 1.136(a) to extend the period for filing a reply in the above-identified application.</p> <p>The requested extension and fee are as follows (check time period desired and enter the appropriate fee below):</p> <table style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="width: 40%;"></th> <th style="width: 15%; text-align: center;"><u>Fee</u></th> <th style="width: 15%; text-align: center;"><u>Small Entity Fee</u></th> <th style="width: 15%; text-align: center;"><u>Micro Entity Fee</u></th> <th style="width: 15%;"></th> </tr> </thead> <tbody> <tr> <td>☐ One month (37 CFR 1.17(a)(1))</td> <td style="text-align: center;">\$235</td> <td style="text-align: center;">\$94</td> <td style="text-align: center;">\$47</td> <td style="text-align: center;">\$ _____</td> </tr> <tr> <td>☒ Two months (37 CFR 1.17(a)(2))</td> <td style="text-align: center;">\$690</td> <td style="text-align: center;">\$276</td> <td style="text-align: center;">\$138</td> <td style="text-align: center;">\$ <u>690.00</u></td> </tr> <tr> <td>☐ Three months (37 CFR 1.17(a)(3))</td> <td style="text-align: center;">\$1,590</td> <td style="text-align: center;">\$636</td> <td style="text-align: center;">\$318</td> <td style="text-align: center;">\$ _____</td> </tr> <tr> <td>☐ Four months (37 CFR 1.17(a)(4))</td> <td style="text-align: center;">\$2,495</td> <td style="text-align: center;">\$998</td> <td style="text-align: center;">\$499</td> <td style="text-align: center;">\$ _____</td> </tr> <tr> <td>☐ Five months (37 CFR 1.17(a)(5))</td> <td style="text-align: center;">\$3,395</td> <td style="text-align: center;">\$1,358</td> <td style="text-align: center;">\$679</td> <td style="text-align: center;">\$ _____</td> </tr> </tbody> </table> <p>☐ Applicant asserts small entity status. See 37 CFR 1.27.</p> <p>☐ Applicant certifies micro entity status. See 37 CFR 1.29. Form PTO/SB/15A or B or equivalent must either be enclosed or have been submitted previously.</p> <p>☐ A check in the amount of the fee is enclosed.</p> <p>☐ Payment by credit card. Form PTO-2038 is attached.</p> <p>☐ The Director has already been authorized to charge fees in this application to a Deposit Account.</p> <p>☒ The Director is hereby authorized to charge any fees which may be required, or credit any overpayment, to Deposit Account Number <u>13-2855</u>.</p> <p>☒ Payment made via USPTO patent electronic filing system.</p> <p>WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.</p> <p>I am the</p> <p>☐ applicant.</p> <p>☒ attorney or agent of record. Registration number <u>43,603</u>.</p> <p>☐ attorney or agent acting under 37 CFR 1.34. Registration number _____.</p> <div style="display: flex; justify-content: space-between; margin-top: 20px;"> <div style="width: 45%;"> <p style="text-align: center;">_____ /Joshua T. Elliott/ Signature</p> <p style="text-align: center;">_____ Joshua T. Elliott Typed or printed name</p> </div> <div style="width: 45%;"> <p style="text-align: center;">_____ March 10, 2025 Date</p> <p style="text-align: center;">_____ (312) 474-6300 Telephone Number</p> </div> </div> <p>NOTE: This form must be signed in accordance with 37 CFR 1.33. See 37 CFR 1.4 for signature requirements and certifications. Submit multiple forms if more than one signature is required, see below*.</p> <div style="border: 1px solid black; padding: 5px; margin-top: 10px;"> ☐ * Total of <u>1</u> forms are submitted. </div>				<u>Fee</u>	<u>Small Entity Fee</u>	<u>Micro Entity Fee</u>		☐ One month (37 CFR 1.17(a)(1))	\$235	\$94	\$47	\$ _____	☒ Two months (37 CFR 1.17(a)(2))	\$690	\$276	\$138	\$ <u>690.00</u>	☐ Three months (37 CFR 1.17(a)(3))	\$1,590	\$636	\$318	\$ _____	☐ Four months (37 CFR 1.17(a)(4))	\$2,495	\$998	\$499	\$ _____	☐ Five months (37 CFR 1.17(a)(5))	\$3,395	\$1,358	\$679	\$ _____
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TERMINAL DISCLAIMER TO OBVIATE A DOUBLE PATENTING REJECTION OVER A "PRIOR" PATENT	Docket Number (Optional) 32471/M202647G
<p>In re Application of: Brian Michael Green et al.</p> <p>Application No.: 18/435,340-Conf. #4214</p> <p>Filed: February 7, 2024</p> <p>For: METHODS FOR MAKING L-GLUFOSINATE</p> <p>The applicant, <u>BASF SE</u>, owner of _____ percent interest in the instant application hereby disclaims, except as provided below, the terminal part of the statutory term of any patent granted on the instant application which would extend beyond the expiration date of the full statutory term of prior patent No. <u>11,905,538</u> as the term of said prior patent is presently shortened by any terminal disclaimer. The applicant hereby agrees that any patent so granted on the instant application shall be enforceable only for and during such period that it and the prior patent are commonly owned. This agreement runs with any patent granted on the instant application and is binding upon the grantee, its successors or assigns.</p> <p>In making the above disclaimer, the applicant does not disclaim the terminal part of the term of any patent granted on the instant application that would extend to the expiration date of the full statutory term of the prior patent, "as the term of said prior patent is presently shortened by any terminal disclaimer," in the event that said prior patent later:</p> <ul style="list-style-type: none"> expires for failure to pay a maintenance fee; is held unenforceable; is found invalid by a court of competent jurisdiction; is statutorily disclaimed in whole or terminally disclaimed under 37 CFR 1.321; has all claims canceled by a reexamination certificate; is reissued; or is in any manner terminated prior to the expiration of its full statutory term as presently shortened by any terminal disclaimer. <p>1. ☐ The undersigned is the applicant. If the applicant is an assignee, the undersigned is authorized to act on behalf of the assignee.</p> <p>I hereby acknowledge that any willful false statements made are punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.</p> <p>2. ☒ The undersigned is an attorney or agent of record. Reg. No. <u>43,603</u></p> <div style="display: flex; justify-content: space-between; margin-top: 20px;"> <div style="width: 60%;"> <p style="text-align: center;">_____ /Joshua T. Elliott/ Signature</p> <p style="text-align: center;">_____ Joshua T. Elliott Typed or printed name</p> <p style="text-align: center;">_____ Attorney for Applicant(s) Title</p> </div> <div style="width: 35%; text-align: center;"> <p>_____ March 10, 2025 Date</p> <p>_____ (312) 474-6300 Telephone Number</p> </div> </div> <p>☒ Terminal disclaimer fee under 37 CFR 1.20(d) included.</p> <p style="text-align: center;">WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.</p>	

APPENDIX A

Additional experimental data

[0001] Methodology - green house experiments

[0002] Greenhouse trials were conducted to evaluate the phytotoxicity of L-glufosinate ammonium. Glufosinate-ammonium (GA) and L-glufosinate-ammonium (LGA) were used as technical grades at 600 g/L. All treatments were applied with an adjuvant Sodium-lauryl-ester-sulfate (SLES) with adjuvant ratios typically used in commercial with Glufosinate formulations. The treatments were applied with a track sprayer using flat fan nozzles, a water volume of 200 L/ha and one replicate per treatment.

[0003] The herbicide combinations were applied post-emergence to soybeans at growth stage GS13. Two glufosinate-tolerant soybean cultivars were used: (i) GLXMA (S1) – CZ 1660 GTLL) and (ii) GLXMA (S1) – CZ 3519 GTLL. A visual crop injury assessment (i.e., % crop injury compared to an untreated control) was made at 7 days after treatment (DAT).

[0004] Experimental Results – greenhouse

[0005] The results of the phytotoxicity experiment are summarized in table 2e below. Surprisingly, all LGA treatments are causing less phytotoxicity than respective GA treatments applied at the same molecular weight. There is some difference in sensitivity to glufosinate between the two soybean varieties but the difference in crop response from LGA as compared to GA is visible in both varieties and at all three application rates 300, 600 and 1200 g/ha. This was not expected.

Table 2e: Ratings of herbicidal activity (phytotoxicity) in an greenhouse experiment against the above mentioned glufosinate tolerant soybeans (GLXMA) after a single post-emergence treatments with racemate glufosinate-ammonium (GA) and L-glufosinate-ammonium (LGA) rated 7 DAA.

Field crop	LGA 300 g/ha	GA 300 g/ha	LGA 600 g/ha	GA 600 g/ha	LGA 1200 g/ha	GA 1200 g/ha
GLXMA (CZ1660)	0	5	10	15	10	40
GLXMA (CZ3519)	5	10	10	35	15	70
average	2.5	7.5	10	25	12.5	55

Docket No.: 32471/M202647G
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Brian Michael Green et al.

Application No.: 18/435,340

Confirmation No.: 4214

Filed: February 7, 2024

Art Unit: 1652

For: METHODS FOR MAKING L-GLUFOSINATE

Examiner: G. Raghu

AMENDMENT IN RESPONSE TO NON-FINAL OFFICE ACTION UNDER 37 C.F.R. § 1.111

MS Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Commissioner:

INTRODUCTORY COMMENTS

In response to the Office Action dated October 10, 2024, please amend the above-identified U.S. patent application as follows:

Amendments to the Claims are reflected in the listing of claims which begins on page 2 of this paper.

Remarks/Arguments begin on page 4 of this paper.

AMENDMENTS TO THE CLAIMS

1. (Currently amended) A formulation comprising:
L-glufosinate ammonium in an amount from 10-30% by weight of the formulation and D-glufosinate, wherein the formulation has greater than about 90% L-glufosinate compared to the sum of the D-glufosinate and the L-glufosinate present in the formulation;
one or more additional components selected from the group consisting of:
sodium alkyl ether sulfate in an amount from 10-40% by weight of the formulation;
1-methoxy-2-propanol in an amount from 0.5-2% by weight of the formulation;
dipropylene glycol in an amount from 4-18% by weight of the formulation;
alkyl polysaccharide in an amount from 4-20% by weight of the formulation;
and
water as the balance of the formulation.
2. (Original) The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 36.75% by weight of the formulation.
3. (Original) The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 31.6% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; dipropylene glycol in an amount of 8.6% by weight of the formulation; alkyl polysaccharide in an amount of 9.8% by weight of the formulation; and water in an amount of 24.5% by weight of the formulation.
4. (Original) The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 15.8% by weight of the formulation; 1-methoxy-2-propanol in an amount of

0.5% by weight of the formulation; dipropylene glycol in an amount of 4.3% by weight of the formulation; alkyl polysaccharide in an amount of 4.9% by weight of the formulation; and water in an amount of 62.25% by weight of the formulation.

5. (Original) The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 12.25% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 58.45% by weight of the formulation.

6. (Original) The formulation of claim 1, wherein the L-glufosinate composition comprises L-glufosinate ammonium in an amount of 24.5% by weight of the formulation; and wherein the one or more additional components comprise sodium alkyl ether sulfate in an amount of 22.1% by weight of the formulation; 1-methoxy-2-propanol in an amount of 1% by weight of the formulation; alkyl polysaccharide in an amount of 6.2% by weight of the formulation; and water in an amount of 46.2% by weight of the formulation.

REMARKS

Upon entry of this amendment, claims 1-6 will be pending. Claim 1 has been amended to recite that the formulation comprises L-glufosinate ammonium in an amount from 10-30% by weight of the formulation and D-glufosinate, wherein the formulation has greater than about 90% L-glufosinate compared to the sum of the D-glufosinate and the L-glufosinate present in the formulation, support for which is found in the application as filed (see, e.g., page 15, line 23 – page 16, line 2). No new matter has been added.

The rejections from the Office Action are addressed below. Reconsideration of the application is requested in view of the foregoing amendments and remarks that follow.

Double Patenting Rejection

Claims 1-6 of the instant application were rejected as being unpatentable over claims 1-15 of US 11,905,538.

A terminal disclaimer referencing U.S. Patent No. 11,905,538 is submitted herewith. In view thereof, Applicant respectfully requests that this rejection be withdrawn.

Claim Rejections – 35 U.S.C. § 102 – Anticipation

Claims 1-6 were rejected as being anticipated by **Long DA (US 8,445,406)**. Applicant respectfully traverses based on the amended claims.

Long discloses that the glufosinate used in the aqueous herbicidal formulations is **racemic** glufosinate:

Note that the compounds of formula (I) contain an asymmetric carbon atom. The L enantiomer has been observed to be the biologically active isomer. The formula (I) therefore is intended to encompass all stereoisomers and mixtures thereof, particularly the racemate, and the biologically active enantiomer in each case. Examples of active ingredients of the formula (I) include glufosinate and/or its ammonium salt such as in a racemic mixture; i.e., 2-amino-4-[hydroxy(methyl)phosphinoyl]butanoic acid and its ammonium salt, the L enantiomer of glufosinate and its ammonium salt or other salts such as potassium, sodium, diethylamine, triethylamine, bilanslow/bialaphos; e.g., L-2-amino-4-[hydroxy(methyl)phosphinoyl]butanoyl-L-alanyl-L-alanine and its sodium salt.

Long at column 3, lines 10-23.

As indicated above, claim 1 has been amended to recite that “the formulation has greater than about 90% L-glufosinate compared to the sum of the D-glufosinate and the L-glufosinate present in the formulation.”

Thus, the recited L-glufosinate composition in amended claim 1 is not racemic glufosinate.

In view thereof, the claims are not anticipated by the cited art, and Applicant respectfully requests that the rejection be withdrawn.

Claim Rejections – 35 U.S.C. § 103 – Obviousness

Claims 1-6 were rejected as being unpatentable over **Long** as applied to claims 1-6 and in view of **Bhoge et al. (US 10,888,095)**. Applicant respectfully traverses based on the amended claims.

As indicated above, Long discloses that the glufosinate used in the aqueous herbicidal formulations is racemic glufosinate.

However, as also indicated above, claim 1 has been amended such that “the formulation has greater than about 90% L-glufosinate compared to the sum of the D-glufosinate and the L-glufosinate present in the formulation.” .

Thus, none of the cited art (alone or in combination) disclose or suggest the claimed formulation with the recited L-glufosinate ammonium.

Moreover, attached hereto as Appendix A is additional experimental data that has been performed by Applicant showing that the claimed herbicidal formulation recited in claim 1 has unexpected results.

As shown in Appendix A, tests were carried out using racemic glufosinate and L-glufosinate to determine the phytotoxicity to glufosinate-tolerant soybean at various concentrations (i.e., 300 g/ha, 600 g/ha, and 1200 g/ha). As shown, the L-glufosinate surprisingly resulted in less phytotoxicity to the soybeans than the same amount of racemic glufosinate (i.e., LGA 300 vs. GA 300, etc.) and when comparing the same amounts of L-glufosinate (i.e., LGA 300 vs. GA 600 or LGA 600 vs. GA 1200).

In view of the above, the claimed subject matter is not obvious over the cited art. Applicant respectfully requests that the rejection be withdrawn.

Conclusion

In view of the above, Applicant believes the claims are in condition for allowance and request notification of the same. Should the Office have any questions regarding this response or the application, the Office is invited to contact the undersigned agent at the telephone number indicated below.

The Director is hereby authorized to charge any additional fees which may be required with respect to this communication, or credit any overpayment, to Deposit Account No. 13-2855, under Order No. 32471/M202647G.

Dated: March 10, 2025

Respectfully submitted,

Electronic signature: /Joshua T. Elliott/
Joshua T. Elliott
Registration No.: 43,603
MARSHALL, GERSTEIN & BORUN LLP
233 S. Wacker Drive
6300 Willis Tower
Chicago, Illinois 60606-6357
(312) 474-6300
Attorney for Applicant



UNITED STATES
PATENT AND TRADEMARK OFFICE

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ELECTRONIC ACKNOWLEDGEMENT RECEIPT

APPLICATION # 18/435,340	RECEIPT DATE / TIME 03/10/2025 06:17:09 PM Z ET	ATTORNEY DOCKET # 32471/M202647G
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Title of Invention

METHODS FOR MAKING L-GLUFOSINATE

Application Information

APPLICATION TYPE	Utility - Nonprovisional Application under 35 USC 111(a)	PATENT #	-
CONFIRMATION #	4214	FILED BY	Rebecca Walsh
PATENT CENTER #	69518980	FILING DATE	02/07/2024
CUSTOMER #	4743	FIRST NAMED INVENTOR	Brian Michael Green
CORRESPONDENCE ADDRESS	-	AUTHORIZED BY	Joshua Elliott

Documents

TOTAL DOCUMENTS: 6

DOCUMENT		PAGES	DESCRIPTION	SIZE (KB)
Amendment.pdf		6	-	52 KB
Amendment-A....pdf	(1-1)	1	Amendment/Request for Reconsideration-After Non- Final Rejection	16 KB
Amendment-CLM.pdf	(2-3)	2	Claims	19 KB
Amendment-REM.pdf	(4-6)	3	Applicant Arguments/Remarks Made in an Amendment	38 KB
Petition for Extension of Time Under 37 CFR 1136 a PTO AIA-22.pdf		1	Extension of Time	31 KB
Terminal Disclaimer Double Patenting Rejection -- Prior Patent PTO AIA-26.pdf		1	Terminal Disclaimer Filed	20 KB

Appendix A.pdf	1	Applicant Arguments/Remarks Made in an Amendment	40 KB
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Digest

DOCUMENT	MESSAGE DIGEST(SHA-512)
Amendment.pdf	64E4BFAC1D6434545DC770EEA967B94B79395C071E6647D4E F2BA24E3497BE974BB8A6A41E45A4FF17781EC78053ABE136 CE9A61A9658C519F88C9367A73C28E
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Petition for Extension of Time Under 37 CFR 1136 a PTO AIA-22.pdf	C3276F40B2ADE79C5B78E4D96D4F5C4A07E0042D6AAA3A68 9CD23A63CCE7701C8F1F15BE579E8FE98B44DC3DD9AF1671 DF8E654B5E70F18A4F81C011D1BA4FBD
Terminal Disclaimer Double Patenting Rejection -- Prior Patent PTO AIA-26.pdf	0D61DE7C15C0632C9544E749804702915E26816AA9C7057C5 10302A9E8D9DDB5D710E8554F07D8A1F40D8B55D0EDBCFD0 120C20E42A53754E8D573942F578621
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This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

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National Stage of an International Application under 35 U.S.C. 371

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New International Application Filed with the USPTO as a Receiving Office

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and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Application Number * 18/435,340 *	Application/Control No. 18/435,340	Applicant(s)/Patent under Reexamination Green et al.	
	Examiner RAGHU, GANAPATHIRAM	Art Unit 1652	
Document Code - DISQ		Internal Document - DO NOT MAIL	

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Date Filed: <u>10 March 2025</u>	This patent is subject to a Terminal Disclaimer	

Approved/Disapproved by:
/LAWANA R HIXON/ Technology Center: <u>OPLC</u> Telephone: <u>(571)272-6074</u> _____

Issue Classification 	Application/Control No. 18/435,340	Applicant(s)/Patent Under Reexamination Green et al.
	Examiner GANAPATHIRAMA RAGHU	Art Unit 1652

CPC						
Symbol					Type	Version
C12P	/	13	/	02	F	2013-01-01
C12P	/	41	/	00	I	2013-01-01
C12P	/	9	/	00	I	2013-01-01
C12P	/	13	/	04	I	2013-01-01
A01N	/	25	/	30	I	2013-01-01
A01N	/	57	/	20	I	2013-01-01
C12Y	/	104	/	03003	I	2013-01-01
C12Y	/	206	/	01019	I	2013-01-01
C12N	/	1	/	20	I	2013-01-01
C12N	/	9	/	10	I	2013-01-01

CPC Combination Sets				
Symbol	Type	Set	Ranking	Version
A01N / 57 / 20	I	1	1	2013-01-01
A01N / 25 / 30	I	1	2	2013-01-01
A01N / 57 / 18	I	1	3	2013-01-01

NONE (Assistant Examiner) _____ (Date) _____		Total Claims Allowed: 6	
/GANAPATHIRAMA RAGHU/ Primary Examiner, Art Unit 1652 (Primary Examiner) _____ (Date) 19 March 2025		O.G. Print Claim(s) 1	O.G. Print Figure None

U.S. Patent and Trademark Office

Part of Paper No.: 20250319

Issue Classification 	Application/Control No. 18/435,340	Applicant(s)/Patent Under Reexamination Green et al.
	Examiner GANAPATHIRAMA RAGHU	Art Unit 1652

INTERNATIONAL CLASSIFICATION				
CLAIMED				
C12P13/02	/	13	/	02
C12P41/00	/	41	/	00
C12P9/00	/	9	/	00
C12P13/04	/	13	/	04
A01N25/30	/	25	/	30
A01N57/20	/	57	/	20
C12N1/20	/	1	/	20
C12N9/10	/	9	/	10
NON-CLAIMED				
	/		/	

US ORIGINAL CLASSIFICATION	
CLASS	SUBCLASS

CROSS REFERENCES(S)						
CLASS	SUBCLASS (ONE SUBCLASS PER BLOCK)					

NONE		Total Claims Allowed:	
(Assistant Examiner)	(Date)	6	
/GANAPATHIRAMA RAGHU/ Primary Examiner, Art Unit 1652	19 March 2025	O.G. Print Claim(s)	O.G. Print Figure
(Primary Examiner)	(Date)	1	None

U.S. Patent and Trademark Office

Part of Paper No.: 20250319

Issue Classification 	Application/Control No. 18/435,340	Applicant(s)/Patent Under Reexamination Green et al.
	Examiner GANAPATHIRAMA RAGHU	Art Unit 1652

☐ Claims renumbered in the same order as presented by applicant ☐ CPA ☒ T.D. ☐ R.1.47															
CLAIMS															
Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original
1	1														
2	2														
3	3														
4	4														
5	5														
6	6														

NONE (Assistant Examiner) _____ (Date) _____		Total Claims Allowed: 6	
/GANAPATHIRAMA RAGHU/ Primary Examiner, Art Unit 1652 (Primary Examiner) _____ (Date) 19 March 2025		O.G. Print Claim(s) 1	O.G. Print Figure None

<i>Search Notes</i> 	Application/Control No. 18/435,340	Applicant(s)/Patent Under Reexamination Green et al.
	Examiner GANAPATHIRAMA RAGHU	Art Unit 1652

CPC - Searched*		
Symbol	Date	Examiner
C12P13/02;A01N25/30;A01N57/20;C12N1/20;C12N9/10;C12P9/00; C 12P13/04;C12P41/00;C12Y104/03003;C12Y206/01019;C12N9/ 0024	03/19/2025	GR

CPC Combination Sets - Searched*		
Symbol	Date	Examiner

US Classification - Searched*			
Class	Subclass	Date	Examiner

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

Search Notes		
Search Notes	Date	Examiner
PE2E Search: US-PGPUB, USPAT, USCOR, FIT, FPRS, EPO, JPO, DERWENT, IBM_TDB, Inventors search (see attachment)	03/19/2025	GR
Google Scholar (see attachment)	09/27/2024	GR

Interference Search			
US Class/CPC Symbol	US Subclass/CPC Group	Date	Examiner
	C12P13/02;A01N25/30;A01N57/20;C12N1/20; C12N9/10;C12P9/00;C12P13/04;C12P41/00; C12Y104/03003;C12Y206/01019;C12N9/0024	03/19/2025	GR

/GANAPATHIRAMA RAGHU/ Primary Examiner, Art Unit 1652	03/19/2025
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PTO/SB/08a (07-09)

Approved for use through 05/31/2024. OMB 0651-0031

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

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Substitute for form 1449/PTO <h1>INFORMATION DISCLOSURE STATEMENT BY APPLICANT</h1> <i>(Use as many sheets as necessary)</i>				Complete if Known	
				Application Number	18/435,340-Conf. #4214
				Filing Date	February 7, 2024
				First Named Inventor	Brian Michael Green
				Art Unit	1652
				Examiner Name	G. Raghu
				Attorney Docket Number	32471/M202647G
Sheet	1	of	2		

[illegible][illegible]

Examiner Signature	/GANAPATHIRAMA RAGHU/	Date Considered	03/19/2025
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*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant. ¹ Applicant's unique citation designation number (optional). ² See Kinds Codes of USPTO Patent Documents at www.uspto.gov or MPEP 901.04. ³ Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). ⁴ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁵ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁶ Applicant is to place a check mark here if English language Translation is attached.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /G.R/

PTO/SB/08b (07-09)

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Substitute for form 1449/PTO INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>				Complete if Known	
				Application Number	18/435,340-Conf. #4214
				Filing Date	February 7, 2024
				First Named Inventor	Brian Michael Green
				Art Unit	1652
				Examiner Name	G. Raghu
Sheet	2	of	2	Attorney Docket Number	32471/M202647G

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials*	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T ²
		How do I link to a specific version of a UniProt entry, downloaded from the Internet at: < https://www.uniprot.org/help/link_old_versions > retrieved September 25, 2024.	
		P22256 GABT_ECOLI, downloaded from the Internet: < https://www.uniprot.org/uniprotkb/P22256/history >, retrieved September 25, 2024.	
		Ricca et al., Multi-enzymatic cascade reactions: overview and perspectives, Adv. Synth. Catal., 353:2239-62 (2011).	

Examiner Signature	/GANAPATHIRAMA RAGHU/	Date Considered	03/19/2025
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*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ Applicant's unique citation designation number (optional). ² Applicant is to place a check mark here if English language Translation is attached.



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BIB DATA SHEET

CONFIRMATION NO. 4214

SERIAL NUMBER	FILING or 371(c) DATE	CLASS	GROUP ART UNIT	ATTORNEY DOCKET NO.		
18/435,340	02/07/2024	504	1652	32471/M202647G		
APPLICANTS BASF SE, Ludwigshafen, GERMANY; INVENTORS Brian Green Michael, Lutherville, MD; Michelle Gradley Lorraine, Canterbury, UNITED KINGDOM; ** CONTINUING DATA ***** This application is a CON of 18/179,136 03/06/2023 PAT 11913048 which is a CON of 17/530,018 11/18/2021 PAT 11905538 which is a CON of 16/997,133 08/19/2020 PAT 11560577 which is a CON of 16/287,290 02/27/2019 PAT 10781465 which is a CON of 15/787,448 10/18/2017 PAT 10260078 which is a DIV of 15/445,254 02/28/2017 PAT 9834802 which claims benefit of 62/413,240 10/26/2016 ABN and claims benefit of 62/336,989 05/16/2016 ABN and claims benefit of 62/302,421 03/02/2016 ABN ** FOREIGN APPLICATIONS ***** ** IF REQUIRED, FOREIGN FILING LICENSE GRANTED ** 02/21/2024						
Foreign Priority claimed ☐ Yes ☒ No 35 USC 119(a-d) conditions met ☐ Yes ☒ No Verified and /GANAPATHIRAMA RAGHU/ Acknowledged Examiner's Signature		☐ Met after Allowance GR Initials	STATE OR COUNTRY MD	SHEETS DRAWINGS 2	TOTAL CLAIMS 6	INDEPENDENT CLAIMS 1
ADDRESS MARSHALL, GERSTEIN & BORUN LLP 233 SOUTH WACKER DRIVE 6300 WILLIS TOWER CHICAGO, IL 60606-6357 UNITED STATES						
TITLE METHODS FOR MAKING L-GLUFOSINATE						
FILING FEE RECEIVED 0.00	FEES: Authority has been given in Paper No. _____ to charge/credit DEPOSIT ACCOUNT No. _____ for following:			☐ All Fees ☐ 1.16 Fees (Filing) ☐ 1.17 Fees (Processing Ext. of time) ☐ 1.18 Fees (Issue) ☐ Other _____		

BIB (Rev. 05/07).

	☐ Credit
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PE2E SEARCH - Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	British Equivalents	Time Stamp
L1	50	<p>(Artificial Intelligence) Similar to: 18/435,340 with 0 CPC Selections and 0 Text Selections</p> <p>Text: (US-11905538-B2 OR US-11732281-B2 OR US-20170253897-A1 OR US-20220064680- A1 OR US- 20200385767-A1 OR US-11913048-B2 OR US-20180030487-A1 OR US-20230265472- A1 OR US-10781465- B2 OR US- 20210395790-A1 OR US-11560577-B2 OR CN-116121315-A OR US-20240301452-A1 OR US-9834802-B2 OR US-10260078-B2 OR CN-109072261-B OR US-20190185889-A1 OR JP-2023061954-A OR JP-2021078506-A OR JP-7041066-B2 OR JP-2024513190-A OR US-20240182502-A1 OR WO-2022207753- A1 OR CN-117120625- A OR CA-3213964-A1 OR KR-20230164054-A OR AU-2015326965-A1 OR EP-0930823-B2 OR DE-69718027-T3 OR US-10888095-B2 OR WO-2024130353-A1 OR AU-2022405733-A1 OR KR-20240121807-A OR AU-2012347377-B2 OR EP-0930823-B1 OR CA-2937009-C OR CA- 2868134-A1 OR AU- 2012347377-A1 OR AU-2022407771-A1 OR IL-262789-A OR US- 20140371069-A1 OR US-20080045415-A1 OR US-20170265477- A1 OR JP-2000517334-</p>	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN))	ADJ	ON	ON	2024/09/27 03:00 PM

L2	5	A OR US-9468213-B2 OR CA-3089399-A1 OR US-10729129-B2 OR CA-3240064-A1 OR US-20190110469-A1 OR CA-2715672- A1).did. ("5130246").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:19 PM
L3	5	("5162212").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:20 PM
L4	5	("5587319").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:21 PM
L5	5	("5753470").pn.	(US-PGPUB; USPAT;	ADJ	ON	ON	2024/09/27

			USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)				03:22 PM
L6	5	("5767309").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:22 PM
L7	5	("5877013").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:23 PM
L8	5	("5919669").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:29 PM

L9	5	("5962281").pn.	NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:30 PM
L10	6	("6051408").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:30 PM
L11	6	("6335186").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:35 PM
L12	6	("6936444").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP,	ADJ	ON	ON	2024/09/27 03:35 PM

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L14	2	("7939702").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:37 PM
L15	2	("7939709").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:38 PM

L16	2	("8383137").pn.	RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:39 PM
L17	2	("8445406").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:39 PM
L18	3	("8716184").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:41 PM
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L20	2	("8981142").pn.	CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:42 PM
L21	11	"20080045415"	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:43 PM
L22	2	("8637432").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:45 PM

L23	5	("6165939").pn.	TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:46 PM
L24	2	("7842647").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:48 PM
L25	4	("10260078").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:51 PM
L26	2	("10781465").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:52 PM

L27	2	("10888095").pn.	CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:53 PM
L28	5	("9834802").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:56 PM
L29	2	("11560577").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:56 PM

L30	2	("11634442").pn.	VN); FPRS; EPO; JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:57 PM
L31	2	("11905538").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:57 PM
L32	2	("11913048").pn.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 03:58 PM
L33	3239	(L-glufosinate OR L- phosphinothricin OR 2- amino-4- [hydroxy(methyl)phosph oryl]butanoic acid)	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA,	ADJ	ON	ON	2024/09/27 04:04 PM

			EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)				
L34	103	L33 AND (Sodium alkyl ether OR Sodium lauryl ether sulfate OR Sodium laureth sulphate OR Sodium lauryl ether sulphate)	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:05 PM
L35	72	L34 AND (Propylene glycol methyl ether OR PGME OR 1-methoxy-2-propanol OR Methoxypropanol OR α -Propylene glycol monomethyl ether)	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:05 PM
L36	71	L35 AND (Dipropylene glycol OR 1,1'-Oxybis(1-propanol) OR 1,1'-Oxybis(2-propanol))	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO;	ADJ	ON	ON	2024/09/27 04:06 PM

L37	70	L36 AND (Alkyl polysaccharide OR Alkyl polyglucoside OR Alkyl polyglycoside)	JPO; DERWENT; IBM_TDB) (US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:06 PM
L38	0	L37 AND @ad<="20160516"	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:07 PM
L39	262658	(C12P13/02;A01N25/30;A01N57/20;C12N1/20;C12N9/10;C12P9/00;C12P13/04;C12P41/00;C12Y104/03003;C12Y206/01019;C12N9/0024). CPC.	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:09 PM
L40	66	L39 AND L37	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB,	ADJ	ON	ON	2024/09/27 04:09 PM

L41	200	(green brian michael OR gradley\$).in.	HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:10 PM
L42	44	L41 AND L37	(US-PGPUB; USPAT; USOCR; FIT (AU, AP, AT, BE, BG, BR, BY, CA, CH, CN, CS, CU, CZ, DD, DE, DK, EA, EE, EP, ES, FI, FR, GB, HR, HU, ID, IE, IL, IS, IT, JP, KR, LT, LU, LV, MA, OA, RU, SU, WO, MC, MD, MY, NL, NO, NZ, PH, PL, PT, RO, RS, SE, SG, SI, SK, TH, TN, TR, TW, UA, VN); FPRS; EPO; JPO; DERWENT; IBM_TDB)	ADJ	ON	ON	2024/09/27 04:11 PM

PE2E SEARCH - Search History (Interference)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	British Equivalents	Time Stamp
N1	403	(L-glufosinate OR L-phosphinothricin OR 2-amino-4-[hydroxy(methyl)phosphoryl]butanoic acid)	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:29 AM
N2	37	N1 AND (Sodium alkyl ether OR Sodium lauryl ether sulfate OR Sodium laureth	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:30 AM

N3	32	sulphate OR Sodium lauryl ether sulphate) N2 AND (Propylene glycol methyl ether OR PGME OR 1-methoxy-2-propanol OR Methoxypropanol OR α -Propylene glycol monomethyl ether)	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:30 AM
N4	32	N3 AND (Dipropylene glycol OR 1,1'-Oxybis(1-propanol) OR 1,1'-Oxybis(2-propanol))	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:31 AM
N5	32	N4 AND (Alkyl polysaccharide OR Alkyl polyglucoside OR Alkyl polyglycoside)	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:31 AM
N6	0	N5 AND @ad<="20160516"	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:31 AM
N7	23202	(C12P13/02;A01N25/30;A01N57/20;C12N1/20;C12N9/10;C12P9/00;C12P13/04;C12P41/00;C12Y104/03003;C12Y206/01019;C12N9/0024). CPC.	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:36 AM
N8	32	N7 AND N5	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:36 AM
N9	40	(green brian michael OR gradley\$).in.	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:38 AM
N10	29	N9 AND N5	(US-PGPUB; USPAT)	ADJ	ON	ON	2025/03/19 10:39 AM



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NOTICE OF ALLOWANCE AND FEE(S) DUE

4743 7590 04/03/2025
MARSHALL, GERSTEIN & BORUN LLP
233 SOUTH WACKER DRIVE
6300 WILLIS TOWER
CHICAGO, IL 60606-6357

EXAMINER

RAGHU, GANAPATHIRAM

ART UNIT

PAPER NUMBER

1652

DATE MAILED: 04/03/2025

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
18/435,340	02/07/2024	Brian Michael Green	32471/M202647G	4214

TITLE OF INVENTION: METHODS FOR MAKING L-GLUFOSINATE

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	\$1290	\$0.00	\$0.00	\$1290	07/03/2025

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. PROSECUTION ON THE MERITS IS CLOSED. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.

THE ISSUE FEE AND PUBLICATION FEE (IF REQUIRED) MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED. SEE 35 U.S.C. 151. THE ISSUE FEE DUE INDICATED ABOVE DOES NOT REFLECT A CREDIT FOR ANY PREVIOUSLY PAID ISSUE FEE IN THIS APPLICATION. IF AN ISSUE FEE HAS PREVIOUSLY BEEN PAID IN THIS APPLICATION (AS SHOWN ABOVE), THE RETURN OF PART B OF THIS FORM WILL BE CONSIDERED A REQUEST TO REAPPLY THE PREVIOUSLY PAID ISSUE FEE TOWARD THE ISSUE FEE NOW DUE.

HOW TO REPLY TO THIS NOTICE:

I. Review the ENTITY STATUS shown above. If the ENTITY STATUS is shown as SMALL or MICRO, verify whether entitlement to that entity status still applies.

If the ENTITY STATUS is the same as shown above, pay the TOTAL FEE(S) DUE shown above.

If the ENTITY STATUS is changed from that shown above, on PART B - FEE(S) TRANSMITTAL, complete section number 5 titled "Change in Entity Status (from status indicated above)".

For purposes of this notice, small entity fees are 40% the amount of undiscounted fees, and micro entity fees are 20% the amount of undiscounted fees.

II. PART B - FEE(S) TRANSMITTAL, or its equivalent, must be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed. If an equivalent of Part B is filed, a request to reapply a previously paid issue fee must be clearly made, and delays in processing may occur due to the difficulty in recognizing the paper as an equivalent of Part B.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Maintenance fees are due in utility patents issuing on applications filed on or after Dec. 12, 1980. It is patentee's responsibility to ensure timely payment of maintenance fees when due. More information is available at www.uspto.gov/PatentMaintenanceFees.

PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via the USPTO patent electronic filing system.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

By fax, send to: (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications. **Because electronic patent issuance may occur shortly after issue fee payment, any desired continuing application should preferably be filed prior to payment of this issue fee in order not to jeopardize copendency.**

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

4743 7590 04/03/2025
MARSHALL, GERSTEIN & BORUN LLP
233 SOUTH WACKER DRIVE
6300 WILLIS TOWER
CHICAGO, IL 60606-6357

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

Certificate of Mailing or Transmission

I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being transmitted to the USPTO via the USPTO patent electronic filing system or by facsimile to (571) 273-2885, on the date below.

(Typed or printed name)
(Signature)
(Date)

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
18/435,340	02/07/2024	Brian Michael Green	32471/M202647G	4214

TITLE OF INVENTION: METHODS FOR MAKING L-GLUFOSINATE

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	\$1290	\$0.00	\$0.00	\$1290	07/03/2025

EXAMINER	ART UNIT	CLASS-SUBCLASS
RAGHU, GANAPATHIRAM	1652	504-127000

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

☐ Change of correspondence address (or Change of Correspondence Address form PTO/AIA/122 or PTO/SB/122) attached.

☐ "Fee Address" indication (or "Fee Address" Indication form PTO/AIA/47 or PTO/SB/47; Rev 03-02 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list

(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,

(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1 _____

2 _____

3 _____

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE

(B) RESIDENCE: (CITY and STATE OR COUNTRY)

Please check the appropriate assignee category or categories (will not be printed on the patent): ☐ Individual ☐ Corporation or other private group entity ☐ Government

4a. Fees submitted: ☐ Issue Fee ☐ Publication Fee (if required)

4b. Method of Payment: (Please first reapply any previously paid fee shown above)

☐ Electronic Payment via the USPTO patent electronic filing system ☐ Enclosed check ☐ Non-electronic payment by credit card (Attach form PTO-2038)

☐ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. _____

5. **Change in Entity Status** (from status indicated above)

☐ Applicant certifying micro entity status. See 37 CFR 1.29

☐ Applicant asserting small entity status. See 37 CFR 1.27

☐ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.

NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.

NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature _____

Date _____

Typed or printed name _____

Registration No. _____



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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
18/435,340	02/07/2024	Brian Michael Green	32471/M202647G	4214
4743 7590 04/03/2025 MARSHALL, GERSTEIN & BORUN LLP 233 SOUTH WACKER DRIVE 6300 WILLIS TOWER CHICAGO, IL 60606-6357			EXAMINER RAGHU, GANAPATHIRAM	
			ART UNIT 1652	PAPER NUMBER
DATE MAILED: 04/03/2025				

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b) (Applications filed on or after May 29, 2000)

The Office has discontinued providing a Patent Term Adjustment (PTA) calculation with the Notice of Allowance.

Section 1(h)(2) of the AIA Technical Corrections Act amended 35 U.S.C. 154(b)(3)(B)(i) to eliminate the requirement that the Office provide a patent term adjustment determination with the notice of allowance. See Revisions to Patent Term Adjustment, 78 Fed. Reg. 19416, 19417 (Apr. 1, 2013). Therefore, the Office is no longer providing an initial patent term adjustment determination with the notice of allowance. The Office will continue to provide a patent term adjustment determination with the Issue Notification Letter that is mailed to applicant approximately three weeks prior to the issue date of the patent, and will include the patent term adjustment on the patent. Any request for reconsideration of the patent term adjustment determination (or reinstatement of patent term adjustment) should follow the process outlined in 37 CFR 1.705.

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at 1-(888)-786-0101 or (571)-272-4200.

OMB Clearance and PRA Burden Statement for PTOL-85 Part B

The Paperwork Reduction Act (PRA) of 1995 requires Federal agencies to obtain Office of Management and Budget approval before requesting most types of information from the public. When OMB approves an agency request to collect information from the public, OMB (i) provides a valid OMB Control Number and expiration date for the agency to display on the instrument that will be used to collect the information and (ii) requires the agency to inform the public about the OMB Control Number's legal significance in accordance with 5 CFR 1320.5(b).

The information collected by PTOL-85 Part B is required by 37 CFR 1.311. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 30 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, Virginia 22313-1450. **DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450.** Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. The United States Patent and Trademark Office (USPTO) collects the information in this record under authority of 35 U.S.C. 2. The USPTO's system of records is used to manage all applicant and owner information including name, citizenship, residence, post office address, and other information with respect to inventors and their legal representatives pertaining to the applicant's/owner's activities in connection with the invention for which a patent is sought or has been granted. The applicable Privacy Act System of Records Notice for the information collected in this form is COMMERCE/PAT-TM-7 Patent Application Files, available in the Federal Register at 78 FR 19243 (March 29, 2013).

<https://www.govinfo.gov/content/pkg/FR-2013-03-29/pdf/2013-07341.pdf>

Routine uses of the information in this record may include disclosure to:

- 1) law enforcement, in the event that the system of records indicates a violation or potential violation of law;
- 2) a federal, state, local, or international agency, in response to its request;
- 3) a contractor of the USPTO having need for the information in order to perform a contract;
- 4) the Department of Justice for determination of whether the Freedom of Information Act (FOIA) requires disclosure of the record;
- 5) a Member of Congress submitting a request involving an individual to whom the record pertains, when the individual has requested the Member's assistance with respect to the subject matter of the record;
- 6) a court, magistrate, or administrative tribunal, in the course of presenting evidence, including disclosures to opposing counsel in the course of settlement negotiations;
- 7) the Administrator, General Services Administration (GSA), or their designee, during an inspection of records conducted by GSA under authority of 44 U.S.C. 2904 and 2906, in accordance with the GSA regulations and any other relevant (i.e., GSA or Commerce) directive, where such disclosure shall not be used to make determinations about individuals;
- 8) another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c));
- 9) the Office of Personnel Management (OPM) for personnel research purposes; and
- 10) the Office of Management and Budget (OMB) for legislative coordination and clearance.

If you do not furnish the information requested on this form, the USPTO may not be able to process and/or examine your submission, which may result in termination of proceedings, abandonment of the application, and/or expiration of the patent.

Notice of Allowability	Application No. 18/435,340	Applicant(s) Green et al.	
	Examiner GANAPATHIRAMA RAGHU	Art Unit 1652	AIA (FITF) Status Yes

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☒ This communication is responsive to 03/10/2025.
☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2. ☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

3. ☒ The allowed claim(s) is/are 1-6 . As a result of the allowed claim(s), you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see http://www.uspto.gov/patents/init_events/pph/index.jsp or send an inquiry to PPHfeedback@uspto.gov.

4. ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

Certified copies:
a) ☐ All b) ☐ Some* c) ☐ None of the:

1. ☐ Certified copies of the priority documents have been received.
2. ☐ Certified copies of the priority documents have been received in Application No. _____ .
3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

* Certified copies not received: _____ .

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.
THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.

5. ☐ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
☐ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date _____ .

Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).

6. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

Attachment(s)

1. ☐ Notice of References Cited (PTO-892)	5. ☐ Examiner's Amendment/Comment
2. ☒ Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date <u>11/07/2024</u> .	6. ☒ Examiner's Statement of Reasons for Allowance
3. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material _____.	7. ☐ Other _____.
4. ☐ Interview Summary (PTO-413), Paper No./Mail Date. _____.	

/GANAPATHIRAMA RAGHU/ Primary Examiner, Art Unit 1652	03/19/2025
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Notice of Pre-AIA or AIA Status

The present application, filed on or after March 16, 2013, is being examined under the first inventor to file provisions of the AIA.

Application Status

Applicants' response, arguments, amendments and a Terminal disclaimer filed on 03/10/2025 have been fully considered and are deemed to be persuasive to overcome the rejections previously applied; in said amendment, applicants' have amended claim 1. Thus, amended claims 1-6 are pending in this application for examination. Rejections and/or objections not reiterated from previous office action are hereby withdrawn.

The Terminal Disclaimer filed on 03/10/2025 disclaiming the terminal portion of any patent granted on this application which would extend beyond the expiration date of allowed patent US 11,905,538 B2 has been reviewed and accepted. The Terminal Disclaimer has been recorded.

Information disclosure statement

The information disclosure statements (IDS) submitted on 11/07/2024 is in compliance with the provisions of 37 CFR 1.97. Accordingly, the IDS statement is considered and initialed by the examiner.

Withdrawn-Double Patenting rejection

Previous rejection of claims 1-6 of the instant application rejected under the judicially created doctrine of obviousness-type double patenting as being unpatentable over claims 1-15 of US 11,905,538 B2, is being withdrawn due to submission of a Terminal Disclaimer.

Claim Rejections: 35 USC § 102 (AIA)

Claims 1-6 rejected under 35 U.S.C. 102(a)(1) and 35 U.S.C. 102(a)(2) as being anticipated by Long DA., (US 8,445,406 B2, in IDS), is being withdrawn due to claim

amendments, persuasive arguments and an Appendix A showing unexpected results (for details, see applicants' arguments/remarks dated 03/10/2025; pages 4-5).

Withdrawn-Claim Rejections: 35 USC § 103 (AIA)

Previous rejection of claims 1-6 rejected under 35 U.S.C. 103(a) as being unpatentable over Long DA., (US 8,445, 406 B2, in IDS) as applied to claims 1-6 (see 35 U.S.C. 102(a)(1) and 35 U.S.C. 102(a)(2) rejection above) and in view of Bhoge et al., (US 10,888,095 B2), is being withdrawn due to claim amendments, persuasive arguments and an Appendix A showing unexpected results (for details, see applicants' arguments/remarks dated 03/10/2025; pages 4-5).

Allowable Subject Matter

Claims 1-6 are allowed.

Any comments considered necessary by applicant must be submitted no later than the payment of issue fee and, to avoid processing delays, should preferably accompany the issue fee. Such submissions should be clearly labeled "Comments on Statement of Reasons for Allowance".

Any inquiry concerning this communication or earlier communications from the examiner should be directed to GANAPATHIRAMA RAGHU whose telephone number is (571)272-4533. The examiner can normally be reached on M-F 8:30am-5pm EST. Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at <http://www.uspto.gov/interviewpractice>.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Robert Mondesi can be reached on 408-918-7584. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/GANAPATHIRAMA RAGHU/
Primary Examiner, Art Unit 1652



UNITED STATES
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ELECTRONIC ACKNOWLEDGEMENT RECEIPT

APPLICATION # 18/435,340	RECEIPT DATE / TIME 04/18/2025 01:49:33 PM Z ET	ATTORNEY DOCKET # 32471/M202647G
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Title of Invention

METHODS FOR MAKING L-GLUFOSINATE

Application Information

APPLICATION TYPE	Utility - Nonprovisional Application under 35 USC 111(a)	PATENT #	-
CONFIRMATION #	4214	FILED BY	Rebecca Walsh
PATENT CENTER #	70067795	FILING DATE	02/07/2024
CUSTOMER #	4743	FIRST NAMED INVENTOR	Brian Michael Green
CORRESPONDENCE ADDRESS	-	AUTHORIZED BY	Joshua Elliott

Documents

TOTAL DOCUMENTS: 1

DOCUMENT	PAGES	DESCRIPTION	SIZE (KB)
Fee Transmittal -- Part B PTOL-85.pdf	1	Issue Fee Payment (PTO-85B)	38 KB

Digest

DOCUMENT	MESSAGE DIGEST(SHA-512)
Fee Transmittal -- Part B PTOL- 85.pdf	246DF9A4934A8D9C2855FE48BE5E0E58BA7F31ED3EA905873 C2A1390B96811F0FE24246C030A79D231B3A3C96205B58177 CCD2FF30A286A42CDB150C62EC7C60

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

PART B-FEE(S) TRANSMITTAL

Complete and send this form, together with the applicable fee(s), by mail or fax, or via the USPTO patent electronic filing system.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

By fax, send to: (571) 273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications. **Because electronic patent issuance may occur shortly after issue fee payment, any desired continuing application should preferably be filed prior to payment of this issue fee in order not to jeopardize copendency.**

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

MARSHALL, GERSTEIN & BORUN LLP
233 S. Wacker Drive
6300 Willis Tower
Chicago, Illinois 60606-6357

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

Certificate of Mailing or Transmission

I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being transmitted to the USPTO via the USPTO patent electronic filing system or by facsimile to (571) 273-2885, on the date below.

Joshua T. Elliott	(Typed or printed name)
/Joshua T. Elliott/	(Signature)
April 18, 2025	(Date)

APPLICATION NO	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO	CONFIRMATION NO
18/435,340	02/07/2024	Brian Michael Green	32471/M202647G	4214

TITLE OF INVENTION: METHODS FOR MAKING L-GLUFOSINATE

APPLN: TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	\$1,290.00			\$1,290.00	07/03/2025

EXAMINER	ART UNIT	CLASS-SUBCLASS
G. Raghu	1652	

<p>1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363)</p> <p>☐ Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.</p> <p>☐ "Fee Address" indication (or "Fee Address" Indication form PTO/AIA/47 or PTO/SB/47; Rev 03-09 or more recent) attached. Use of a Customer Number is required.</p>	<p>2. For printing on the patent front page, list</p> <p>(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,</p> <p>(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.</p>	<p>1. MARSHALL, GERSTEIN & BORUN LLP</p> <p>2.</p> <p>3.</p>
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3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE: BASF SE

(B) RESIDENCE: (CITY and STATE or COUNTRY) Ludwigshafen, Germany

Please check the appropriate assignee category or categories (will not be printed on the patent): ☐ Individual ☒ Corporation or other private group entity ☐ Government

4a. Fees Submitted: ☒ Issue Fee ☐ Publication Fee (if required)

4b. Method of Payment (Please first reapply any previously paid fee shown above):

☒ Electronic Payment via the USPTO patent electronic filing system ☐ Enclosed check ☐ Non-electronic payment by credit card (Attach form PTO-2038)

☒ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. 13-2855

5. Change of Entity Status (from status indicated above)

☐ Applicant certifying micro entity status. See 37 CFR 1.29. NOTE: Absent a valid Certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.

☐ Applicant asserting small entity status. See 37 CFR 1.27. NOTE: If the application was previously under micro entity status, checking this box will be taken as a notification of loss of entitlement to micro entity status.

☐ Applicant changing to regular undiscounted fee status. NOTE: Checking this box will be taken as a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature	/Joshua T. Elliott/	Date	April 18, 2025
Typed or printed name	Joshua T. Elliott	Registration No.	43,603



UNITED STATES
PATENT AND TRADEMARK OFFICE

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Alexandria, VA 22313 - 1450
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ELECTRONIC PAYMENT RECEIPT

APPLICATION # 18/435,340	RECEIPT DATE / TIME 04/18/2025 01:49:33 PM Z ET	ATTORNEY DOCKET # 32471/M202647G
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Title of Invention

METHODS FOR MAKING L-GLUFOSINATE

Application Information

APPLICATION TYPE	Utility - Nonprovisional Application under 35 USC 111(a)	PATENT #	-
CONFIRMATION #	4214	FILED BY	Rebecca Walsh
PATENT CENTER #	70067795	AUTHORIZED BY	Joshua Elliott
CUSTOMER #	4743	FILING DATE	02/07/2024
CORRESPONDENCE ADDRESS	-	FIRST NAMED INVENTOR	Brian Michael Green

Payment Information

PAYMENT METHOD CARD / 8755	PAYMENT TRANSACTION ID E20254HD50135888	PAYMENT AUTHORIZED BY Rebecca Walsh
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FEE CODE	DESCRIPTION	ITEM PRICE(\$)	QUANTITY	ITEM TOTAL(\$)
1501	UTILITY ISSUE FEE	1290.00	1	1290.00
TOTAL AMOUNT:				\$1,290.00

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	ISSUE DATE	PATENT NO.	ATTORNEY DOCKET NO.	CONFIRMATION NO.
18/435,340	05/20/2025	12305206	32471/M202647G	4214

4743 7590 05/07/2025
MARSHALL, GERSTEIN & BORUN LLP
233 SOUTH WACKER DRIVE
6300 WILLIS TOWER
CHICAGO, IL 60606-6357

ISSUE NOTIFICATION

The projected patent number and issue date are specified above. The patent will issue electronically. The electronically issued patent is the official patent grant pursuant to 35 U.S.C. § 153. The patent may be accessed on or after the issue date through Patent Center at <https://patentcenter.uspto.gov/>. The patent will be available in both the public and the private sides of Patent Center. Further assistance in electronically accessing the patent, or about Patent Center, is available by calling the Patent Electronic Business Center at 1-888-217-9197.

The USPTO is implementing electronic patent issuance with a transition period, during which period the USPTO will mail a ceremonial paper copy of the electronic patent grant to the correspondence address of record. Additional copies of the patent (i.e., certified and presentation copies) may be ordered for a fee from the USPTO's Certified Copy Center at <https://certifiedcopycenter.uspto.gov/index.html>. The Certified Copy Center may be reached at (800)972-6382.

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)

(application filed on or after May 29, 2000)

The Patent Term Adjustment is 0 day(s). Any patent to issue from the above-identified application will include an indication of the adjustment on the front page.

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Center (<https://patentcenter.uspto.gov>).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Application Assistance Unit (AAU) of the Office of Patents Stakeholder Experience (OPSE), Stakeholder Support Division (SSD) at (571)-272-4200.

INVENTOR(s) (Please see PATENT CENTER site <https://patentcenter.uspto.gov> for additional inventors):

Brian Michael Green, Lutherville, MD;
Michelle Lorraine Gradley, Canterbury, UNITED KINGDOM;

APPLICANT(s) (Please see PATENT CENTER site <https://patentcenter.uspto.gov> for additional applicants):

BASF SE, Ludwigshafen, GERMANY;

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The USA offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to encourage and facilitate business investment. To learn more about why the USA is the best country in the world to develop technology, manufacture products, and grow your business, visit SelectUSA.gov.



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4743	7590	05/20/2025	EXAMINER	
MARSHALL, GERSTEIN & BORUN LLP			RAGHU, GANAPATHIRAM	
233 SOUTH WACKER DRIVE				
6300 WILLIS TOWER				
CHICAGO, IL 60606-6357			ART UNIT	PAPER NUMBER
			1652	
			NOTIFICATION DATE	DELIVERY MODE
			05/20/2025	ELECTRONIC

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

mgbdocket@marshallip.com

APPLICATION NO.	ISSUE DATE	PATENT NO.
18/435,340	20-May-2025	12305206

MARSHALL, GERSTEIN & BORUN LLP
233 SOUTH WACKER DRIVE
CHICAGO, IL 60606-6357

EGRANT NOTIFICATION

Your electronic patent grant (eGrant) is now available, which can be accessed via Patent Center at <https://patentcenter.uspto.gov>

The electronic patent grant is the official patent grant under 35 U.S.C. 153. For more information, please visit <https://www.uspto.gov/electronicgrants>