UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

SYMRISE AG and SYMRISE INC.,
Petitioners,

v.

INOLEX INVESTMENT CORP.,
Patent Owner.

IPR2023-01035
U.S. Patent No. 10,995,048

DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

I, Michael E. Wright, Ph.D, declare and state as follows:

1. I have been retained by Patent Owner, Inolex Investment Corp. ("Inolex" or "Patent Owner") to review and analyze certain facts concerning U.S. Patent No. 10,995,048 ("the '048 patent"), as well as the petition for *Inter Partes* Review filed by Symrise AG and Symrise Inc. ("Petitioners"), which alleges that claims 1-6, 8-10, and 22-28 of the '048 patent are invalid as obvious over certain prior art references.

2. The opinions and conclusions I express in this declaration are based on my education, my extensive experience in the fields of heterogeneous catalysis, homogeneous catalysis, organic and organometallic synthesis, and my review of materials related to this matter.

I. QUALIFICATIONS

3. I currently carry out consulting services and continue to do investigative research as founder and president of BioWright Technology.

4. I earned my undergraduate degree in 1979 from San Diego State University, and my doctorate in 1983 from University of Arizona under the direction of Dr. Gregory Nelson and Dr. Henry Hall, Jr. My thesis research concerned the synthesis of bimetallic organometallic complexes/catalysts as models for the Fischer Tropsch synthesis of fuels from carbon monoxide.

5. After earning my doctorate, I continued my education as a post-doctoral researcher at Colorado State University under the direction of Prof. John K. Stille. The research involved organic synthesis based on novel homogeneous palladium catalyzed chemical transformations.

6. I have more than 30 years of experience as a principal investigator in academic, governmental, and industrial labs. Over the course of my career, I have co-authored over 100 peer-reviewed publications and am named as an inventor or co-inventor on more than 40 patents.

7. Over the past 10 years, I have focused much of my work on developing biofuels biopolymers, novel catalysts, and creating energy-efficient and cost-effective ways to make these materials. More specifically, some of my work has focused on the synthesis of new bio-monomers based on bio-n-butanol for fully renewable and sustainable 1,3-butadiene that can be used in the production of "green" tires. I was also a co-author of the ASTM International task force report on Alcohol-to-Jet Synthetic Paraffinic Kerosene ("ATJ-SPK") Jet fuels, which is a process for the conversion of alcohols to a Sustainable Aviation Fuel ("SAF") blendstocks, with the specific aim to reduce the carbon footprint of airline operations worldwide.

8. A more detailed account of my work experience, professional services, publications, and other qualifications is listed in my *Curriculum Vitae*, which is attached here as **Appendix A**.

9. I have no financial interest in the outcome of this proceeding. I am not being compensated for my time spent working on this matter. In the past, I served as a consultant to Inolex, Inc. and assisted with developing the catalytic processes that are described and claimed in the ’048 patent.

10. I am named as a co-inventor on the ’048 patent. I believe that I am also a co-inventor of the subject matter of the substitute claims in this proceeding.

II. MATERIALS REVIEWED

11. In forming my opinions, I have reviewed, among other things, the ’048 patent (**Ex. 1001**), as well as the Declaration of Douglass F. Taber (**Ex. 1002**) and the materials referenced therein (**Ex. 1007**-**Ex. 1011**, **Ex. 1013**, **Ex. 1020**-**Ex. 1037**). I also reviewed the following materials:

Materials Reviewed	
Ex. 2002	J. Ross, “Heterogeneous Catalysis,” Elsevier B.V. (2012)
Ex. 2003	Kim *et al*., “Dual-bed catalytic system comprising Al2O3 and Ba/Al2O3 with enhanced 1-octene productivity in 1-octanol dehydration for linear α-olefin production,” *J. Ind. Eng. Chem. 119*:376-85 (2023)

Ex. 2004	Experimental data for Comparative Example 10 of U.S. Patent No. 10,995,048 for the conversion of 1-octanol to octenes
Ex. 2005	Deposition Testimony of Douglass F. Taber, January 23, 2024

III. TECHNICAL BACKGROUND

A. Dehydration of 1-Alcohols to Alkenes

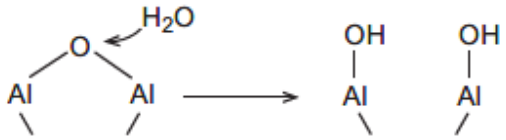
12. Dr. Taber cites an organic chemistry textbook, Wade, for background discussion on the *liquid phase*, acid-catalyzed dehydration of an alcohol to afford alkene(s). (*See* **Ex. 1002** at pp. 20-23, ¶ 35-¶ 40 (citing **Ex. 1030** at pp. 312-14).) As discussed there, the acid catalyst protonates the hydroxyl group, which upon leaving forms a solvent-stabilized carbocation that then loses a proton to form an alkene. (**Ex. 1030** at p. 313.) Because the carbocation intermediate can readily undergo rearrangement, the major alkene product is almost always the one with the most substituted double bond. (**Ex. 1030** at p. 314.) In addition, Brönsted acids (i.e., proton donors) are well known to isomerize alkenes to the more thermodynamically stable regioisomer. Such liquid phase catalytic dehydrations is referred to as "homogeneous" catalysis because the reactants and catalyst are in the same phase. (*See, e.g.,* **Ex. 2002** at pp. 2-4.)

13. The 1-alkenes are useful and valuable as feedstocks to prepare a wide range of chemical products and polymeric materials. Significant energy, time, and

equipment is required to separate and purify 1-alkenes, if even possible, from a mixture of alkene regioisomeric products. In general, commercial processes for making 1-alkenes seek to optimize alcohol conversion, alkene product regioselectivity, and chemical yield. (*See* **Ex. 1027** at p. 146.)

B. Catalytic Vapor Phase Dehydration of 1-Alcohols to 1-Alkenes

14. A wide range of solid catalysts can be used for alcohol dehydration to alkenes; however, γ-alumina is one of the most commonly used catalyst support for C_4 and higher alcohols, as it is inexpensive and can be prepared to have a high surface area. (*See* **Ex. 1027** at p. 145; *see also* **Ex. 2002** at pp. 71-75.) The alumina surface is acidic in a Brönsted sense owing to the presence of hydroxyl groups, some that form via hydrolysis of atmospheric water, as depicted in the following schematic.



(From **Ex. 2002**, p. 74.) Referring to the above schematic, in the precursor structure (left-hand side), the aluminum atoms can function as a Lewis acid (i.e., an electron acceptor), whereas the product structure (right-hand side) can function as a Brönsted acid (i.e., a proton donor). (*See* **Ex. 2002**, pp. 74-75.)

15. While acidic alumina catalysts can provide higher rates of conversion, this comes at the price of a reduction in 1-alkene regioselectivity and thus chemical yield due to double-bond migration, skeletal isomerizations occurring, and other deleterious chemical reactions. (*See* **Ex. 1027** at p. 145.) Thus, a basic alumina catalyst, free of strong Brönsted and Lewis Acid sites, is typically preferred in order to optimize 1-alkene regioselectivity, even if this comes at the price of a lower conversion efficiency. Net conversion can be improved, for example, by recycling the unreacted 1-alcohol; however, at a cost of increased energy use, more process time, and with an additional cost and complexity of processing equipment.

16. In contrast to homogeneous acid-catalyzed liquid phase dehydration, heterogeneous vapor phase dehydration proceeds via a surface interaction between the 1-alcohol and alumina, in which water is eliminated from the 1-alcohol without the formation of a solvent-stabilized intermediate. (*See* **Ex. 1027** at pp. 145-46.) Thus, the mechanistic pathway for homogeneous catalysts is for all practical purposes irrelevant when compared to heterogeneous catalytic alcohol dehydrations to an alkene. This points out a major and fundamental flaw in Dr. Taber's approach to understanding the significance of the '048 patent, which does not involve solution based, sophomore organic chemistry. (*See* **Ex. 1002** at ¶ 34-¶ 39 (discussing **Ex. 1030**, pp. 312-14).)

17. A 2005 publication from Sasol discusses the inherent trade-off between high-conversion efficiency to alkenes and the needed regioselectivity. (*See* **Ex. 1027**.) For example, 1-hexene having a purity greater than 90% can only be achieved at alcohol conversion up to about 80%. (**Ex. 1027** at p. 147 (Fig. 1).) At higher conversion of the alcohol the 1-hexene selectivity decreases dramatically concomitant with other alkene regioisomers being formed. (*See* **Ex. 1027** at p. 147 (Fig. 1).) Further, the purity of the alcohol feedstock largely controls the purity of the resulting alpha-olefin, regardless of catalyst. (*See* **Ex. 1027** at p. 149-150.)

18. The Sasol scientists also reported that regioselectivity can decrease dramatically as a function of the catalyst's time on stream ("TOS"), going from >96% 1-hexene regioselectivity for a new γ-alumina catalyst to less than 90% in a mere 250 hours of run time. (*See* **Ex. 1027** at p. 147 (Fig. 3).) Since most industrial processes are run continuously, this means that regioselectivity decreased to unacceptable levels after only about 10 days of use. (*See* **Ex. 1027** at p. 147 (a target minimum 1-alkene purity of 95%).) Upon investigating this phenomenon, the scientists concluded that the loss of regioselectivity with TOS was attributable to the presence of silicon impurities in the 1-hexanol feed, which deposited on the catalyst surface forming acidic sites that led to isomerization of the 1-alkene product. (*See* **Ex. 1027** at pp. 147-150.) Loss in heterogeneous catalyst

regioselectivity, in general, has been attributed to 1-alkene isomerization by strong Lewis and/or Brönsted acid sites. (*See* **Ex. 1027** at p. 146.) Notably, this deactivation does not happen with catalysts described in '048 patent for 1-octanol dehydration to 1-octene.

19. The literature frequently refers to catalysts that are "dried," which refers to the removal of water and other volatile components (that are typically liquids/solvents at ambient temperature and pressure) from a commercial catalyst. In contrast, "calcination" refers to heating to a sufficient temperature that chemical changes result to the catalyst itself. For example, aluminum hydroxides, oxo-hydroxides, and oxides can be converted to the more desired and catalytically active γ-alumina by heating (i.e., calcining) the material at temperatures of 450-600 °C. (*See* **Ex. 2002** at p. 73.) Going to extremely high temperatures can result in further conversion to other polymorphic forms of alumina, and eventually to α-alumina at temperatures above 1200 °C. (*See* **Ex. 2002** at p. 73.) Addition of a promotor followed by a calcination in the range of 400-600 °C affords a chemically modified γ-alumina catalyst. (*See* **Ex. 1004** at p. 44 of 617, ¶ [0076], Table 1; **Ex. 1005** at p. 24 of 236, ¶ [0076], Table 1.).

IV. U.S. PATENT NO. 10,995,048

20. I have reviewed the ’048 patent. In general, the ’048 patent is directed to methods of dehydrating bio-1-alcohols to provide corresponding bio-1-alkenes with high-regioselectivity and do so with a high alcohol conversion in a single pass through an isothermal fixed bed (or similar) chemical reactor. Original claim 1 of the ’048 patent was directed to a method to dehydrate a bio-1-alcohol having about 5 to about 20 carbon atoms, wherein the product had a bio-1-alkene regioselectivity of at least about 80%, while dependent claims 9 and 10 required a bio-1-alkene regioselectivity of “about 92% to about 99%” and “about 95% to about 99%,” respectively. (*See* **Ex. 1001** at p. 24, cols. 29-30.)

21. In preferred embodiments of the ’048 patent, the dehydration reaction is conducted using a γ-alumina catalyst that is first treated with a promotor and then calcined. (*See* **Ex. 1001** at pp. 22-23, col. 26, l. 10 – col. 27, l. 35 (Examples 1-5); *see also* **Ex. 1004** at pp. 58-60 of 617, ¶¶ [0121]-[0127]; **Ex. 1005** at pp. 38-40 of 236, ¶¶ [0121]-[0127].) The ’048 patent reports the use of a promotor-treated and calcined γ-alumina catalyst to dehydrate 1-hexanol and 1-octanol. In the case of 1-hexanol, a γ-alumina catalyst was first treated with a calcium acetate solution, followed by calcination in air at 440 °C for 12 hours. Dehydration reactions conducted at 330 °C and 350 °C provided conversion (to hexenes) of 96.3% and

96.9%, respectively, with a regioselectivity of 95.8% and 96.8% (as shown in Table 5, below).

TABLE 5

Catalyst	Reactor Temperature (° C.)	1-Hexene (%)	2-Hexenes & 3-Hexene (%)	1-Hexanol (%)	1-Hexene Selectivity (%)
EXAMPLE 11A	330	92.8	3.5	1.8	95.8
EXAMPLE 11B	350	93.8	3.1	1.7	96.8
EXAMPLE 11C	370	90.1	3.2	1.6	96.6

(**Ex. 1001**, col. 29, ll. 1-25 (Example 11); **Ex. 1004** at p. 63 of 617, ¶ [0133], **Ex. 1005** at p. 43 of 236, ¶ [0133].)

22. In the case of 1-octanol, γ-alumina was first treated with a calcium acetate (i.e., promotor) solution, followed by calcination in air at 440 °C or 500 °C for 12 hours. Dehydration conducted at 315 °C provided conversion (to octenes) of 97.2% and 95.5%, respectively, with a regioselectivity of 97.4% and 93.1% (per Table 2, below). Thus, the ’048 patent discloses that base-treatment and calcination were important to obtain the claimed conversion and bio-1-alkene regioselectivity. Further, while both catalysts far exceed any known catalyst performance, it is noteworthy that a higher calcination temperature led to a less selective catalyst. Thus, the ’048 patent teaches that higher calcination temperatures can be detrimental to catalyst performance.

TABLE 2

Catalyst	Calcination Temperature (° C.)	1-Octene (%)	2-Octene & 3-Octenes (%)	1-Octanol (%)	1-Octene Selectivity (%)
EXAMPLE 1	440	94.0	3.2	1.6	97.4
EXAMPLE 2	500	88.9	6.6	3.5	93.1

(**Ex. 1001**, col. 26, l. 10 – col. 27, l. 9 (Examples 1-3); **Ex. 1004** at pp. 58-59 of 617, ¶¶ [0121]-[0125]; **Ex. 1005** at pp. 38-39 of 236, ¶¶ [0121]-[0125].)

23. The catalyst prepared in '048 patent was also removed from the reactor and photographed. The catalyst showed only minor discoloration, and notably, after 1400 h (~58 days) of TOS, conversion efficiency and 1-octene regioselectivity remained steady. (*See* **Ex. 1001**, col. 29, ll. 26-35 (Example 12), Fig. 7; **Ex. 1004** at p. 63 of 617, ¶ [0135], Example 12, p. 80 of 617, Fig. 7; **Ex. 1005** at p. 40 of 236, ¶ [0135], Example 12, p. 54 of 236, Fig. 7.) The bio-1-octanol was not dried nor were there any precautions taken to remove silicon containing compounds from the 1-octanol feedstock. (*Compare* with **Ex. 1027** at pp. 149-50.) In my opinion, a person of ordinary skill in the art ("POSITA") would view the TOS achieved by the catalyst of the '048 patent as being entirely unexpected.

V. LEGAL STANDARDS

24. I am not an attorney, and counsel for the Patent Owner informed me of the following legal standards that are relevant to my opinions.

A. Obviousness

25. I understand that a claim is invalid as obvious "if the differences between the claimed invention and the prior art are such that the claimed invention as a whole would have been obvious before the effective filing date of the claimed invention to a POSITA to which the claimed invention pertains." 35 U.S.C. § 103. I understand that obviousness may be based on a combination of references, and that the combination of familiar elements according to known methods is likely obvious when it does no more than yield predictable results.

26. I also understand that an obviousness inquiry requires consideration of the following factors: (1) the scope and content of the prior art; (2) the differences between the claims and the prior art; (3) the level of ordinary skill in the pertinent art; and (4) any objective indicia of non-obviousness, such as unexpected results.

27. I understand that the obviousness analysis requires a comparison of the properly construed claim language to the prior art to determine whether the claimed subject matter as a whole would have been obvious.

28. It is my understanding that all prior art references are to be looked at from the viewpoint of a POSITA, and obviousness is analyzed from the perspective of one of skill in the art at the time the invention was made. Thus, the use of hindsight is not permitted.

29. I also understand that a claimed invention can be obvious where, if a single reference does not disclose each limitation of a claim, the missing limitations would have been obvious to a POSITA based on his/her background knowledge and skill at the relevant time. I also understand that obviousness based on a combination of references does not require an explicit suggestion in any of the references to combine them, if, as a matter of skill or practice in the field it would be known to do so.

30. I understand that any objective indicia of non-obviousness, such as unexpected results, must be commensurate with the scope of the claims and that there in order to have relevance to the question of obviousness, an unexpected result must be attributable to, or have a "nexus" with, the claimed invention.

B. The Field of the Invention and the Level of Ordinary Skill in the Art

31. The technical field relevant to the subject matter of the '048 patent relates to organic synthesis using heterogeneous catalysis.

32. I understand the PTAB has found that a POSITA at the time of the invention would have had "a bachelor's degree and two or more years of experience or a master's degree in organic synthesis or chemical engineering, with education substituting for experience and vice versa." **Paper 7** (Decision to Institute) at 6-7 (citing **Ex. 1002** at ¶ 16).

33. I note that Dr. Taber also testified that a POSITA would also "have experience in industrial chemistry or industrial processes" and "experience working at a chemical company." (**Ex. 2005** at p. 22, ll. 4-13.) In my opinion, experience with homogeneous catalytic dehydration reactions (i.e., in the liquid phase) does not necessarily translate to skill in the art of heterogeneous catalytic dehydration of alkenes, which are performed in the vapor phase over a catalyst.

34. I have experience working in both industry and academia, as well as for the U.S. Government. Thus, as of the earliest priority date of the substitute claims (i.e., January 2018 to January 2019), I was a person of at least ordinary skill in the art relevant to the '048 patent under either the Board's definition or Dr. Taber's revised definition. My opinions provided herein are made through the lens of a POSITA in the January 2018 to January 2019 time frame.

C. Claim Construction

35. I understand that in deciding to institute Petitioners' request for *Inter Partes* Review, the PTO found that "about 5" carbon atoms at the lower end of a range encompasses at least a carbon chain length of 4, and "about 20" carbon atoms at the upper end of a range encompasses at least a carbon chain length of 21. (**Paper 7**, at 8-10.) I understand that Patent Owner's substitute claims delete the term "about" in reference to the length a carbon chain.

36. In my opinion, a POSITA reviewing the substitute claims would, in view of the specification of the '048 patent, understand the words used in substitute claims 29-44 to have their plain and ordinary meaning.

VI. THE SCOPE AND CONTENT OF THE PRIOR ART

37. Petitioners cite three primary references: Le (**Ex. 1007**), DeBruyn (**Ex. 1008**), and Wright (**Ex. 1009**) in their Grounds 1-6 concerning the invalidity of cancelled claims 1-6, 8-10, and 22-28 of the '048 patent. The Petition (**Paper 1**) and Dr. Taber's Declaration (**Ex. 1002**) also cite several references as background information a POSITA would have been aware of.

A. Le (Ex. 1007) & De Bruyn (Ex. 1008)

38. U.S. Patent Pub. No. US 2011/0196114 ("*Le*," **Ex. 1007**) is titled "Polyolefin derived from renewable resources, and method for producing the

same," and was published on August 11, 2011.

39. U.S. Patent Pub. No. US 2005/0065389 ("*De Bruyn*", **Ex. 1008**) was published on March 24, 2005.

40. *Le* discloses the preparation of C_6 to C_9 olefins from their corresponding alcohols, which are obtained from a renewable source that is a fatty acid or fatty ester. (**Ex. 1007** at ¶ [0030]-¶ [0034].) According to *Le*, the catalyst for the dehydration can be either acidic or basic, with the former providing improved conversion and the latter providing improved regioselectivity. (*See* **Ex. 1007** at ¶ [0063]-¶ [0064].) *Le* discloses two commercially available γ-alumina catalysts suitable for performing the dehydration reaction: ESM 110® from the company Eurosupport, and Pural® KR1 from the company Sasol. (*See* **Ex. 1007** at ¶ [0064], ¶ [0066].) While *Le* discloses that these catalysts were γ-alumina, in my opinion a POSITA would not know how either of those catalysts was prepared—for example, whether either of those commercially available catalysts was chemically modified and/or calcined.

41. *De Bruyn* is a published patent application from Sasol Technology Ltd. that discloses methods to increase the chain length of an olefin by one carbon by hydroformylation of a C_4-C_7 α-olefin to form a linear C_5-C_8 aldehyde, hydrogenating said aldehyde to form an alcohol, then dehydrating the alcohol to

form a C_5-C_8 α-olefin, respectively. (*See* **Ex. 1008**, ¶ [0006]-¶ [0010].) In preferred embodiments, the resulting α-olefin is 1-hexene or 1-octene. (**Ex. 1008**, ¶ [0006].) *De Bruyn* states that "many different dehydration processes are known, and are accordingly not disclosed in any detail in this specification," and also discloses that "the catalyst may comprise a gamma-alumina catalyst or a promoted alumina catalyst e.g. $CaO \cdot Al_2O_3$, $Ca_2O_3 \cdot Al_2O_3$." (**Ex. 1008**, ¶ [0037].) However, *De Bruyn* provides no teaching or suggestion as to which catalyst is preferred, and whether such catalyst would be chemically modified or calcined.

42. *De Bruyn* discloses three experimental examples in which 1-hexanol (Example 3 and Example 4, Step 5) or 1-octanol (Example 5, Step 4) were dehydrated using a γ-alumina catalyst. Example 3 discloses the preparation of 1-hexene from 1-hexanol using a γ-alumina catalyst with a regioselectivity of 97.5%, and an alleged conversion efficiency of 94%. (*See* **Ex. 1008**, ¶ [0053].) Example 4, Step 5 discloses that 1-hexene was prepared from 1-hexanol using a Sasol Chemie Pural KR1 γ-alumina catalyst with regioselectivity of 96.5% at 76.5% conversion. (*See* **Ex. 1008**, ¶ [0067]-¶ [0068], Table 10.) It is noteworthy to mention that dihexyl ether is a significant by-product from alcohol conversion in this example. (*See* **Ex. 1008**, ¶ [0067]-¶ [0068], Table 10.) Example 5, Step 4 discloses that 1-octene was prepared from 1-octanol using a Sasol Chemie Pural

KR1 γ-alumina catalyst with regioselectivity of 97% to 98% at 59% to 73% conversion. (*See* **Ex. 1008**, ¶ [0080]-¶ [0083], Figure 2, Table 14.)

43. I note that *De Bruyn*'s Examples 4 and 5 provide a detailed summary of specific products, regioselectivity, and conversion, and in the case of Example 5, as a function of TOS. In contrast *De Bruyn*'s Example 3 provides no such data. In my opinion, a POSITA reading *De Bruyn* would view Examples 4 and 5, which are fully supported by Tables and/or Figures as a more reliable disclosure of catalyst performance. Also, *De Bruyn* is silent as to the specific catalyst that was used for Example 3, whereas a commercially available γ-alumina Pural KR1 catalyst was used for Examples 4 and 5. Therefore, a POSITA could more easily reproduce *De Bruyn*'s Examples 4 and 5 as a starting point for further investigation, but this could not necessarily be done for *De Bruyn*'s Example 3.

44. It is also important to note that, a POSITA reading *De Bruyn* would not know whether either catalyst used by *De Bruyn* was chemically modified or calcined. It should also be noted that the chemical yield of octenes from the 1-octanol was just over 20% after only 7 hours TOS for the catalyst—meaning that nearly 80% of the 1-octanol went to by-products or was left unreacted. (*See* **Ex. 1008**, ¶ [0080]-¶ [0083], Figure 2, Table 14.) What is more, De Bruyn also teaches

that the purity of the alcohol starting material largely controls the purity of the resulting alpha-olefin. (*See* **Ex. 1008**, ¶ [0033].)

45. In contrast to *De Bruyn*, *Le* does not disclose any details concerning the regioselectivity or conversion obtained in any exemplary embodiments (*see* **Ex. 1007** at ¶ [0171]-¶ [0172]), but instead only discloses that "when a saturated alcohol of which the only alcohol function is in the alpha-position is dehydrated, a single isomer (an alpha-olefin) is obtained." (**Ex. 1007** at ¶ [0068].) A POSITA would know that a single isomer is never obtained. In addition, I note that *Le* discloses that the same γ-alumina Pural KR1 catalyst utilized Example 4 and 5 of *De Bruyn* can be used in *Le*'s process. (**Ex. 1007**, ¶ [0066].) A summary of Le's and De Bruyn's dehydration conditions and results is provided in the following Table.

	***De Bruyn* (Example 3)**	***De Bruyn* (Example 4, Step 5)**	***De Bruyn* (Example 5, Step 4)**	***Le***
Alkene	1-hexene	1-hexene	1-octene	1-heptene
Catalyst	γ-alumina	γ-alumina Pural KR1	γ-alumina Pural KR1	γ-alumina ESM110®
Temp.	315 °C	290 °C	280 °C	345 °C
Flow Rate (HSV)[a]	5 h^{-1}	8 h^{-1}	10 h^{-1}	1 h^{-1}
1-alkene regioselectivity	97.5%	96.5%	97%-98%	Not reported
Conversion	94%[1]	76.5%	73.2%-59.4%	Not reported

a: Flow rate is reported as "Liquid Hourly Space Velocity", "Weight Hour Space Velocity", or "Mass Hour Space Velocity."

1: I note that data in **Ex. 1027** (by a common co-author) shows a 1-hexanol conversion of about 75% at a 1-hexene regioselectivity of about 95% (*see* **Ex. 1027** at p. 147, Fig. 2), which is commensurate with the data in *De Bruyn* Example 4, Step 5. Based on the lack of detail in Example 3 concerning both the catalyst and the products, in my opinion Example 3 of *De Bruyn* would not enable a POSITA to reproduce this result.

46. In my opinion, a POSITA would reasonably expect that the 1-alkene regioselectivity and alkene conversion obtained in *De Bruyn*'s Examples 4 and 5 would be obtained for the 1-heptanol dehydration disclosed in *Le*. It is important to note that *Le* utilizes the alkene products to prepare polyethylene-olefin copolymers using a Ziegler-Natta catalyst. A POSITA would have known at the time that Ziegler-Natta catalysts selectively utilize 1-alkenes, and that 2-alkenes (and other alkene impurities) would not co-polymerize nor interfere with polymerization of the 1-alkene. Thus, the regioselectivity of the dehydration reaction employed by *Le* would only affect the overall polymer yield, but the 2- and/or 3-alkene isomers could be removed following polymerization (rather than performing a costly purification by distillation). Second, *Le* makes no mention of the percent conversion achieved or the time on stream for which the dehydration reaction was run, which again would suggest to a POSITA that the yield and regioselectivity were not determined after the TOS reported in the presented Tables. Third, *Le* does not teach or suggest the use of promotors and/or calcination to improve conversion or 1-alkene regioselectivity.

47. In sum, a POSITA would have understood *Le* and *De Bruyn* as disclosing processes to dehydrate C_6 to C_8 1-alkanols using commercially available γ-alumina catalysts with a reasonable expectation of obtaining an alcohol

conversion efficiency in the range of 59% to 77%, a 1-alkene regioselectivity in the range of 95% to 98%, and chemical yield of 1-alkene at 20% to 35%. (*See, e.g.*, **Ex. 1008** at p. 11, Fig. 2 (describing percentage conversion, yield of octenes and purity versus TOS).) Importantly, the only discussion of catalyst activity as a function of TOS in *Le* and *De Bruyn* shows a catalyst that performed poorly with even modest amounts of TOS. (*See* **Ex. 1008**, ¶ [0081], Figure 2, Table 14.)

B. Wright (Ex. 1009)

48. U.S. Patent Pub. No. US 2012/0238788 ("*Wright*", **Ex. 1009**) was published on September 20, 2012. I am the same Michael E. Wright who is the sole named inventor on this published patent application, and *Wright* describes work that I did as a Department of Defense employee at the Naval Air Warfare Center Weapons Division ("NAWCWD") in China Lake, California, a U.S. NAVY base.

49. *Wright* discloses methods to make 1-butene from 1-butanol with high 1-butene regioselectivity (i.e., at least 92% (**Ex. 1009**, ¶ [0016]) and high conversion efficiency using a specific modified γ-alumina catalyst. Because the 1-butanol starting material was obtained from a biological source via a fermentation process, the 1-butanol may contain a significant amount of water, possibly in the range of 15% to 20% by weight. (*See* **Ex. 1009**, ¶ [0014] disclosing 15 wt-% water,

¶ [0034] disclosing 18 wt-% water.) Another goal was to develop a robust catalyst that could be used for 6-12 months TOS without diminished 1-butene regioselectivity, high butanol conversion, and high chemical yield. (*See* **Ex. 1009**, ¶ [0024], ¶ [0027].)

50. *Wright* discloses a γ-alumina catalyst that is first treated with an aqueous base solution, washed with water, dried, and then reacted with a chlorosilane, washed with a hydrocarbon solvent, and air-dried. (*See* **Ex. 1009**, ¶ [0020].) In its exemplary embodiment, *Wright* discloses a γ-alumina catalyst that was washed with an aqueous base, dried, and then reacted with a 2 wt-% solution of diphenyldichlorosilane in hexanes. The silanized catalyst was air dried and then 1-butanol containing 18 wt-% water was reacted at 380 °C at a flow rate of 0.8 mL/min at 30 psig. (*See* **Ex. 1009**, ¶ [0034].) The resulting bio-1-butene was produced with a conversion of 96% to 98% and a 1-butene regioselectivity of 94% to 95%, which remained steady for a period of 123 days TOS. (*See* **Ex. 1009**, ¶ [0034], Table 1.) Notably, *Wright* also discloses a yield for 1-butene of 96-98% (**Ex. 1009**, ¶ [0034], Table 1), whereas *Le* was silent as to 1-alkene yield and De Bruyn disclosed an octenes yield of only about 20-40% (**Ex. 1008**, Figure 2).

51. While experiments were only performed for the dehydration of 1-butanol to 1-butene, *Wright* discloses that the same catalyst can be used for the

dehydration of C_5 to C_{23} 1-alkanols. (*See* **Ex. 1009**, ¶ [0017], ¶ [0022].) In my opinion, a POSITA would view *Wright* as the closest prior art to the substitute claims because *Wright* discloses a catalyst that provides 1) high 1-alkene regioselectivity, 2) constant high alcohol conversion and yield, as well as selectivity for 1-alkenes, and 3) maintains exceptional catalytic activity and regioselectivity over a large TOS. Notably, all of the work disclosed in Wright involved silanated γ-alumina catalysts.

C. Other Prior Art References

52. Petitioners cite many other references that generically disclose the use of γ-alumina catalysts to dehydrate C_6 to C_8 1-alkanols. For example, early literature on the vapor phase catalytic dehydration of alcohols to form alkenes reported that alumina catalyst could selectively provide 1-alkenes. (*See, e.g.,* **Ex. 1028**, **Ex. 1029**.) Scientists at Shell Oil Company reported on the formation of 1-heptene from 1-heptanol using an activated alumina catalyst. Despite the limited analytical methods available at that time to fully characterize the reaction products, those authors reported yields of 87% 1-heptene, 3% 2-heptene, 2% unconverted heptanol, and 8% of a residue having a boiling point above 230 °C. (*See* **Ex. 1028** at p. 1938, col. 2.) Similarly, researchers at the Illinois Institute of Technology reported the use of dried alumina catalyst to dehydrate 1-hexanol and 1-octanol,

with the "main" products being the corresponding 1-olefins. (*See* **Ex. 1029** at p. 558, col. 2.) Collectively, none of the prior art presented taught how to prepare a catalyst that could maintain weeks of TOS and maintain a chemical yield of greater than 90% for the desired 1-alkene. To attain that kind of chemical yield requires 1) high conversion, 2) high alkene product selectivity, and then 3) high regioselectivity for the 1-alkene, which is disclosed by the '048 patent.

53. As summarized by Sasol scientists, other prior art details various efforts to improve the 1-alkene regioselectivity, which include: co-feeding of ammonia or amines during the reaction (*see* **Ex. 1026**), and the use of metal cations to modify the catalyst (by minimizing the number of strong Lewis and/or Brönsted acid sites that are thought to increase the rate of isomerization, **Ex. 1025**). (*See* **Ex. 1027**, p. 146, col. 1.) I note that the latter strategy is still being employed today in attempts to improve conversion of 1-octanol with high regioselectivity and chemical yield of 1-octene. (*See, e.g.,* **Ex. 2003**.) The Sasol catalysts showed decreased regioselectivity as a function of γ-alumina catalyst TOS, presumably due to silicon contaminants in the 1-hexanol feedstock forming acidic sites on the catalyst surface. (**Ex. 1027**, pp. 147-48.) Purification of the 1-alcohol feedstock to reduce silicon impurities to ~0.1 ppm provided 1-alkene regioselectivity greater than 95%, which was maintained for more than 900 hours time on stream. (*See* **Ex.**

1027, p. 149, Fig 6.) However, I note that this 1-octene regioselectivity was obtained at a low conversion of between 60% to 70% and a chemical yield of 1-octene of about 35%. (*See* **Ex. 1027**, p. 149, Fig. 6.) This low conversion would require separation and a time/energy consuming process to utilize the unconverted 1-octanol. It is also noteworthy to mention that the additional by-product dioctyl ether would have to be dealt with.

VII. SUBSTITUTE CLAIMS 29-44 ARE PATENTABLE OVER THE PRIOR ART

54. Independent substitute claim 29 requires the following:

> 29.A method for making a bio-alkene, comprising:
>
> dehydrating a bio-1-alcohol by heating the bio-1-alcohol in a reactor with a base-treated and calcined γ-alumina catalyst, wherein the catalyst has not been silanized, and
>
> wherein the resulting bio-alkene has a carbon chain of 6 to 14 carbon atoms and is produced in a conversion of at least 95%, and has a bio-1-alkene regioselectivity of 92% to about 99%.

(Patent Owner's, Non-Contingent Motion to Amend, Appendix A.) Notably, substitute claim 29 is a method for making a bio-alkene that requires a specific γ-alumina catalyst, which is both base-treated and calcined, but not silanized. The claimed catalyst is dehydrates C_6 to C_{14} bio-1-alcohols with both high bio-1-alkene regioselectivity of 92% to about 99%, and with a high conversion of at least 95%.

A. Substitute Claim 29 is Patentable Over *Le*, Optionally in View of *De Bruyn*

55. As discussed above, *Le* and *De Bruyn* disclose the dehydration of 1-alcohols using commercially available γ-alumina catalysts; however, *Le* and *De Bruyn* make no mention whether the commercial catalysts were chemically modified or calcined. As discussed above, a POSITA would read *De Bruyn* as disclosing a γ-alumina catalyst was used to dehydrate 1-hexanol and 1-octanol with a 1-alkene regioselectivity of 96% to 98% and at a conversion efficiency of 59% to 77%. (*See* **Ex. 1008**, ¶ [0053], ¶ [0067]-¶ [0068], Table 10, ¶ [0080]-¶ [0083], Figure 2, Table 14.) And, whereas *Le* does not specifically disclose a 1-alkene regioselectivity or conversion that was obtained, based on the catalysts disclosed in *Le* a POSITA would reasonably expect similar results to those disclosed in *De Bruyn*. In sum, a POSITA reviewing *Le* and *De Bruyn* would expect that the commercially available γ-alumina catalysts disclosed therein could be used to dehydrate a C_6 to C_{14} bio-1-alcohol with an alcohol conversion efficiency in the range of 59% to 77%, a 1-alkene regioselectivity in the range of 95% to 98%, and a chemical yield around 35% for the 1-alkene.

56. Based on *Le*'s and *De Bruyn*'s teaching to use commercially available γ-alumina catalyst, a POSITA reviewing *Le* and/or *De Bruyn* would have done just that. And a POSITA reviewing *Le* and/or *De Bruyn* would not have known

whether any of the alumina catalysts used were chemically modified and/or calcined. Thus, *Le* and/or *De Bruyn* in view of any background knowledge in the art would not have motivated a POSITA to utilize a base-treated and calcined (but not silanized) γ-alumina catalyst to dehydrate a C_6 to C_{14} bio-1-alcohol. Indeed, nothing in *Le* or *De Bruyn* would provide a POSITA any reasonable expectation that a base-treated and calcined γ-alumina catalyst could be used to dehydrate a C_6 to C_{14} bio-1-alcohol, wherein the resulting bio-alkene is produced in a conversion of at least 95%, and has a bio-1-alkene regioselectivity of 92% to about 99%. In sum, *Le*, or the combination of *Le* in view of *De Bruyn*, provide no reasonable expectation that the claimed catalyst could be used with any reasonable expectation of success to provide the claimed result. For at least these reasons, in my opinion substitute claim 29 is patentable over *Le*, and/or *Le* in view of *De Bruyn*.

57. I note that Petitioners cite *Cheng* (**Ex. 1010**) for its teaching that 1-alkenes can be used as starting materials to make other chemicals, and *Cheng* does not teach or suggest the use of any specific catalysts to prepare bio-1-alkenes with high alcohol conversion, high chemical yields, and bio-1-alkene regioselectivity. Thus, substitute claim 29 is similarly non-obvious over *Le* in view of *Cheng*, or *Le* in view of *De Bruyn* and *Cheng*.

B. Substitute Claim 29 is Patentable Over *Wright*

58. As discussed above, *Wright* discloses a γ-alumina catalyst that is first treated with an aqueous base solution, washed with water, dried, and then reacted with a chlorosilane, washed with a hydrocarbon solvent, and air-dried. (*See* **Ex. 1009**, ¶ [0020].) And, *Wright* discloses an exemplary embodiment in which a γ-alumina catalyst was base-treated, dried, and then silanized by reacting with a 2 wt-% solution of diphenyldichlorosilane. (*See* **Ex. 1009**, ¶ [0034].) After air-drying, the silanized catalyst dehydrated 1-butanol to provide butene with a alcohol conversion of 96% to 98%, a 1-butene regioselectivity of 94% to 95%, and a chemical yield of 1-butene of approximately 90%. (*See* **Ex. 1009**, ¶ [0034], Table 1.) Based on these superior results, in my opinion a POSITA would have viewed *Wright* as the closest prior art.

59. Based on *Wright*'s disclosure that C_5 to C_{23} 1-alkanols can be similarly dehydrated (Ex. 1009 at ¶ [0017]), in my opinion a POSITA would have reasonably expected that a base-treated and silanized γ-alumina catalyst could be used to dehydrate a C_6 to C_{14} bio-1-alcohol, wherein the resulting bio-alkene is produced in a conversion of at least 95%, and has a bio-1-alkene regioselectivity of 92% to about 99%. Experiments reported in the '048 patent as Example 10 were conducted in which 1-octanol was dehydrated using a γ-alumina catalyst that was

first treated with a base, dried, silanized, and then calcined at 400 °C. (*See* **Ex. 1001** at p. 23, col. 28, ll. 51-67; **Ex. 1004** at pp. 62-63 of 617; **Ex. 1005** at pp. 42-43 of 236.) The results of these experiments show that the claimed combination of 1-alkene regioselectivity and alcohol conversion to alkenes could not be achieved with a γ-alumina catalyst that was first treated with a base, dried, silanized, and then calcined at 400 °C. (*See* **Ex. 2004**.) Referring to **Ex. 2004**, a series of 16 experiments were conducted in which the catalyst remained the same, while the pump rate (mL/min) was varied. The data show that the base-treated, silanized, and calcined γ-alumina catalyst provided under certain conditions a bio-1-octene regioselectivity of 92% or higher, but with a conversion of at most 89%. (*See* **Ex. 2004**, Run 4.) Similarly, where the base-treated, silanized, and calcined γ-alumina catalyst provided an octene conversion of at least 95%, the bio-1-octene regioselectivity dropped between 61% and 85%. (*See* **Ex. 2004**, Runs 1-3.) In my opinion, a POSITA would view these results as unexpected, particularly given the high regioselectivity and conversion with which the base-treated and silanized γ-alumina catalyst of *Wright* dehydrated 1-butanol to 1-butene. Indeed, based on Wright's disclosure in view of the prior art of record, in my opinion a POSITA would have expected that high conversion and high 1-alkene regioselectivity would require a silanized γ-alumina catalyst.

60. As recited by substitute claim 29, the γ-alumina catalyst used to obtain the superior combination of high 1-alkene regioselectivity and conversion is "a base-treated and calcined γ-alumina catalyst, wherein the catalyst has not been silanized." In my opinion, a POSITA would not view *Wright* as teaching or suggesting both base-treatment and calcination. In my opinion, *Wright* fails to teach or suggest base-treatment of a γ-alumina catalyst followed by calcination, and *Wright* would not have provided a POSITA any reasonable expectation that the claimed catalyst would provide the requisite conversion,bio-1-alkene regioselectivity, and TOS stability. Further, a POSITA would be motivated to use a silanized catalyst described by *Wright*, whereas substitute claim 29 specifically requires that "the catalyst has not been silanized." Therefore, the specific γ-alumina catalyst required by substitute claim 29 would not have been obvious to a POSITA based on *Wright*'s disclosure.

61. Based on *Wright*'s teaching to use a base-treated and silanized γ-alumina catalyst, *Wright* in view of any background knowledge in the art would not have motivated a POSITA to utilize a base-treated and calcined (but not silanized) γ-alumina catalyst to dehydrate a C_6 to C_{14} bio-1-alcohol. What is more, a POSITA would not have had any reasonable expectation that using the claimed base-treated and calcined (but not silanized) γ-alumina catalyst would provide the

claimed combination of high alkene conversion, high bio-1-alkene regioselectivity, and chemical yield of bio-1-alkene. Finally, a POSITA would have viewed the claimed results as unexpected over the results obtained using Wright's catalyst to dehydrate bio-1-octanol. In sum, *Wright* provides no motivation to use the claimed catalyst with any reasonable expectation of success, and the claimed catalyst provides unexpected results compared to *Wright*, which is the closest prior art. For these reasons, in my opinion substitute claim 29 is non-obvious and patentable over *Wright*.

62. I note that Petitioners cite *Cheng* (**Ex. 1010**) for its teaching that 1-alkenes can be used as starting materials to make other chemicals, and *Cheng* does not teach or suggest the use of any specific catalysts to prepare bio-1-alkenes with high alcohol conversion, bio-1-alkene regioselectivity, and high chemical yield of the bio-1-alkene. Thus, substitute claim 29 is similarly non-obvious over *Wright* in view of *Cheng*.

C. Substitute Claims 30-44 Are Patentable Over the Prior Art

63. Substitute claims 30-44 depend directly or indirectly from independent substitute claim 29, and incorporate all of its limitations. Thus, for the same reasons discussed above with respect to substitute claim 29, dependent claims 30-44 are also non-obvious and patentable over the prior art.

D. Substitute Claims 34, 37, 40, and 43 Are Additionally Patentable Over the Prior Art of Record

64. Further, substitute claims 34, 37, 40, and 43 depend indirectly from claim 29 and require that "prior to the dehydrating, the catalyst has been calcined at a temperature of about 400 °C to about 480 °C." (Patent Owner's, Non-Contingent Motion to Amend at Appendix A.) As discussed above, a POSITA would not know based on the disclosures of *Le* and *De Bruyn* whether the commercial γ-alumina catalysts disclosed therein were chemically modified and/or calcined. are wholly silent as to whether calcination of a γ-alumina catalyst (let alone a base-treated γ-alumina catalyst) can be used to improve 1-alkene regioselectivity and TOS stability. Further, *Wright* does not disclose or suggest base-treatment of a γ-alumina catalyst followed by calcination.

65. As set forth in the '048 patent specification, base-treatment followed by calcination at a temperature of about 400 °C to about 480 °C, provides a bio-1-alkene regioselectivity of 97%, alcohol conversion of 97%, and a chemical yield of 94%; whereas, calcination at a temperature of 500 °C reduced bio-1-alkene regioselectivity to 93%, alcohol conversion to 95.5%, and the chemical yield of 1-octene drops to 88%. (*See* **Ex. 1004** at p. 44 of 617, ¶ [0076], Table 1; **Ex. 1005** at p. 24 of 236, ¶ [0076], Table 1.) In my opinion, a POSITA would find the fact that

a calcining ceiling temperature afforded an optimum catalyst to be a truly unexpected result.

66. In my opinion, nothing in *Le*, *De Bruyn*, *Wright* or the background knowledge in the art teaches or suggests that base treatment and calcination at a temperature of about 400 °C to about 480 °C can improve 1-alkene regioselectivity, alcohol conversion, and chemical yield of the target 1-alkene. For this additional reason, claims 34, 37, 40, and 43 are non-obvious over the prior art of record in this proceeding, including *Le*, *De Bruyn*, *Wright*, or any combination thereof.

VIII. CONCLUSION

67. In view of the foregoing, I conclude that substitute claims 29-44 of the ’048 patent would not have been obvious to a POSITA in view of the prior art cited in Petitioners’ petition for *Inter Partes* Review. Accordingly, for the reasons discussed herein it is my opinion that substitute claims 29-44 of the ’048 patent are patentable over the prior art.

I declare under penalty of perjury that, to the extent of my knowledge and belief, the foregoing is true and correct. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with

the understanding that knowing and willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of title 18 of the United States Code.

Date: Feb 7, 2024

Michael E. Wright, Ph.D.

Appendix A

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Professional Preparation

1983-1984	Post-Doctoral Associate	Colorado State Univ., Ft. Collins
1983	Ph.D. (Organic Chemistry)	University of Arizona, Tucson
1979	B.S. (Chemistry)	San Diego State Univ., California

Appointments/Positions

2015-present	**Founder/President**, BioWright Technology, Hurricane, UT
2014-2015	**Chief Chemist**, WHITE DOG LABS, New Castle, DE
2013-2014	**Director of Catalysis**, Cobalt Technologies, Mountain View, CA
2010-2013	**DP5/GS15 Senior Scientist**, US NAVY, China Lake, CA
2008-2010	**Senior Scientist** (Level VII) ATK Launch Systems, UT
2001-2008	**DP4 Senior Scientist**, US NAVY, China Lake, CA
1998-2002	**Professor of Chemistry** (w Tenure), VA Commonwealth University
1985-1998	**Professor of Chemistry** (w Tenure) Utah State University

100+ peer reviewed publications, selected for relevance to the production of SAF from 1-Butanol

Wright, M.E.; Harvey, B.G.; Quintana, R.A. "Highly Efficient Zirconium Catalyzed Batch-Conversion of 1-Butene: A New Route to Jet Fuels" *Energy & Fuels*, **2008**, *22* (5), 3299–3302

Wright, M.E.; Harvey, B.G.; Quintana, R.A. "High-Density Renewable Fuels Based on the Selective Dimerization of Pinenes" *Energy & Fuels*, **2010**, *24* (1), 267–273.

40+ patents, selected for relevance to the production of SAF from 1-Butanol

US 8,912,373 PROCESS FOR THE DEHYDRATION OF AQUEOUS BIO-DERIVED TERMINAL ALCOHOLS TO TERMINAL ALKENES

US 8,242,319 SELECTIVE ISOMERIZATION AND OLIGOMERIZATION OF OLEFIN FEEDSTOCKS FOR THE PRODUCTION OF TURBINE AND DIESEL FUEL

While in academics Dr. Wright received a prestigious Alexander von Humboldt fellowship for a sabbatical leave in 1993 at the Universität de Regensburg for his published work in catalytic, organic, and polymer chemistry. While at the China Lake

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Navair Research Department, Dr. Wright received numerous internal awards for his work on biofuels and in 2011 was awarded the SEC NAV Dr. Dolores M. Etter award for Top Scientists and Engineers for his leadership and accomplishments in creating jet fuel from bio-1-butanol. During his tenure at Cobalt technologies his work on 1-butanol dehydration chemistry led to improvements and as a result, a cost effective commercial catalytic process to bio-1-butene from 1-butanol (100,000 MT/yr). This latter result cleared an unobstructed pathway to commercial production of SAF from 1-butanol. Dr. Wright coauthored the original ASTM task force report leading to the approval of D7566 ANNEX 5, ATJ-SPK turbine fuel approval in 2017. Dr. Wright has consulted for several small and large paint/coatings and chemical production companies over the past 8 years. In addition, Dr. Wright wrote/participated in a NSF SBIR phase I grant aimed at replacing toxic chromate primers with novel and commercially viable “green” coating technology (based on a Wright/Navy patent).