Symrise AG & Symrise Inc. v. Inolex Investment Corp.

Patent Owner Inolex Investment Corp.'s Demonstratives

IPR2023-01035

U.S. Patent No. 10,995,048

Oral Hearing

September 19, 2024, 1:00 PM

Inolex's Expert: Dr. Michael Wright

- Doctorate in organic chemistry, with thesis research on the synthesis of bimetallic organometallic complexes/catalysts
- 30+ years' experience working in academic, governmental, and industrial laboratories
  - Professor of Chemistry, Utah State and VCU (1985-2002)
  - Senior Scientist, US Navy (2001-08 & 1010-13)
  - Director of Catalysis, Cobalt Technologies (2013-14)
- 100+ peer-reviewed publications
- Named inventor on 40+ patents, including the patent at issue
- No financial interest in the outcome of this proceeding

Ex. 2001, ¶¶3-10, Appendix A.

U.S. Patent No. 10,995,048

U.S. Patent No. 10,995,048 – Ex. 1001



Ex. 1001 – The '048 Patent

United States Patent
Burgo et al.

(10) **Patent No.: US 10,995,048 B2**
(45) **Date of Patent: May 4, 2021**

NATURAL 1,2-ALKANEDIOLS, COMPOSITIONS HAVING NATURAL 1,2-ALKANEDIOLS AND PROCESSES FOR MAKING THE SAME

(71) Applicant: **Inolex Investment Corporation**, Wilmington, DE (US)

(72) Inventors: **Rocco V. Burgo**, Mullica Hill, NJ (US); **Michael E. Wright**, Hurricane, UT (US); **Gary B. Mosser**, Tabernacle, NJ (US); **Michael J. Fevola**, Belle Mead, NJ (US)

(73) Assignee: **INOLEX INVESTMENT CORPORATION**, Wilmington, DE (US)

(21) Appl. No.: **16/682,059**

(22) Filed: **Nov. 13, 2019**

(65) **Prior Publication Data**

US 2020/0189995 A1 Jun. 18, 2020

Related U.S. Application Data

(63) Continuation of application No. 16/262,902, filed on Jan. 30, 2019.

(60) Provisional application No. 62/623,985, filed on Jan. 30, 2018.

U.S. Patent No. 10,995,048 – Ex. 1001



United States Patent
Burge et al.

(10) Patent No.: US 10,995,048 B2
(45) Date of Patent: May 4, 2021

(54) NATURAL 1,2-ALKANEDIOLS, COMPOSITIONS HAVING NATURAL 1,2-ALKANEDIOLS AND PROCESSES FOR MAKING THE SAME

(71) Applicant: Ingles Investment Corporation, Wilmington, DE (US)

(72) Inventors: Bruce V. Burge, Mullica Hill, NJ (US); Michael E. Wright, Hurricane, UT (US); Gary B. Mower, Tabernacle, NJ (US); Michael J. Ferola, Delle Mead, NJ (US)

(73) Assignee: INOLEX INVESTMENT CORPORATION, Wilmington, DE (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 16/682,699

(22) Filed: Nov. 13, 2019

(65) Prior Publication Data

US 2020/0190395 A1 Jun. 18, 2020

Related U.S. Application Data

(63) Continuation of application No. 16/262,902, filed on Jan. 30, 2019.

(60) Provisional application No. 62/623,995, filed on Jan. 30, 2018.

(51) Int. Cl.
C07C 29/04 (2006.01)
C07C 33/20 (2006.01)
C07C 29/60 (2006.01)
B01J 35/06 (2006.01)
B01J 37/08 (2006.01)
B01J 21/04 (2006.01)
C12D 3/20 (2006.01)
C07C 1/24 (2006.01)
C07C 29/48 (2006.01)
C07C 41/02 (2006.01)
C07C 41/09 (2006.01)
C07B 41/02 (2006.01)
C07B 43/00 (2006.01)
A61L 2/18 (2006.01)
A61P 31/02 (2006.01)
A61K 8/34 (2006.01)
A61Q 5/00 (2006.01)
A61Q 19/00 (2006.01)
A61Q 19/08 (2006.01)
C12D 3/00 (2006.01)

(52) U.S. Cl.
CPC C07C 29/04 (2013.01); B01J 21/04 (2013.01); B01J 35/06 (2013.01); B01J 37/08 (2013.01); C07C 1/24 (2013.01); C07C 41/02 (2013.01); C07C 41/09 (2013.01); C07C 43/00 (2013.01); C07C 29/48 (2013.01); C07C 29/60 (2013.01); C07C 33/20 (2013.01); C12D 3/20 (2013.01)

(58) Field of Classification Search
None
See application file for complete search history.

(56) References Cited

U.S. PATENT DOCUMENTS

FOREIGN PATENT DOCUMENTS

OTHER PUBLICATIONS

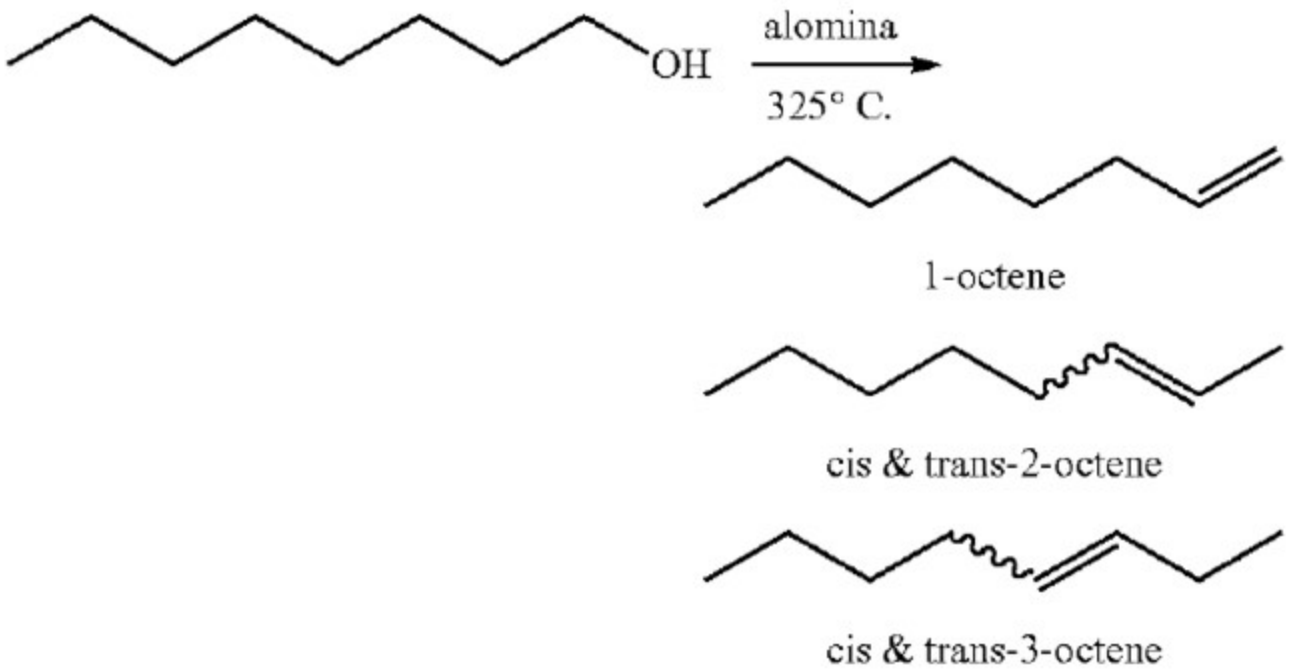
(Continued)

Primary Examiner — Natasha Hellman
(74) Attorney, Agent, or Firm — Cozen O'Connor

(57) ABSTRACT

Ex. 1001 – The '048 Patent

In one known path of forming caprylyl glycol (1,2-octanediol) starting with 1-octanol, made from petrochemically-derived (i.e., petroleum-derived or fossil fuel-derived) feedstocks, the following reaction is known to occur:



Ex. 1001, Col. 5:12-30.

Makgoba – Ex. 1027



Silication of γ-alumina catalyst during the dehydration of linear primary alcohols

N.P. Makgoba*, T.M. Sakaneka, J.G. Koortzen, C. van Schalkwyk, J.M. Botha, C.P. Nicolaides*

Symrise AG, EX1027
Symrise AG v. Isodex Inc.
IPR2023-01035

Ex. 1027 – *Makgoba*

2006 Publication from SASOL

The dehydration of alcohols with four or more carbons over solid acid catalysts is expected to be accompanied by the double-bond shift reaction of the alkene product. This is because the two reactions occur readily and at comparable rates [4]. In a chemical process for the dehydration of primary alcohols to produce 1-alkenes, the isomerization of the 1-alkene product to the internal alkenes presents many disadvantages. Firstly, the isomers have boiling points very close to that of the 1-alkene, and are therefore difficult and expensive to separate. Secondly, the isomerization reaction results in a loss of the useful 1-alkene products and therefore affects the efficiency of the process, since the isomers may not be suitable for the same end-use as the 1-alkene. This can have considerable cost implications for the process. As can be anticipated, therefore, it is desirable to have a high selectivity to the 1-alkene.

Ex. 1027, 146 (Col. 1)

U.S. Patent No. 10,995,048 – Ex. 1001



Ex. 1001 – The ’048 Patent

The reactor can be designed as a simple heated reaction tube packed with catalyst and a flow of the gaseous bio-1-alcohol over the catalyst, preferably in a single pass affording greater than about 95% conversion, with greater than about 98% a preferred embodiment, and in all cases with no recycle stream used whatsoever in the overall process flow.

Ex. 1001, Col. 15:27-32;
Ex. 2006, p. 13, ¶20; Ex. 2010, p. 3, ¶6.

Substitute Claim 29



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

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.

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

Title: NATURAL 1,2-ALKANEDIOLS, COMPOSITIONS HAVING NATURAL 1,2-ALKANEDIOLS AND PROCESSES FOR MAKING THE SAME

PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND AND REQUEST FOR PRELIMINARY GUIDANCE

(Paper 18, 6-8, Appendix A at A-1.)

APPENDIX A

* * *

29. (Proposed Substitute for Claim 1) A method for making a bio-alkene, comprising:

dehydrating a bio-1-alcohol by heating the bio-1-alcohol in a continuous flow reactor at a temperature of 240 °C to 360 °C with a calcium-promoted γ-alumina catalyst calcined at a temperature of about 420 °C to about 480 °C, wherein the catalyst has not been silanized, and

wherein the resulting bio-alkene has a carbon chain of 6 to 10 ~~about 5 to about 20~~ carbon atoms and is produced in a conversion of at least 95% % and ~~has~~ a bio-1-alkene regioselectivity of 95% to about 99% from a single-pass reaction after at least 20 hours time-on-stream~~at least about 80%~~.

Paper 18 at Appendix A-1, *see also* pp. 6-8.

The Person of Ordinary Skill in the Art

The Level of Ordinary Skill in the Art

- A POSITA 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 1 at 15 (citing Ex. 1002 ¶16); Paper 7 at 6-7.

Claim Construction

"calcined"



Trials@uspto.gov
571-272-7822

Paper 16
Date: May 29, 2024

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

SYMRISE AG AND SYMRISE INC.,
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Patent Owner.

IPR2023-01035
Patent 10,995,048 B2

Before ULRIKE W. JENKS, TINA E. HULSE, and ROBERT A. POLLOCK, *Administrative Patent Judges*.

HULSE, *Administrative Patent Judge*.

PRELIMINARY GUIDANCE
PATENT OWNER'S MOTION TO AMEND

Paper 16 – Preliminary Guidance

No construction necessary for the term *"calcined."*

"calcined"

Petitioner argues that Dr. Wright stated "'calcined' is 'heating to a sufficient temperature that chemical changes result into the catalyst itself." Opp. 4 (citing Ex. 2001 ¶ 19). Petitioner asserts that "[c]alcined should be interpreted to be sufficient heating to complete the chemical change to the catalyst." *Id.* (citing Ex. 1038 ¶ 20). Patent Owner does not propose a construction for this term. *See generally* Mot.

"[C]laim terms need only be construed 'to the extent necessary to resolve the controversy.'" *Vivid Techs., Inc. v. Am. Sci. & Eng'g, Inc.*, 200 F.3d 795, 803 (Fed. Cir. 1999). We decline to construe "calcined" at this stage because, in light of the teachings of the asserted references, such a construction appears to be unnecessary to resolve the patentability arguments discussed below.

Paper 16 at 8.

"catalyst has not been silanized"



Trials@uspto.gov
571-272-7822

Paper 16
Date: May 29, 2024

UNITED STATES PATENT AND TRADEMARK OFFICE

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Patent 10,995,048 B2

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HULSE, *Administrative Patent Judge*.

PRELIMINARY GUIDANCE
PATENT OWNER'S MOTION TO AMEND

Paper 16 – Preliminary Guidance

"catalyst has not been silanized" means that
"no affirmative step is taken to silanize the catalyst."

"catalyst has not been silanized"

Petitioner contends Dr. Wright "testified that 'silanized' requires an affirmative step during the catalyst preparation" and therefore "silanization requires an affirmative preparation step." Opp. 4 (citing Ex. 1040, 23:14–24:2; *In re Kollar*, 286 F.3d 1326, 1332 (Fed. Cir.2002)). Patent Owner does not propose a construction for this phrase. *See generally* Mot.

On this record, we agree that "catalyst has not been silanized" means that "no affirmative step is taken to silanize the catalyst." Opp. 4.

Paper 16 at 8.

"a calcium-promoted γ-alumina catalyst"


United States Patent
Burgo et al.
Patent No.: US 10,995,048 B2
Date of Patent: May 4, 2021

Ex. 1001 – The '048 Patent

Plain and ordinary meaning:

"a γ-alumina catalyst that has been modified by reacting with a Ca^{+2} compound" such that the promoted γ-alumina catalyst *"contains sufficient calcium as Ca^{+2} in order to enhance or 'promote' the reactivity of the γ-alumina catalyst."*

Ex. 1001 at ¶34; Paper 18 at 25; Ex. 2006, p. 20, ¶34.

"from a single pass reaction"



(12) **United States Patent**
Burgo et al.

(10) **Patent No.: US 10,995,048 B2**
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(54) **NATURAL 1,2-ALKANEDIOLS, COMPOSITIONS HAVING NATURAL 1,2-ALKANEDIOLS AND PROCESSES FOR MAKING THE SAME**

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(73) Assignee: **INDEX INVESTMENT CORPORATION**, Wilmington, DE (US)

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(21) Appl. No.: **16/682,699**

(22) Filed: **Nov. 13, 2019**

(65) **Prior Publication Data**

US 2020/0339995 A1 Oct. 29, 2020

Related U.S. Application Data

(63) Continuation of application No. 16/262,902, filed on Jan. 30, 2019.

(60) Provisional application No. 62/623,995, filed on Jan. 30, 2018.

(51) **Int. Cl.**
C07C 29/04 (2006.01)
C07C 33/20 (2006.01)
C07C 29/60 (2006.01)
B01J 35/06 (2006.01)
B01J 37/08 (2006.01)
B01J 21/04 (2006.01)
C12P 3/26 (2006.01)
C07C 29/48 (2006.01)
C07C 33/02 (2006.01)
C07C 33/20 (2006.01)
C07B 63/00 (2006.01)
A61L 2/18 (2006.01)
A61K 8/34 (2006.01)
A61Q 5/00 (2006.01)
A61Q 19/00 (2006.01)
C12P 3/00 (2006.01)

(52) **U.S. Cl.**
CPC **C07C 29/04** (2013.01); **B01J 21/04** (2013.01); **B01J 35/06** (2013.01); **B01J 37/08** (2013.01); **C07C 33/20** (2013.01); **C07C 33/02** (2013.01); **C07C 33/20** (2013.01); **C07C 33/48** (2013.01); **C07C 29/60** (2013.01); **C07C 29/48** (2013.01); **C07C**

(58) **Field of Classification Search**
None
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

FOREIGN PATENT DOCUMENTS

OTHER PUBLICATIONS

(57) **ABSTRACT**

Symrise AG, EX1001
Symrise AG v. Index Inc.
IPR2021-01035

Ex. 1001 – The '048 Patent

Plain and ordinary meaning:

"a single pass of the bio-1-alcohol through the specified continuous flow reactor" (at a temperature of 240°C to 360°C with a calcium-promoted γ-alumina catalyst calcined at a temperature of about 420°C to about 480°C, wherein the catalyst has not been silanized)

- Excludes any affirmative steps of isolating unreacted bio-1-alcohol, ethers (and possible contaminants) from the effluent stream of the continuous flow reactor and recycling it in a second pass through the reactor in order to attempt an apparent improvement of the overall conversion of bio-1-alcohol to bio-1-alkene.

Ex. 1001 at ¶35; Paper 18 at 25; Ex. 2006, pp. 20-21, ¶35.

"about" as it refers to *"temperature"*



(12) **United States Patent**
Burgo et al.

(10) **Patent No.: US 10,995,048 B2**
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(54) **NATURAL 1,2-ALKANEDIOLS, COMPOSITIONS HAVING NATURAL 1,2-ALKANEDIOLS AND PROCESSES FOR MAKING THE SAME**

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(73) Assignee: **INOLEX INVESTMENT CORPORATION**, Wilmington, DE (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **16/682,699**

(22) Filed: **Nov. 13, 2019**

(65) **Prior Publication Data**

US 2020/0339595 A1 Oct. 29, 2020

Related U.S. Application Data

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(60) Provisional application No. 62/623,995, filed on Jan. 30, 2018.

(51) **Int. Cl.**
C07C 29/04 (2006.01)
C07C 33/20 (2006.01)
C07C 29/60 (2006.01)
B01J 19/06 (2006.01)
B01J 17/00 (2006.01)
B01J 21/04 (2006.01)
C12D 1/20 (2006.01)
C07C 1/24 (2006.01)
C07C 29/48 (2006.01)
C07C 41/02 (2006.01)
C07C 41/16 (2006.01)
C07B 41/02 (2006.01)
C07B 43/00 (2006.01)
A61L 2/18 (2006.01)
A61K 8/34 (2006.01)
A61K 8/36 (2006.01)
B01J 3/00 (2006.01)
B01J 3/02 (2006.01)
B01J 19/00 (2006.01)
C12D 3/00 (2006.01)

(52) **U.S. Cl.**
CPC **C07C 29/04** (2013.01); **B01J 3/02** (2013.01); **B01J 19/06** (2013.01); **B01J 17/00** (2013.01); **C07C 1/24** (2013.01); **C07C 41/02** (2013.01); **C07C 41/16** (2013.01); **C07C 43/00** (2013.01); **C07C 29/48** (2013.01); **C07C 29/60** (2013.01); **C07C 33/20** (2013.01); **A61K 8/34** (2013.01); **A61K 8/36** (2013.01); **A61L 2/18** (2013.01); **B01J 19/00** (2013.01); **B01J 21/04** (2013.01); **C12D 1/20** (2013.01); **C12D 3/00** (2013.01)

(58) **Field of Classification Search**
None
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

FOREIGN PATENT DOCUMENTS

OTHER PUBLICATIONS

(57) **ABSTRACT**

Symrise AG, EX1001
Symrise AG v. Inolex Inc.
IPR2021-01035

Ex. 1001 – The '048 Patent

The plain and ordinary meaning of *"about"* is *"approximate"*

- In the context of the technology, an approximate *"temperature"* for example, does not refer to a POSITA's ability to monitor temperature (which can be done within a tenth of a degree) but to control the temperature of a reactor or a calcination process.

- A POSITA would understand *"about"* to include deviations of at most plus or minus five degrees centigrade from a specified temperature.

Ex. 1001 at ¶36; Paper 18 at 24; Ex. 2006, p. 21, ¶36.

The Grounds

Petitioners' Grounds

- **Ground I:** Lack of Written Description under §112(a)
- **Ground II:** Indefiniteness §112(b)
- **Ground III:** Obviousness over Wright '451 (Ex. 1039)
- **Ground IV:** Obviousness over *De Bruyn* (Ex. 1008) in view of *Takamura* (Ex. 1044)
- **Ground V:** Patent Owner Estoppel

Paper 20, ¶¶3-10, Appendix A.

Ground I: Written Description

Petitioners' Ground I – § 112(a)





(Paper 20 at 2-3; Paper 29 at 6-8.)

PO has not carried its burden to show where the specification of the '048 patent does not indicate possession of "a conversion of at least 95% % [sic] and has a bio-1-alkene regioselectivity of 95% to about 99%...". Ex. 1046 at ¶31

Dr. Wright states "Further, it was known that for a specific catalyst, at any given reaction temperature the regioselectivity and conversion are largely a function of flow-rate." Ex. 2006 at ¶115; Ex. 1046 at ¶32. Yet, despite this being "known", no flow rate is claimed or disclosed in the '048 patent. Because reaction temperature and flow-rate largely control the claimed outcomes, without a disclosure of both flow-rate and temperature, it is not possible for a POSITA to understand that the inventors possessed the Substitute Claims. Ex. 1046 at ¶32.

Paper 20 at 3.

Petitioners' Opposition Cites *Novozymes A/S v. DuPont*



Novozymes A/S v. DuPont Nutrition Biosciences APS,
723 F.3d 1336 (Fed. Cir. 2013)

V. Claims 29-44 are invalid under 35 USC §112

The Federal Circuit has stated that "to satisfy §112, a patent's written description 'must 'clearly allow persons of ordinary skill in the art to recognize that [the inventor] invented what is claimed.'" *Novozymes v. DuPont Nutrition Biosciences APS*, 723 F.3d 1336, 1346 (Fed. Cir. 2013) (internal citations omitted).

Paper 20 at 2-3.

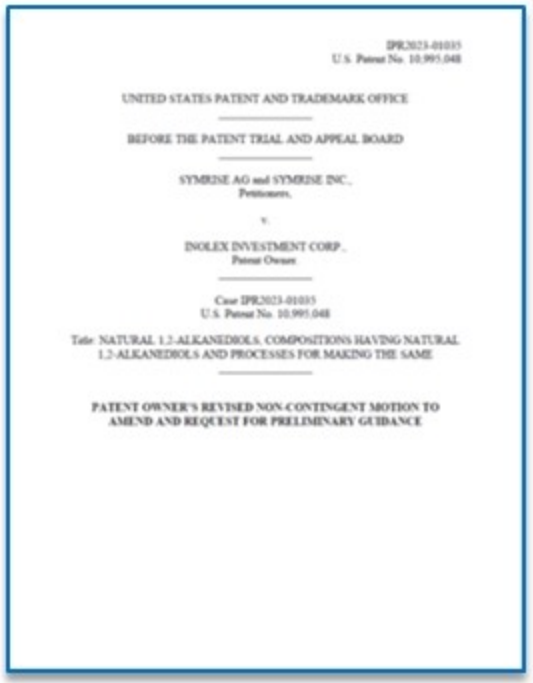
As described, claim 1 of the '23 patent recites an alpha-amylase variant that (1) has at least 90% sequence identity to BSG alpha-amylase, (2) includes an amino acid substitution at serine 239, and (3) has increased thermostability at pH 4.5, 90°C, and 5 ppm calcium.

* * *

The 2000 application, however, contains no disclosure of any variant that actually satisfies the claims, nor is there anything to suggest that Novozymes actually possessed such a variant at the time of filing.

723 F.3d at 1348.

Written Support for Substitute Claim 29



IPR2023-01035
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PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND AND REQUEST FOR PRELIMINARY GUIDANCE

(Paper 18 at 8.)

	Element(s) of Substitute Claims	Citation to Support in Non-Provisional Priority Application (Ex. 1006), and As-Filed Specification (Ex. 1005)
[29e]	wherein the resulting bio-alkene has a carbon chain of 6 to 10 carbon atoms and	**Ex. 1006** (pp. 23-79) at [0031], [0066], claim 3. **Ex. 1005** (pp. 4-54) at [0031], [0066], claim 3.
[29f]	is produced in a conversion of at least 95% and a bio-1-alkene regioselectivity of 95% to about 99% from a single-pass reaction after at least 20 hours time-on-stream.	**Ex. 1006** (pp. 23-79) at [0031], [0033], [0066], [0074], [0076], Table 1, [0125], Table 2, [0127], [0133], Table 5, [0134], claim 11. **Ex. 1005** (pp. 4-54) at [0023], [0031], [0033], [0066], [0074], [0076], Table 1, [0125], Table 2, [0127], [0133], Table 5, [0134], [0135], Fig. 7.

Paper 18 at 8.

Exhibits 1005 & 1006



NON-PROVISIONAL
UTILITY PATENT APPLICATION
TRANSMITTAL - 37 CFR 1.53(b)

Attorney Docket No.: 10113-198E3
First Named Inventor: Rocco Burgo
ELECTRONICALLY FILED
Applicant Name: Index Investment Corporation
Total Pages of Transmittal Form: 3

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

Transmitted herewith for filing is the non-provisional utility patent application entitled:

NATURAL 1,2-ALKANEDIOLS, COMPOSITIONS HAVING NATURAL 1,2-ALKANEDIOLS AND PROCESSES FOR MAKING THE SAME

which is:
as [ ] Original; or
a [X] Continuation, [ ] Divisional, or [ ] Continuation-in-part (CIP) of prior U.S. Non-Provisional Patent Application No. 16/262,902, filed January 30, 2019, which in turn is based on Provisional Patent Application No. 62/623,985, filed January 30, 2018.

Enclosed are:
[X] Specification (including Abstract) and claims 44 pages.
[X] 7 sheets of drawings (formal).
[X] Application Data Sheet Under 37 C.F.R. 1.76.
[X] Unexecuted: [X] Inventor Declaration, [ ] Inventor Declaration and Power of Attorney, [ ] Inventor Oath, [ ] Substitute Statement.
[ ] Copy of [ ] Declaration, [ ] Declaration and Power of Attorney from prior application.
[ ] Copy of separate Power of Attorney (including 37 CFR 3.73(b) statement, if applicable).
[ ] Microfiche computer program (Appendix).
[ ] Nucleotide and/or Amino Acid Sequence Submission, including:
[ ] Computer readable copy [ ] Paper Copy [ ] Verified Statement.
[X] Name of Assignee: Index Investment Corporation.

1

Syngrise AG, EX1005
Syngrise AG v. Index Inc.
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1 of 236

(Ex. 1005)

EXAMPLE 3

[0125] Catalysts prepared in EXAMPLE 1 and EXAMPLE 2 were loaded into separate 75 mL (Sigma Aldrich Part No. Z173592) reactors, each was wrapped with heating tape and covered with fiberglass insulation. The temperature was controlled by affixing a thermocouple to the outside skin of the reactor, this input was used for a temperature controller. Bio-1-octanol was fed into the reactor maintained at 315°C using an HPLC pump. The bio-1-octanol feed rate was adjusted in a manner to obtain conversion more than 95%, but less than 100%. After 1 h of continuous operation a sample was collected and analyzed for analyzing chemical conversion of the bio-1-octanol and regioselectivity of bio-1-octene. The results are shown below:

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

Paper 18 at 14; Ex. 1005 at 39, ¶125, Table 2; Ex. 1006 at 58, ¶125, Table 2.

Dr. Wright's Testimony



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

THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

62. First, Dr. Taber does not disagree with my prior statement that a POSITA would know that temperature and flow rate (or, more precisely, WHSV) are two fundamental process parameters that can be controlled by an operator. **Ex. 1046**, ¶32 (citing **Ex. 2005**, ¶115). Therefore, for a specific catalyst at any specific temperature and decreasing flow rate typically lead to increased 1-alcohol conversion but decreased regioselectivity (and *vice versa*). Therefore, a process that specifies the optimum temperature range that a specific catalyst dehydrates a specific 1-alcohol, provides sufficient information for a POSITA to be able to practice that process. Indeed, a POSITA would understand that the inventors were in possession of the claimed process—and could reproduce the claimed process—because it specifies that the catalyst is calcium-promoted and then calcined at a specific temperature, which is then heated in a reactor at a specific temperature.

Paper 25 at 7; Ex. 2010 at ¶62.

Exhibits 1005 & 1006



NON-PROVISIONAL
UTILITY PATENT APPLICATION
TRANSMITTAL - 37 CFR 1.53(b)

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

Attorney Docket No.: 10113-198E3
First Named Inventor: Rocco Burgo
ELECTRONICALLY FILED
Applicant Name: Index Investment Corporation
Total Pages of Transmittal Form: 3

Transmitted herewith for filing is the non-provisional utility patent application entitled

NATURAL 1,2-ALKANEDIOLS, COMPOSITIONS HAVING NATURAL 1,2-ALKANEDIOLS AND PROCESSES FOR MAKING THE SAME

which is:
an [ ] Original; or
a [X] Continuation, [ ] Divisional, or [ ] Continuation-in-part (CIP) of prior U.S. Non-Provisional Patent Application No. 16/262,902, filed January 30, 2019, which in turn is based on Provisional Patent Application No. 62/623,985, filed January 30, 2018.

Enclosed are:
[X] Specification (including Abstract) and claims 44 pages
[X] 7 sheets of drawings (formal)
[X] Application Data Sheet Under 37 C.F.R. 1.76
[X] Unexecuted [X] Inventor Declaration, [ ] Inventor Declaration and Power of Attorney, [ ] Inventor Oath, [ ] Substitute Statement
[ ] Copy of [ ] Declaration, [ ] Declaration and Power of Attorney from prior application
[ ] Copy of separate Power of Attorney (including 37 CFR 3.73(b) statement, if applicable)
[ ] Microfiche computer program (Appendix)
[ ] Nucleotide and/or Amino Acid Sequence Submission, including:
[ ] Computer readable copy [ ] Paper Copy [ ] Verified Statement
[X] Name of Assignee: Index Investment Corporation

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Syzorise AG, EX1005
Syzorise AG v. Index Inc.
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(Ex. 1005)

EXAMPLE 5

[0127] The 48 g of the catalyst prepared in EXAMPLE 4 was loaded into a 75 mL (Sigma Aldrich Part No. Z173592) reactor, wrapped with heating tape, and covered with fiberglass insulation. The temperature was controlled by affixing a thermocouple to the outside skin of the reactor, this input was used for a temperature controller. Bio-1-octanol was fed at 0.2 mL/min into the reactor maintained at 315°C. After >1,400 h of TOS, that included regular shut-downs and start-ups, catalyst performance with regard 1) chemical conversion of the bio-1-octanol, 2) product selectivity, 3) and regioselectivity for bio-1-octene was unchanged. A GC chromatogram of the crude product collected from the reactor is shown in Fig. 3 with an octene selectivity of 96% and with chemical yield of bio-1-octene of 94%.

Paper 18 at 14; Ex. 1005 at 40, ¶127, Table 5; Ex. 1006 at 59, ¶127.

Dr. Wright's Testimony



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

THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

63. Second, Dr. Taber ignores the fact that Example 5 of the '048 patent specifically discloses a flow rate of 0.2 mL/min. **Ex. 1001**, col. 27, ll. 20-28 and provides the catalyst weight used (Example 5). Thus, a POSITA reviewing Example 5 of the'048 patent would understand that the inventors were in possession of a method of using the claimed calcium-promoted catalyst at a flow rate of 0.2 mL/min. A POSITA would also recognize that similar flow rates could be utilized to practice the claimed processes. These conditions provide the POSITA with an exact WHSV for the invention's catalyst.

Paper 25 at 7; Ex. 2010, p. 36, ¶63.

Exhibits 1005 & 1006



(Ex. 1001, '048 Patent)

EXAMPLE 11

[0133] Petro-1-hexanol was fed into a 75 mL reactor packed with the catalyst prepared in EXAMPLE 4. The catalyst used in this example had greater than 1,400 h of TOS used for bio-1-octanol dehydration to bio-1-octene. For this series of dehydration reaction examples, the temperature was varied from 330°C to 370°C and the feed rate was adjusted in order to achieve between 1 and 2% of hexanol passing through the reactor. The results for the hexanol dehydration are shown 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

Paper 18 at 14; Ex. 1005 at 43, ¶133, Table 5; Ex. 1006 at 62, ¶133, Table 5; Ex. 2006, p. 44, ¶80.

Petitioners' Sur-Reply Cites *Ariad Pharma. v. Eli Lilly & Co.*



Ariod Pharma., Inc. v. Eli Lilly & Co., 598 F.3d 1336 (Fed. Cir. 2010)

Finally, an argument improperly incorporated by reference from Dr. Wright's declaration suggests that because some prior art references can render obvious the claims, they support PO's argument that Example 5 alone shows possession by the inventors. Ex. 2010 at ¶64. However, Dr. Wright misunderstands the test for written description. PO needs to show possession of the full scope of the claims, which they have failed to do. *Ariad Pharma.*, 598 F.3d at 1352 ("[A] description that merely renders the invention obvious does not satisfy the requirement" of §112). Since Dr. Wright relies on the test for obviousness, not written description, his testimony on this point should be given little, if any, weight.

Paper 29 at 8.

Dr. Wright's Testimony



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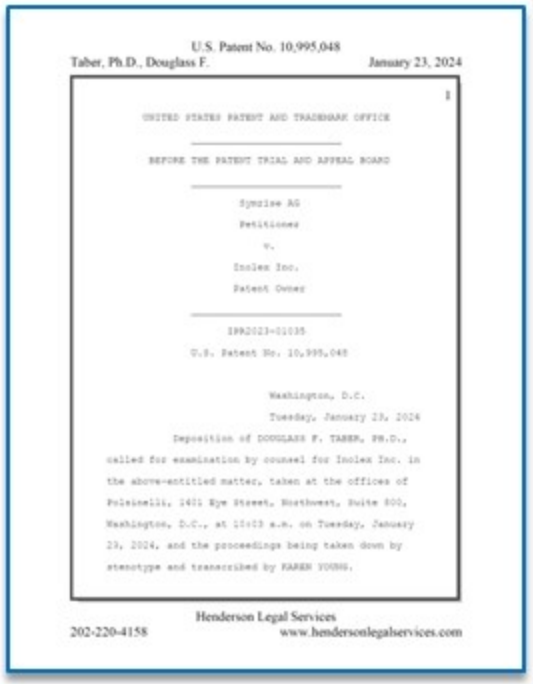
THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

64. Third, Dr. Taber testified that process control variables such as flow rate would not have been important to a POSITA reviewing prior art dehydration processes described in references such as *Appleby* and *Komarewsky* (**Ex. 1028** and **Ex. 1029**, respectively). Rather, the most important information showing that those authors had developed a dehydration process was the results:

Ex. 2010 at ¶64; Paper 25 at 8.

Dr. Taber's Testimony



U.S. Patent No. 10,995,048
Taber, Ph.D., Douglass F. January 23, 2024

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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

Syncise AG
Petitioner
v.
Inolex Inc.
Patent Owner

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

Washington, D.C.
Tuesday, January 23, 2024

Deposition of DOUGLASS F. TABER, PH.D., called for examination by counsel for Inolex Inc. in the above-entitled matter, taken at the offices of Polsinelli, 1401 Eye Street, Northwest, Suite 800, Washington, D.C., at 10:03 a.m. on Tuesday, January 23, 2024, and the proceedings being taken down by stenotype and transcribed by KAREN YOUNG.

Henderson Legal Services
202-220-4158 www.hendersonlegalservices.com

Ex. 2005 (Taber Dep. Tr. 01/23/2024)

Q. Okay, so would a person of ordinary skill in the art expect there to be a difference between the 350 degree reaction temp described in Komarewsky and the 380 to 400 degree reaction temp described in Appleby?

A. **There are many variables that are not mentioned in these articles,** what was the mass of the aluminum oxide, the activated alumina that was used as a catalyst, what were the dimensions of this tubular heater. We do know that it was 40 centimeters long and 12 millimeters in diameter. Was that inside diameter or outside diameter? **So it's a little difficult to compare the two precisely because we really don't know flow rates, all these things. We know they're both successful.**

Ex. 2005, p. 36:8-22; Paper 25 at 8; Ex. 2010, p. 37, ¶64.

Dr. Wright's Testimony



UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

SYMRISE AG and SYMRISE INC.,
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INOLEX INVESTMENT CORP.,
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THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

Ex. 2005, p. 36, ll. 8-22. Indeed, both Appleby and Komarewsky disclose how the catalysts were prepared and the temperatures at which the processes were performed. Therefore, I agree with Dr. Taber that a POSITA would both understand that *Appleby* (**Ex. 1028**), *Komarewsky* (**Ex. 1029**), and the '048 patent inventors were in possession of a dehydration process that provided the disclosed results, and a POSITA that wanted to reproduce the processes of *Appleby*, *Komarewsky*, or the '048 patent could do so based on those disclosures. Notably, the '048 patent gives exact part numbers for the reactor, temperature probe location, flow rate, and catalyst weight, which would clearly demonstrate to a POSITA that the inventors were in possession of the claimed process—and, unlike so many of the prior art references that do not include such detailed information—enable the process to be reproduced.

Ex. 2010, p. 37, ¶64; Paper 25 at 8.

Dr. Wright's Testimony



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SYMRISE AG and SYMRISE INC.,
Petitioners,

v.

INOLEX INVESTMENT CORP.,
Patent Owner

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

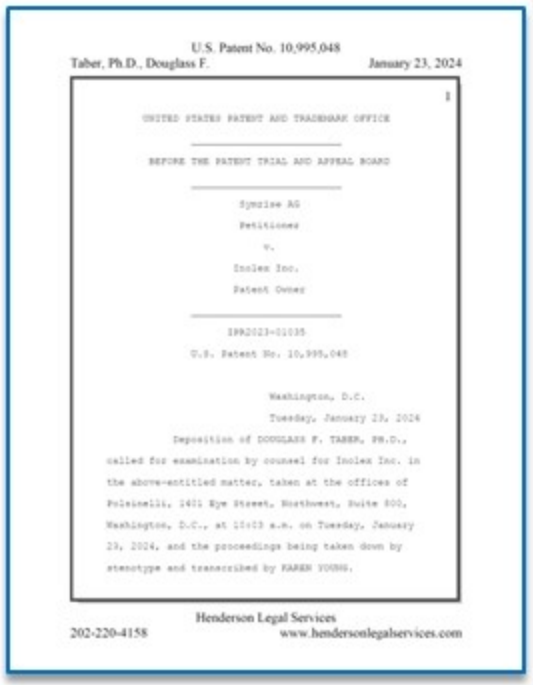
THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

65. Further, it is rather surprising that Dr. Taber is now alleging that flow rate would be essential information for a POSITA to know in order to practice the claimed invention, when he himself testified "I have no idea" what "WHSV" refers to, which is a basic concept in heterogeneous catalysis a POSITA would surely be aware of and is an acronym for "Weight Hour Space Velocity". **Ex. 2005**, p. 64, ll. 10-19. WHSV is analogous to flow rate and defines the weight of the reactant feed flowing per unit weight of catalyst per hour, and optimizing and controlling the WHSV is one of only two fundamental process control parameters any POSITA would understand before undertaking the dehydration of a bio-1-alcohol using a heterogeneous catalyst, the other being temperature. Again, the POSITA in this matter has a B.S. in chemistry with at 1-2 years of industrial experience, or a masters degree with commensurate experience. In the general area of heterogeneous catalysis, any POSITA would understand WHSV and how to adjust it for achieving optimum catalyst performance.

Ex. 2010, p. 38, ¶65; Paper 25 at 8.

Dr. Taber's Testimony


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Taber, Ph.D., Douglass F. January 23, 2024

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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

Synclae AG
Petitioner
v.
Inolex Inc.
Patent Owner

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

Washington, D.C.
Tuesday, January 23, 2024

Deposition of DOUGLASS F. TABER, PH.D., called for examination by counsel for Inolex Inc. in the above-entitled matter, taken at the offices of Polsinelli, 1401 Eye Street, Northwest, Suite 800, Washington, D.C., at 11:13 a.m. on Tuesday, January 23, 2024, and the proceedings being taken down by stenotype and transcribed by KAREN YOUNG.

Henderson Legal Services
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Ex. 2005 (Taber Dep. Tr. 01/23/2024)

10 Q. There's also a reference to -- the top of
11 the page says the WHSV as being about 6. Do you see
12 that?
13 **A. Yes.**
14 Q. Do you know what WHSV is referring to
15 there?
16 **A. I have to stop and think about that. I**
17 **have no idea.**
18 Q. Okay.
19 **A. I still have no idea.**

Ex. 2005, p. 64:10-19 (discussing Ex. 1035); Ex. 2010, p. 38, ¶65; Paper 25 at 8.

Ground II: Indefiniteness

Petitioners' Ground II – § 112(b)



UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

SYNBIOSE AG AND SYNBIOSE INC.
PETITIONER,
v.
INOLEX INVESTMENT CORPORATION,
PATENT OWNER.

IPR2021-01015
Patent 10,995,048

OPPOSITION TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND



UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

SYNBIOSE AG AND SYNBIOSE INC.
PETITIONER,
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PATENT OWNER.

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Patent 10,995,048

SUR-REPLY TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND

(Paper 20 at 4; Paper 29 at 8-10.)

29. (Proposed Substitute for Claim 1) A method for making a bio-alkene, comprising:

dehydrating a bio-1-alcohol by heating the bio-1-alcohol in a continuous flow reactor at a temperature of 240 °C to 360 °C with a calcium-promoted γ-alumina catalyst calcined at a temperature of about 420 °C to about 480 °C, wherein the catalyst has not been silanized, and

wherein the resulting bio-alkene has a carbon chain of 6 to 10 ~~about 5 to about 20~~ carbon atoms and is produced in a conversion of at least 95% % and ~~has~~ a bio-1-alkene regioselectivity of 95% to about 99% from a single-pass reaction after at least 20 hours time-on-stream~~at least about 80%~~.

See Paper 20 at 4; Paper 29 at 8-10.

Petitioners' Ground II – § 112(b)



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OPPOSITION TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND

Paper 20

PO has chosen language that does not reasonably inform a POSITA of the scope of the invention. Ex. 1046 at ¶36. The claim language "heating the bio-1-alcohol in a continuous flow reactor at a temperature of 240 °C to 360 °C" has two distinct and incompatible meanings. In one interpretation the claim covers a reactor with a single temperature in the temperature range of 240 °C to 360 °C. The second interpretation is that the 1-alcohol is heated in a reactor that starts at 240 °C and ends at 360 °C, encompassing the claimed temperature range.

Paper 20 at 4.

Petitioners' Ground II – § 112(b)



UNITED STATES PATENT AND TRADEMARK OFFICE

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OPPOSITION TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND

Paper 20

The specification of the '048 patent supports both interpretations. Ex. 1046 at ¶37. In Examples 3, 4, 5, 10, and 11, the dehydration takes place at a single temperature. *See e.g.,* Ex. 1001 at 26:40-67; 27:12-35; and 28:55-11:25. The specification also discusses that in a single-pass reactor, "the reaction tube is heated isothermally, the tube having from 1 to 10 reaction zone temperatures along the length of the tube, each heated in the range of about 240 °C to 360 °C in order to optimize catalyst performance." Ex. 1001 at 15:33-37. Accordingly, a POSITA is not informed with reasonable certainty about the scope of the claim. Ex. 1046 at ¶38.

Paper 20 at 4-5.

Legal Standard for Indefiniteness Under § 112(b)



Nautilus, Inc. v. Biosig Instruments, Inc., 572 U.S. 898 (2014)

Cognizant of the competing concerns, we read § 112, ¶2 to require that a patent's claims, viewed in light of the specification and prosecution history, inform those skilled in the art about the scope of the invention with reasonable certainty. The definiteness requirement, so understood, mandates clarity, while recognizing that absolute precision is unattainable. The standard we adopt accords with opinions of this Court stating that *"the certainty which the law requires in patents is not greater than is reasonable, having regard to their subject-matter."* *Minerals Separation, Ltd. v. Hyde*, 242 U.S. 261, 270 (1916). *See also United Carbon Co. v. Binney & Smith Co.*, 317 U.S. 228, 236 (1942) (*"claims must be reasonably clearcut"*); *Markman v. Westview Instruments, Inc.*, 517 U.S. 370, 389 (1996) (claim construction calls for *"the necessarily sophisticated analysis of the whole document,"* and may turn on evaluations of expert testimony).

572 U.S. at 910-911; Paper 25 at 9.

Dr. Wright's Testimony



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Ex. 2010 – Third Wright Declaration

68. First, as disclosed in the '048 patent as well as many other references of record, a POSITA would readily understand that continuous flow reactors can comprise one or more heated "zones," the temperature of which can be independently controlled and monitored. *See* **Ex. 1001**, col. 15, ll. 34-39; **Ex. 1007**, ¶[0175] (discussing Fig. 1, which includes separate zones for reagent entry and the fluidized bed); **Ex. 2003**, p. 376, 379 (Fig. 1, describing a dual bed reactor that includes a pre-heater, and two separate thermocouple controls within the reactor);

Ex. 2010, pp. 39-40, ¶68; Paper 25 at 8-9.

Dr. Wright's Testimony



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THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

70. A POSITA reviewing the specification of the '048 patent (as well as other references of record in this proceeding) would understand that a reactor (or separate zones of a reactor) are controlled at a specific temperature over the course of the process. *See* **Ex. 1001**, col. 26, ll. 52-67 (Example 3, in which the reactor temperature was maintained at 315 °C using a thermocouple); col. 27, ll. 20-35 (Example 5, in which the reactor temperature was maintained at 315 °C using a thermocouple); col. 29, ll. 1-11 (Example 11, in which the reactor temperature was maintained at 330 °C, 350 °C, or 370 °C). Indeed, a POSITA would readily understand that "[i]t is also possible [and common] to carry out a series of experiments *each at different temperatures* … ." **Ex. 2002**, p. 108. Further, Dr.

Ex. 2010, pp. 40-41, ¶70; Paper 25 at 8-9.

Petitioners' Sur-Reply – § 112(b)



UNITED STATES PATENT AND TRADEMARK OFFICE

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SUR-REPLY TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND

Paper 20

In its Reply, PO agrees that the POSITA can read the specification to include both interpretations provided by Petitioner. *See* Reply at 8-9. However, PO and Dr. Wright make the argument that the claim should implicitly be construed to mean "the continuous flow reactor be heated at a temperature within the specified range. *Id*. at 8; Ex. 2010 at 75. PO's attempt to rewrite the claim to provide definiteness on what is heated is improper. The claim states "heating the bio-1-alcohol"–not heating the reactor. The claim can, therefore, include both interpretations of the claim as argued by Petitioner. *See* Opp. at 4-5 (The second interpretation is that the 1-alcohol is heated at 240 °C to 360 °C).

Paper 29 at 9.

Dr. Wright's Testimony



UNITED STATES PATENT AND TRADEMARK OFFICE

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THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

69. A POSITA would also understand that a reactor having more than one heated zone is typically maintained at a constant temperature through the use of a feedback mechanism such as a thermocouple. In the case of a reactor having multiple heating zones, a POSITA would recognize that this permits the temperature of each zone of the reactor to be independently controlled and maintained at a constant temperature. Thus, a POSITA would understand that a "reaction tube" or "reactor" can either be "heated isothermally" (in which all the reactor zones are at the same temperature), or each of the reactor zones can be separately set to a temperature in the range of 240 °C to 360 °C. *See* **Ex. 1001**, col. 15, ll. 33-37.

Ex. 2010, p. 40, ¶69; Paper 25 at 8-9.

Dr. Wright's Testimony



UNITED STATES PATENT AND TRADEMARK OFFICE

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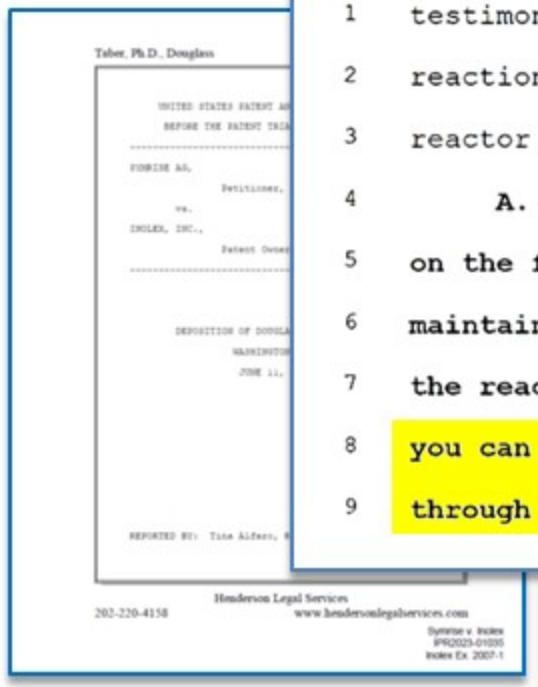
THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

71. Nor is there anything about the specific words of the phrase at issue that fails to inform a POSITA as to the scope of the claimed process. Indeed, based on my review of the prior art of record, similar language is commonly used to describe processes that can be carried out over a temperature range. *See, e.g.*, **Ex. 1007**, ¶[0065] (describing a "reaction in which the alcohol formed is dehydrated can be carried out, for example, *in the temperature range included from 250 to 400 °C*"; **Ex. 1008**, ¶[0037] (describing "a dehydration reaction *at temperatures from 200 to 450 °C*…"); **Ex. 1027**, p. 146, col. 2 (which is Petitioners' own published patent applications, and describes a reactor having multiple catalyst beds in one reactor, with *reactions carried out at temperatures ranging from 280 to 325 °C*). Indeed, Petitioners' own published patent application describes dehydration reactions for 1-alcohols "wherein the dehydration and/or cleavage *is performed at a temperature ranging from 255 °C to 290 °C*" using a reactor having multiple zones that can be independently controlled and maintained at different temperatures. *See* **Ex. 2008**, ¶¶[0015], [0126].

Ex. 2010, p. 42, ¶71; Paper 25 at 8-9.

Dr. Taber's Testimony


Taber, Ph.D., Douglass

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(Ex. 2007)

22 Q. Okay. And even at 315 degrees it's your
1 testimony that the exothermic nature of the
2 reaction could influence the temperature of the
3 reactor by up to 20 degrees?
4 A. The temperature of the reactor will depend
5 on the flow rate. If the flow rate can be
6 maintained very consistently and the heat loss from
7 the reactor can be maintained very consistently,
8 you can maintain a very consistent temperature
9 through most of the procedure.

10 Q. Okay. And just to close the loop, when a
11 POSITA reads that a procedure of dehydration
12 reaction was performed at a temperature of 315,
13 what would be the variance that you would expect as
14 a POSITA?
15 A. Well, the POSITA would be motivated to
16 initially heat the reactor. And so now you've got
17 heat tapered around your reactor, whatever you're
18 doing, you're going to heat the reactor to get the
19 temperature. And then you start to run your
20 substrate in, temperature goes up, okay, turn the
21 heat off, keep running it in, get things under
22 control. And so on a single run the temperature
1 might be highly variable. Once the POSITA's gained
2 experience with the reactor and with the flow rates
3 and all such things, then probably they can be more
4 consistent about maintaining the temperature.

Ex. 2007, p. 162:22 – 164:4; Ex. 2010, pp. 40-41, ¶70; Paper 25 at 8-9.

Dr. Wright's Testimony



UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

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THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

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75. Finally, based on the lack of ambiguity in the phrase at issue, in my opinion there is no reason to further construe the phrase "heating the bio-1-alcohol in a continuous flow reactor at a temperature of 240 °C to 360 °C," and it should have its plain and ordinary meaning that would be readily understood by a POSITA. In the event that the PTAB believes this phrase requires construction, a POSITA would understand that this phrase as it is used in substitute claim 29 (as well as substitute claims 35, 38, 41, and 44) merely requires the continuous flow reactor to be "heated at a temperature within the specified range."

Ex. 2010, p. 43, ¶75; Paper 25 at 8.

Grounds III & IV: Obviousness

Petitioners' Ground IV – § 103



UNITED STATES PATENT AND TRADEMARK OFFICE

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OPPOSITION TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND



UNITED STATES PATENT AND TRADEMARK OFFICE

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SUR-REPLY TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND

(Paper 20 at 2-3; Paper 29 at 6-8.)

- Ground IV: Substitute claims 29-44 are allegedly obvious over U.S. Pub. No. 2005/00389 (Ex. 1008, *"De Bruyn"*) in view of EP 2 990 395 B (Ex. 1044, *"Takamura"*)

Paper 20 at 16-19; Paper 29 at 5-6.

De Bruyn – Ex. 1008



United States
Patent Application Publication
De Bruyn et al.
Pub. No.: US 2005/0065389 A1
Pub. Date: Mar. 24, 2005

METHOD OF INCREASING THE CARBON CHAIN LENGTH OF OLEFINIC COMPOUNDS

Publication Classification

ABSTRACT

Synrise AG, EX1008
Synrise AG v. Index Inc.
IPR2023-01035

Ex. 1008 – *De Bruyn*

2001 Patent Application by SASOL (Pub'd 2005)

[0005] The inventors of the present invention have now developed a novel process of increasing the carbon chain length of olefinic compounds, including and especially α-olefins. Accordingly, shorter α-olefins such as 1-pentene can be converted to 1-hexene.

* * *

[0036] Any suitable dehydration process may be used to convert the alcohol with the increased carbon chain length, to the olefinic compound. In cases where the alcohol is a n-alcohol (or significant concentrations thereof are present) the dehydration process is preferably controlled to produce α-olefinic compounds.

[0037] Many different dehydration processes are known and they are accordingly not discussed in any detail in this specification. Preferably the dehydration is carried out under low acidity conditions and a low acidity catalysts support such as Al_2O_3; SiO_2; TiO_2 or ZrO_2 may be employed to afford a dehydration reaction at temperatures from 200 to 450° C., typically from 250 to 350° C. and at pressures from 0 to 30 bar_g, typically 0 to 5 bar_g. The catalyst may comprise a gamma-alumina catalyst or a promoted alumina catalyst e.g $CaO.Al_2O_3$, $Ca_2O_3.Al_2O_3$.

Ex. 1008, ¶¶ 5, 36-37; Ex. 2010, p. 46, ¶80.

De Bruyn – Ex. 1008

2001 Patent Application by SASOL (Pub'd 2005)



(19) United States
(12) Patent Application Publication (10) Pub. No.: US 2005/0065389 A1
De Bruyn et al. (43) Pub. Date: Mar. 24, 2005

(54) METHOD OF INCREASING THE CARBON CHAIN LENGTH OF OLEFINIC COMPOUNDS

Publication Classification

(51) Int. Cl.[7] C07C 1/00
(52) U.S. Cl. 585/639

(75) Inventors: Cornelis Johannes De Bruyn, Secunda (ZA); Ewald Wattenmayer De Wet, Pearl (ZA); Jan Matthews Botha, Sasolburg (ZA); Jan Petrus Kotzé Bronkhorstspruit, Vosloorus (ZA)

(57) ABSTRACT

Correspondence Address:
Finnegan Henderson Farabow
Garrett & Dunner
1300 I Street NW
Washington, DC 20005-3315 (US)

(21) Appl. No.: 10/495,214
(22) PCT Filed: Sep. 11, 2002
(86) PCT No.: PCT/IB02/03638
(30) Foreign Application Priority Data
Sep. 17, 2001 (ZA) 2001/7636

Sasol Ex. 1008
Sasol v. Iodex Inc.
IPR2023-01035

Ex. 1008 – *De Bruyn*

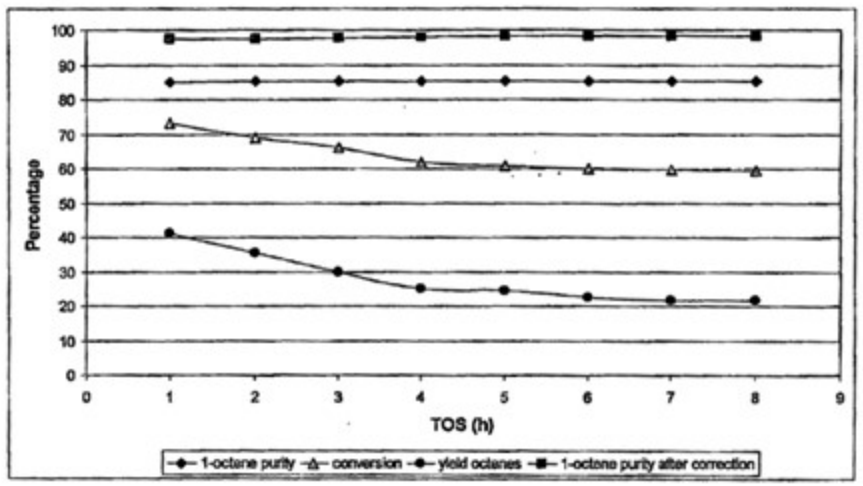
EXAMPLE 3

Dehydration of the Formed Alcohol

[0053] In a vertical pipe reactor 400 mm in length and 25.4 mm inner diameter, a catalyst bed of about 12 grams of gamma-Al_2O_3 was loaded, supported on quartz wool. The reactor was heated to 315° C. and 1-hexanol (98% pure) was fed into the reactor at a LHSV of 5 hr^{-1} at atmospheric pressure. The reactor product (after removal of water and hexenes) was recycled to the reactor at a mass ratio of feed:recycle of 0.75: 2.0. A total of 94% of the 1-hexanol was converted to alkenes, with a selectivity towards hexenes of 98.6%, of which 97.5% was 1-hexene.

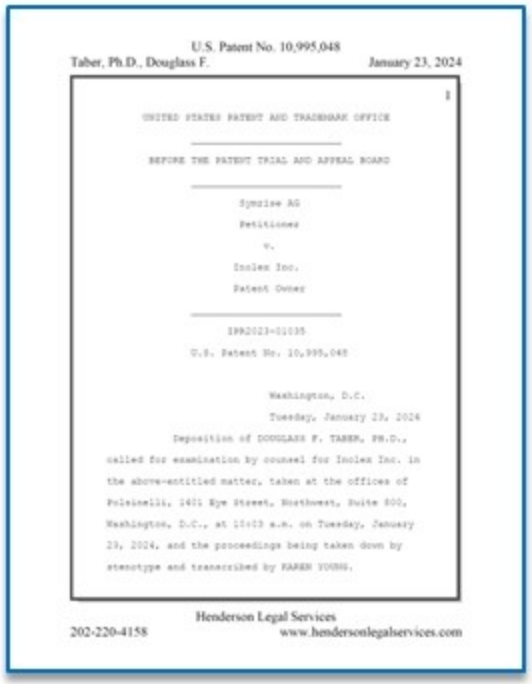
Ex. 1008, ¶53;
Ex. 2010, pp. 45-46, ¶79, p. 57, ¶101, p. 62, ¶106.

Figure 2. Percentage conversion, yield of octenes and purity versus time-on-stream



Ex. 1008, 10 (Fig. 2);
Ex. 2010, p. 46, ¶81.

Dr. Taber's Testimony



U.S. Patent No. 10,995,048
Taber, Ph.D., Douglass F. January 23, 2024

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Syncise AG
Petitioner
v.
Inolex Inc.
Patent Owner

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

Washington, D.C.
Tuesday, January 23, 2024

Deposition of DOUGLASS F. TABER, PH.D., called for examination by counsel for Inolex Inc. in the above-entitled matter, taken at the offices of Polsinelli, 1401 Eye Street, Northwest, Suite 800, Washington, D.C., at 10:03 a.m. on Tuesday, January 23, 2024, and the proceedings being taken down by stenotype and transcribed by KAREN YOUNG.

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Ex. 2005 (Taber Dep. Tr. 01/23/2024)

Q. Right. So yeah, we go back to De Bruyn at -- and I think the example that they give is again paragraph 67 and 68 on page 8 of De Bruyn.

A. Yes, and they were running to partial conversion in this example.

Q. Right, and so this is --

A. And so if you want to do complete conversion, presumably you're going to need slower flow rate or higher temperature or more catalyst to get full conversion to --

Q. Okay.

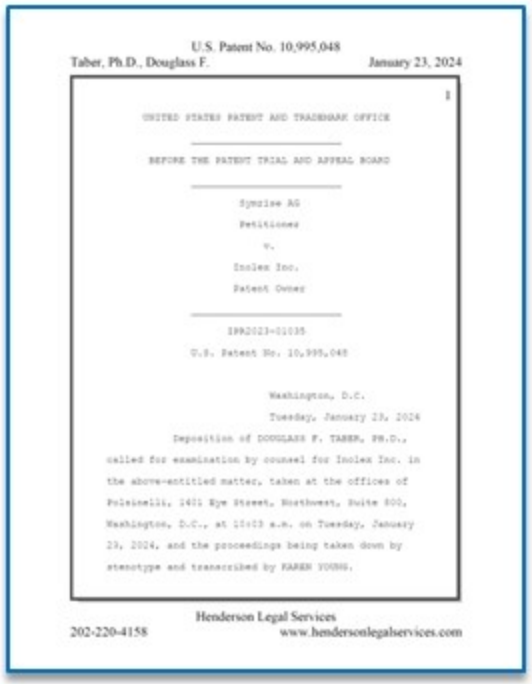
A. -- 1-hexene.

Q. And would you need to do the experiment to find out if -- if you could get full conversion?

A. Yes.

Ex. 2005, p. 92:7-21; Ex. 2010, p. 27, ¶47.

Dr. Taber's Testimony



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Ex. 2005 (Taber Dep. Tr. 01/23/2024)

14 Q. And then you conclude on the top of page 27
15 with, "The process at commercial scale is well
16 understood and predictable," and my question is what
17 are you referring to about the process as being well
18 understood and predictable?
19 **A. My understanding is the company produces**
20 **1-octene on a metric ton scale, and does so reliably.**
21 Q. Okay, and so a person of ordinary skill in
22 the art would be aware of that, right?
1 **A. Yes.**
2 Q. Would a person of ordinary skill in the art
3 know what catalyst SASOL is using for that conversion
4 on a commercial scale?
5 **A. They would know that they were using**
6 **gamma-alumina, but how the gamma-alumina was**
7 **optimized they would not necessarily know. As we**
8 **saw, they can purchase that, and it works.**

Ex. 2005, p. 90:14 - 91:8;
Ex. 2010, p. 67, ¶113.

Makgoba – Ex. 1027



Silication of γ-alumina catalyst during the dehydration of linear primary alcohols

N.P. Makgoba *, T.M. Sakaneka, J.G. Koortzen, C. van Schalkwyk, J.M. Botha, C.P. Nicolaides *

Symrise AG, EX1027
Symrise AG v. Isodex Inc.
IPR2023-01035

Ex. 1027 – Makgoba

2006 Publication from SASOL

It is our intention in this paper to provide data showing that γ-alumina on its own, without any modifications, can give high 1-alkene selectivities. It is also our aim to show that the loss of 1-alkene selectivity observed in the present study is a consequence of the accumulation of silicon on the catalyst during the dehydration reaction. The silicon is present in the alcohol feeds (even in small concentrations at the 1 ppm range and lower) and implants on the catalyst during the reaction.

Ex. 1027, 146 (Col. 1)

* * *

fraction. The 3-hexenes constitute only a small fraction of the total, but their boiling points are closer to that of the 1-hexene, which makes their separation more difficult. In Fig. 1, it can be observed that the 1-hexene purity is high (95–97%) at conversions less than 80%. At higher conversions, the extent of double-bond shift is more pronounced and the 1-hexene purity drops to below 95%. Based on process considerations, we have set a minimum 1-alkene purity of 95%. The data presented in Fig. 1 were obtained for runs of up to 8 h on-stream, and the conversion and the purity, were virtually constant for each reaction.

Ex. 1027, 147 (Col. 2)

Makgoba



Silication of γ-alumina catalyst during the dehydration of linear primary alcohols

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Symrise AG, EX1027
Symrise AG v. Isodex Inc.
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Ex. 1027 – Makgoba

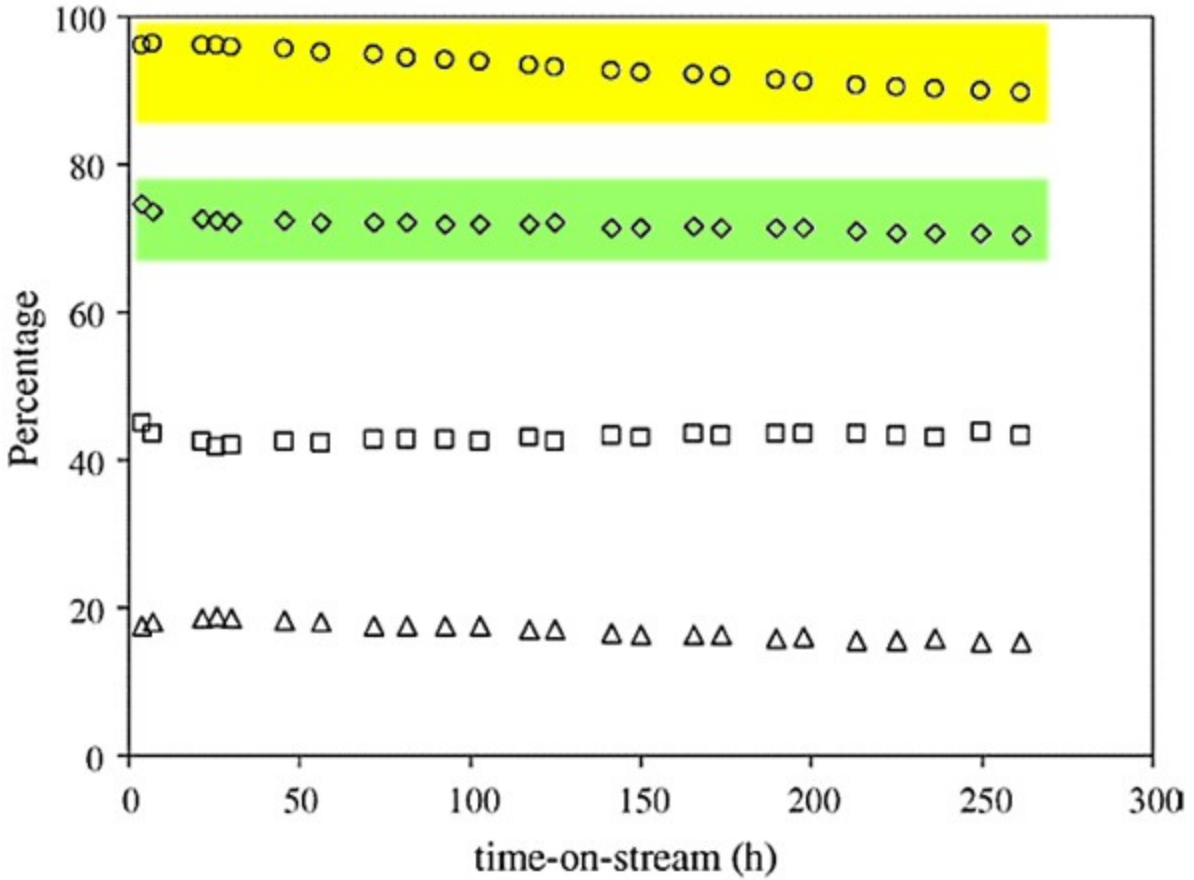
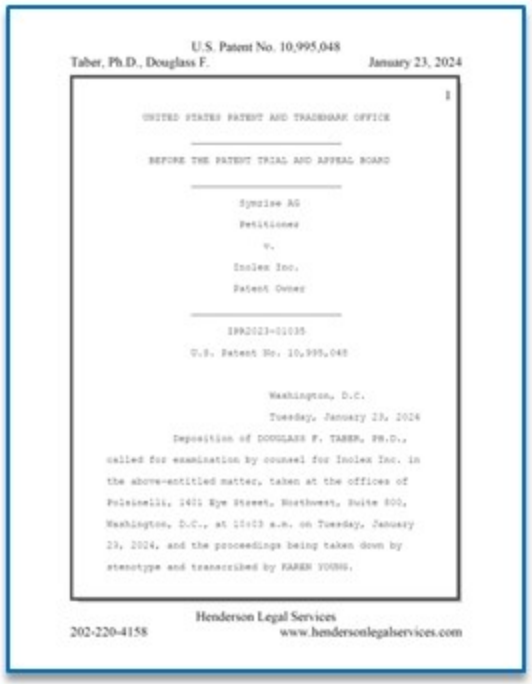


Fig. 2. 1-Hexanol dehydration over γ-alumina at 300 °C and WHSV = 8 h^{-1}: 1-hexene purity (○), conversion (◇), yield of hexenes (□) and yield of dihexyl ether (△).

Ex. 1027, 147 (Fig. 2)

Dr. Taber's Testimony


U.S. Patent No. 10,995,048
Taber, Ph.D., Douglass F. January 23, 2024

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Ex. 2005 (Taber Dep. Tr. 01/23/2024)

Q. Would -- would a person of ordinary skill in the art only look to commercially available catalysts, or would they also try to develop their own catalyst?

A. The person of ordinary skill in the art would have a choice. They could use a known catalyst from a commercial vendor such as SASOL if SASOL would sell it to them, or they could go back to commercial untreated gamma-alumina, or there may be some other vendor who's selling gamma-alumina for alcohol dehydration, you know.

Q. Uh-huh.

A. They would look first. Before they set about doing the experiments themselves, they would look and see what was commercially available.

Q. Okay, so first -- first priority is let's -- let's do the thing that's easiest. Is that fair?

A. That's right.

Ex. 2005, p. 94:17 – 95:12.

Dr. Wright's Testimony



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U.S. Patent No. 10,993,048

THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

77. *De Bruyn* utilizes commercially available γ-alumina catalysts, but a POSITA would not know whether any of those catalysts had been chemically modified or calcined. **Ex. 2001**, ¶40, ¶55, ¶64; *see also* **Ex. 2005** at p. 97, l. 21 – p. 99, l. 20; p. 101, ll. 6-20; **Ex. 2007**, p. 33, ll. 7-11; p. 125, l. 2 – p. 127, l. 20. However, Dr. Taber has stated—without any evidence—that a POSITA would know that the commercial catalyst Sasol uses is the product of treatment with calcium acetate followed by calcination. **Ex. 2007**, p. 32, ll. 14-19; p. 34, ll. 1-7. I disagree—not only is *De Bruyn* silent about whether the commercially available alumina catalysts used in *De Bruyn*'s examples were calcium-promoted or calcined, there is also no information about the temperature of calcination. In

Ex. 2010, p. 44, ¶77; Paper 25 at 10.

Takamura – Ex. 1044



EP 2 990 395 A1

EUROPEAN PATENT APPLICATION

Ex. 1044 – *Takamura*

European Patent filed by Kao Corp. in 2014

[0004] The present inventors have found that an olefin may be obtained in a short period of time with a high yield by performing liquid phase dehydration reaction of an alcohol by using such a catalyst as γ-alumina having particular weak acid sites, and had filed a patent application (see PTL 3).

* * *

Summary of the Invention

[0008] The present invention relates to a method for producing an olefin, containing subjecting a raw material alcohol to dehydration reaction in the presence of an acid catalyst, in which the acid catalyst contains a metal oxide, the raw material alcohol contains two or more kinds of alcohols having different numbers of carbon atoms each being a primary aliphatic alcohol having 8 or more and 22 or less carbon atoms, and a temperature of the reaction is 200°C or more and 300°C or less.

* * *

Solution to Problem

[0016] The inventors have found that oligomerization of an olefin can be suppressed, and thereby an olefin can be produced with a high yield, by performing dehydration reaction by combining a particular aliphatic alcohol.

Ex. 1044, ¶¶4, 8, 16.

Takamura – Ex. 1044



EP 2 990 395 A1

EUROPEAN PATENT APPLICATION

METHOD FOR PRODUCING OLEFIN

EP 2 990 395 A1

Ex. 1044 – Takamura

European Patent filed by Kao Corp. in 2014

Catalyst

[0036] In the present invention, an olefin is produced by subjecting primary aliphatic alcohols having 8 or more and 22 or less carbon atoms to dehydration reaction in the presence of an acid catalyst containing a metal oxide. In the present invention, an olefin may be produced by subjecting primary aliphatic alcohols having 12 or more and 22 or less carbon atoms to dehydration reaction in the presence of an acid catalyst containing a metal oxide.

Solid Acid Catalyst

[0037] The acid catalyst containing a metal oxide is preferably a solid acid catalyst from the standpoint of the enhancement of the reaction rate of the dehydration reaction while controlling the reaction safely and the standpoint of producing the olefin with a high efficiency by recovering and reusing the catalyst.

[0038] The ratio of the weak acid amount of the solid acid catalyst is preferably 60% or more, and more preferably 70% or more, from the standpoint of the suppression of the side reaction, and is preferably 100% or less, more preferably 99% or less, further preferably 90% or less, still further preferably 80% or less, and still further preferably 75% or less, from the standpoint of the enhancement of the reaction rate of the dehydration reaction.

Ex. 1044, ¶¶36-38.

Takamura – Ex. 1044



EP 2 990 395 A1

EUROPEAN PATENT APPLICATION

METHOD FOR PRODUCING OLEFIN

EP 2 990 395 A1

Ex. 1044 – Takamura

European Patent filed by Kao Corp. in 2014

[0051] Examples of a compound to be an oxide source of the element include an inorganic acid, such as sulfuric acid, thiosulfuric acid, phosphoric acid, tungstic acid and a silicic acid, and a salt thereof, an oxide, such as silica, an acidic metal alkoxide that is soluble in water through hydrolysis, such as tetramethyl orthosilicate and tetraethyl orthosilicate, and a heteropolyacid, such as silicotungstic acid and phosphotungstic acid, and a salt thereof.

[0052] As more specific compounds, examples of the inorganic acid and a salt thereof include sulfuric acid, sulfuric anhydride, ammonium sulfate, sodium sulfate, potassium sulfate, magnesium sulfate, calcium sulfate, sodium thiosulfate, ammonium thiosulfate, phosphoric acid, phosphorus pentoxide, ammonium phosphate, diammonium hydrogen phosphate, sodium phosphate, potassium phosphate, magnesium phosphate, calcium phosphate, tungstic acid, ammonium tungstate, sodium tungstate, potassium tungstate, calcium tungstate, ammonium paratungstate, ammonium metatungstate and tetramethylammonium silicate; examples of the oxide include colloidal silica, silica gel and water glass; examples of the acidic metal alkoxide include tetramethyl orthosilicate and tetraethyl orthosilicate; examples of the heteropolyacid and a salt thereof include silicotungstic acid, phosphotungstic acid, ammonium phosphotungstate, sodium phosphotungstate and potassium phosphotungstate.

[0053] Among these, an inorganic acid and a salt thereof, and an acidic metal alkoxide are preferred as the oxide source, sulfuric acid, ammonium sulfate, ammonium tungstate, diammonium hydrogen phosphate and tetraethyl orthosilicate are more preferred, and sulfuric acid is further preferred, from the standpoint of the enhancement of the dehydration reaction rate. The use of the catalyst may rapidly progress the dehydration reaction of the alcohol and may provide the target olefin with a high yield.

Ex. 1044, ¶¶51-53.

Dr. Taber's Testimony



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

THIRD DECLARATION OF DOUGLASS F. TABER

Ex. 1046 (Third Taber Declaration)

A. Motivation to Combine

73. In my opinion, a POSITA would refer to both De Bruyn and Takamura as both references address the dehydration of 1-alcohols to the 1-alkene at high conversion and regioselectivity. Ex. 1044 at [0008] and Ex 1008 at [0035]. Further, De Bruyn states that the process to provide the desired dehydration are well known, and not explained in great detail. Ex. 1008 at [0037]. Accordingly, a POSITA would look to Takamura as one of the references that provide further details about how to implement the promoted alumina catalyst discussed by De Bruyn. For example, a POSITA wanting to prepare the promoted catalyst to enhance the dehydration reaction rate while preventing reduction of surface area and maintaining dispersion of the catalyst, would consult the preparation steps of Takamura to make the catalyst. A POSITA would impregnate, dry, calcine, and

Ex. 1046, p. 32, ¶74.

Dr. Taber's Testimony



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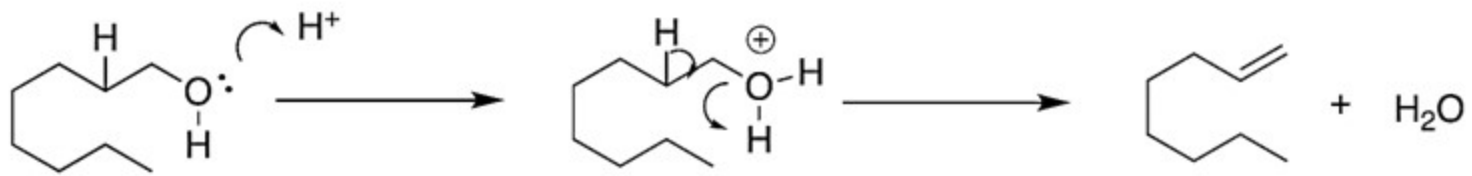
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U.S. Patent No. 10,993,048

THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

37. Acid-catalyzed dehydration of the 1-alcohol occurs when an acid donates a proton to the OH-group of the alcohol. This leads to the protonated alcohol as illustrated below. As the protonated alcohol leaves as water (H_2O) the proton on the adjacent carbon is also lost, resulting in the alkene. Wade p. 312-314. This conversion is represented by the reaction equation below:

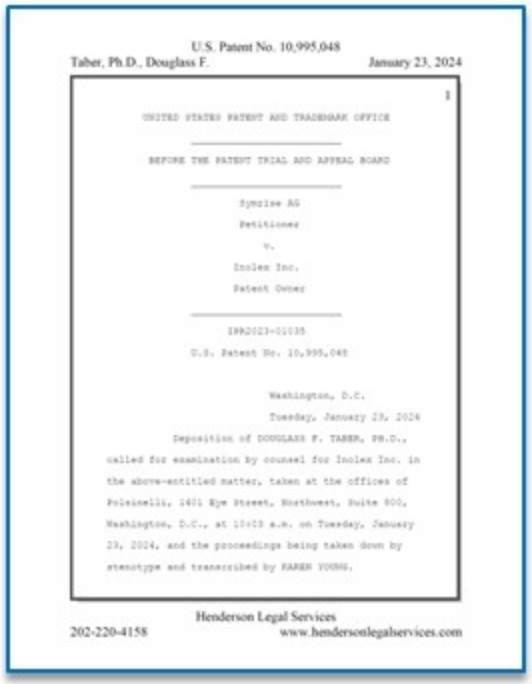


1-Octanol **1-Octene**

Figure 3: Dehydration of 1-octanol to 1-octene under acid catalysis

Ex. 1002, ¶37 (citing Ex. 1030, p. 313-14 (Figure 3)).

Dr. Taber's Testimony



U.S. Patent No. 10,995,048
Taber, Ph.D., Douglass F. January 23, 2024

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Ex. 2005 (Taber Dep. Tr. 01/23/2024)

Q. And then Wade goes through a discussion on page 313 about different mechanisms.

A. Yes.

Q. There's a mechanism 7-4. Is it your understanding that Wade in this section 7-4 is discussing a liquid phase dehydration reaction?

A. He does not comment on this being liquid phase or gas phase.

Q. Okay. So when we talk about step 1, I'm looking at the key mechanism 7-4, protonation of the hydroxyl group, and then step 2, the ionization to a carbocation, and step 3 is deprotonation. What is your understanding of whether this mechanism is referring to a liquid phase or a gas phase reaction or either, or both?

A. This mechanism would hold for both liquid phase and gas phase.

Ex. 2005, p. 24:18 – 25:12 (discussing Ex. 1030).

Dr. Wright's Testimony



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THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

33. Further, in his discussion of the technology of the '048 patent, Dr. Taber cites to a sophomore level organic chemistry textbook, *Wade* (**Ex. 1030**) that discusses the acid-catalyzed solution phase dehydration of alcohols that proceed through a carbocation intermediate followed by deprotonation. **Ex. 1002**, ¶¶35-41 (citing **Ex. 1030**, pp. 313-14). A similar heterogeneous (solid-liquid phase) acid-catalyzed reaction is discussed in *Takamura* (**Ex. 1044**), which utilizes an acid-modified metal catalyst (preferably γ-aluminum) that is treated with an acid. *See* **Ex. 1044**, ¶¶[0037]-[0055]. Both *Wade* and *Takamura* discuss reactions that take place in the liquid phase, optionally with the use of solvents from which the reaction products are isolated after the reaction is complete. **Ex. 1044**, ¶[0074]-[0075], [0080] ("the olefination reaction is preferably performed as a liquid phase reaction."). Dr. Taber's unwavering reliance on such irrelevant material only brings into great question his understanding the fundamentals of heterogeneous base-catalyzed alcohol dehydration (that a POSITA, even with the low-level of experience in this instance, would have).

Ex. 2010, ¶33; Paper 25 at 10.

Dr. Wright's Testimony



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THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

Ex. 1041, p. 164, col. 2 (Scheme 1). Despite having reviewed numerous prior art references such as *Chokkaram*, Dr. Taber's Second and Third Declarations nonetheless cite references such as *Takamura* (**Ex. 1044**), which discusses the catalytic dehydration of 1-alcohols in liquid solution over a solid catalyst. A POSITA would not find references such as *Takamura* (**Ex. 1044**) or *Wade* (**Ex. 1030**) relevant to the technology of the '048 patent. Dr. Taber nonetheless asserts that a POSITA would have been motivated to combine *De Bruyn* with *Takamura* to arrive at the presently claimed invention. *See, e.g.*, **Ex. 1046**, ¶¶59-60, 73.

Ex. 2010, ¶34; Paper 25 at 10.

Dr. Wright's Testimony



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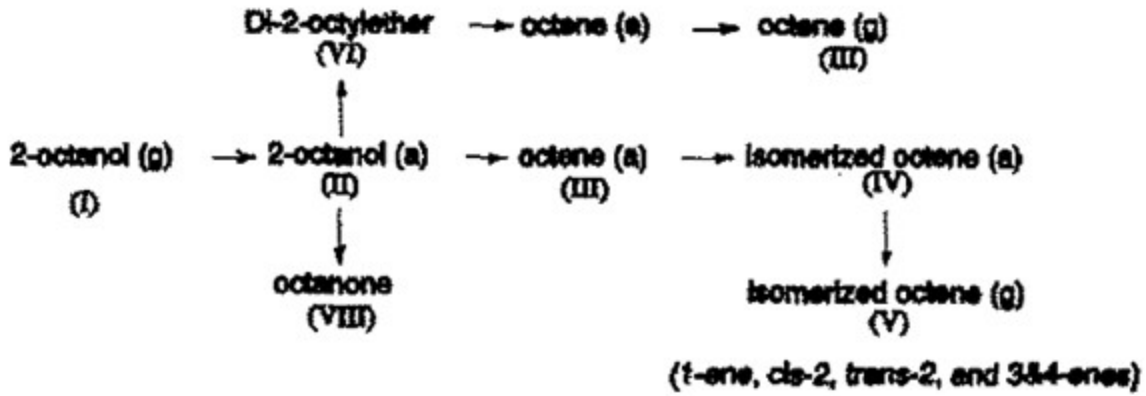
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IPR2013-01035
U.S. Patent No. 10,995,048

THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

34. However, as any POSITA would understand, the catalytic dehydration of 1-alkanols using the γ-alumina catalysts at issue in the '048 patent (and relevant prior art) occurs by flowing the 1-alkanol over the catalyst in the *gas phase*, and molecules react as they adsorb on active base sites of the catalysts. A good summary of the possible reactions that can occur during the catalytic dehydration of 2-octanol is provided in *Chokkaram*:



Scheme 1.

Ex. 1041, p. 164, col. 2 (Scheme 1). Despite having reviewed numerous prior art

Ex. 2010, ¶34 (discussing Ex. 1041, p. 164, col. 2 (Scheme 1)); Paper 25 at 10.

Makgoba - Ex. 1027



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Symrise AG, EX1027
Symrise AG v. Isodex Inc.
IPR2023-01035

Ex. 1027 – *Makgoba*

2006 Publication from SASOL

catalysts. Any of the vast array of available solid acid catalysts can be used for alcohol dehydration [5]. However, γ-aluminas are the most commonly used, especially for the longer chain alcohols (with more than three carbon atoms). This is because catalysts with stronger acidity, such as the silica-aluminas, molecular sieves, zeolites or resin catalysts can promote double-bond shift, skeletal isomerization and other olefin inter-conversion reactions [6,7].

Detailed studies reported in the literature [5,8–11] on the dehydration of alcohols over γ-alumina have shown that the alcohol molecules bond to the alumina surface through hydrogen bonding, where the alcohol and the alumina surface have complimentary functional groups, each being both a proton donor and a proton acceptor. In this proposed mechanism, a proton is transferred from a surface hydroxyl group to the alcohol oxygen, facilitating the transfer of a proton on the neighbouring carbon to a basic oxygen atom on the surface. An olefinic product then desorbs and the water molecule remains adsorbed on the surface and acts as a reaction inhibitor [12]. From these studies, however, there is also

Ex. 1027, 145 (Col. 2)

Dr. Wright's Testimony



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THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

80. A POSITA reviewing *De Bruyn* would understand that promoted γ-alumina catalysts were known—for example, $CaO \cdot Al_2O_3$, $Ca_2O_3 \cdot Al_2O_3$—but nothing in *De Bruyn* teaches or suggests which catalyst is preferred or calcination at the specific temperature range of about 420 °C to about 480 °C (**Ex. 1008**, ¶ [0037].) Thus, *De Bruyn* provides no teaching or suggestion as to which catalyst is preferred, and whether the preferred catalyst would be chemically modified or how and if it was calcined.

Ex. 2010, ¶80 (discussing Ex. 1008).

Dr. Taber's Testimony



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THIRD DECLARATION OF DOUGLASS F. TABER

Ex. 1046 (Third Taber Declaration)

A. Motivation to Combine

73. In my opinion, a POSITA would refer to both De Bruyn and Takamura as both references address the dehydration of 1-alcohols to the 1-alkene at high conversion and regioselectivity. Ex. 1044 at [0008] and Ex 1008 at [0035]. Further, De Bruyn states that the process to provide the desired dehydration are well known, and not explained in great detail. Ex. 1008 at [0037]. Accordingly, a POSITA would look to Takamura as one of the references that provide further details about how to implement the promoted alumina catalyst discussed by De Bruyn. For example, a POSITA wanting to prepare the promoted catalyst to enhance the dehydration reaction rate while preventing reduction of surface area and maintaining dispersion of the catalyst, would consult the preparation steps of Takamura to make the catalyst. A POSITA would impregnate, dry, calcine, and then use the catalyst to dehydrate the alcohol and achieve high conversion and regioselectivity. Ex. 1044 at [0056]-[0065]. Further Takamura teaches that the

Ex. 1046, p. 31, ¶73; Paper 25 at 10.

Dr. Wright's Testimony



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85. The acidic γ-alumina catalysts of *Takamura* stand in stark contrast to the preferred low-acidity catalyst supports such as alumina, silica, titania, or zirconia utilized by *De Bruyn*. **Ex. 1008**, ¶[0037]. Therefore, a POSITA would see no logical relationship between the regioselective dehydration catalysts discussed in *De Bruyn*, and the acidic catalysts of *Takamura*. Not only is there no motivation to combine these references, Dr. Taber provides no specific details about how this would be done.

Ex. 2010, p. 48, ¶85; Paper 25 at 10.

Dr. Wright's Testimony



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102. A POSITA would have had no reason to combine *De Bruyn* with *Takamura*, because a POSITA would not view *Takamura* as relevant to the process described in *De Bruyn*. First, *De Bruyn* discloses a gas phase reaction that utilizes commercially available γ-alumina catalysts (e.g., EuroSupport ESM110®, ¶¶ [0064] and [0171] and Pural® KR1, ¶[0066]). While Dr. Taber argues that *De Bruyn*'s mention of a calcium promoted γ-alumina catalyst (at **Ex. 1008**, ¶ [0037]) would have motivated a POSITA to modify a commercially available γ-alumina catalyst such as ESM110, it is entirely unclear how a POSITA would have done so in view of the teachings of *Takamura*. First, if Dr. Taber is correct that a POSITA would read *De Bruyn* as already providing a 1-alkene regioselectivity of greater than 95%, then a POSITA would have no reason whatsoever to further modify a commercially available γ-alumina catalyst. Second, Dr. Taber provides

Ex. 2010, p. 58, ¶102; Paper 25 at 10.

Dr. Wright's Testimony



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modify a commercially available γ-alumina catalyst. Second, Dr. Taber provides no scientific rationale how the preparation conditions utilized for an acid-modified γ-alumina catalyst would be modified to inform the preparation of a calcium-promoted γ-alumina catalyst (which primarily contains basic surface sites). Indeed, Dr. Taber provides no discussion, let alone any scientific rationale as to why the concentration of *acidic sites* on a γ-alumina catalyst would useful information to a POSITA in choosing to use a calcium-promoted γ-alumina catalyst (having *basic sites* and in which *acidic sites are preferably not present*), let alone what concentration of basic sites should be present on a calcium-promoted catalyst.

Ex. 2010, pp. 58-59, ¶102; Paper 25 at 10.

Dr. Wright's Testimony



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Third, Dr. Taber fails to explain why a POSITA would be motivated to use the calcination process of *Takamura* (which is specifically for an acid-modified γ-alumina catalyst) to calcine an entirely different chemical species (that is modified with a calcium promoter) with any reasonable expectation of success. Fourth, Dr.

Ex. 2010, p. 59, ¶102; Paper 25 at 10.

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with a calcium promoter) with any reasonable expectation of success. Fourth, Dr. Taber fails to explain why a POSITA would selectively use the calcination conditions of *Takamura* (which are specific to acidic γ-alumina catalysts), but not modify the teachings of *De Bruyn* by also using the acidic chemical treatments that are taught by *Takamura*. Indeed, Dr. Taber's Third Declaration is wholly silent as to what, if any, similarities (or differences) between the preferred sulfur oxide (i.e., sulfate) dopants of *Takamura* and the claimed calcium oxide γ-alumina oxides would have motivated a POSITA to use the calcination conditions of *Takamura*, let alone with any reasonable expectation of success.

Ex. 2010, p. 59, ¶102; Paper 25 at 10.

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103. Nor is this a scenario in which it would be "obvious to try" modifying the commercially available γ-alumina catalyst of *De Bruyn* with the teaching of *Takamura* because the latter does not provide a finite number of identified, predictable solutions to the problems of catalyst modification, or methods of improving conversion while maintaining a high 1-alkene regioselectivity.

104. And to the extent that *Takamura* does so, a POSITA would have modified *De Bruyn* with the entire teaching of *Takamura*—including both acid modification and calcination. It is only through the use of impermissible hindsight that a POSITA would cherry pick the calcination conditions of *Takamura* without also applying *Takamura*'s teaching to use an acidic γ-alumina catalyst.

Ex. 2010, p. 60, ¶¶ 103-104; Paper 25 at 10.

Dr. Wright's Testimony



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105. Dr. Taber argues that "Takamura explains to a POSITA that the aluminum oxide should support between 1-3% of the support element (e.g., the calcium of De Bruyn) to enhance the dehydration rate while suppressing side reactions." **Ex. 1046**, ¶74 (citing **Ex. 1044**, ¶¶[0054]). I disagree. *Takamura* is entirely silent as to calcium oxide dopants, let alone their preferred concentration. A POSITA would therefore have no expectation that the level of acidic dopant in Takamura would have any relevance to the amount of calcium-promoted sites on a catalyst. Further, with respect to *Takamura*'s calcination temperatures as providing "enhancement of the dehydration reaction through the provision of a sufficient surface area maintenance of the degree of dispersion of the catalyst," the disclosure in *Takamura* that Dr. Taber points to concerns the preparation of the aluminum oxide support itself, prior to reaction with a dopant. *See* **Ex. 1046** (citing **Ex. 1044**, ¶[0057] ("The *aluminum oxide as the support can be obtained*, for example, by a precipitation method, a sol-gel method or an alkoxide method, *and the aluminum oxide used is preferably calcined* … .") and ¶[0063] (which only includes a discussion of calcination time, not temperature)). I note that *Takamura* at ¶[0062]

Ex. 2010, p. 60-61, ¶ 105; Paper 25 at 10.

Takamura – Ex. 1044


EP 2 990 395 A1

EUROPEAN PATENT APPLICATION

METHOD FOR PRODUCING OLEFIN

EP 2 990 395 A1

Ex. 1044 – Takamura

European Patent filed by Kao Corp. in 2014

Preparation Method of Oxide-supported Solid Catalyst

[0056] The oxide-supported solid catalyst may be prepared by a method of evaporation to dryness, an adsorption method, an equilibrium adsorption method, a pore filling method, a spraying method, a precipitation method or the like.

[0057] Specific examples of the preparation method include a method, in which an aqueous suspension liquid or a water-containing solid of aluminum oxide, the oxide of the element, and an aqueous solvent are mixed to prepare an impregnated product, and the resulting impregnated product is dried and calcined. The aluminum oxide as the support can be obtained, for example, by a precipitation method, a sol-gel method or an alkoxide method, and the aluminum oxide used is preferably calcined at a temperature of 400°C or more, and more preferably 450°C or more, from the standpoint of the enhancement of the dehydration reaction rate through the sufficient crystallization of aluminum oxide, and is preferably calcined at a temperature of 900°C or less, more preferably 800°C or less, further preferably 700°C or less, still further preferably 600°C or less, and still further preferably 550°C or less, from the standpoint of the enhancement of the dehydration reaction rate through the provision of the sufficient surface area. The aluminum oxide used as the support in the present invention is preferably γ-alumina from the standpoint of the catalytic activity, i.e., the enhancement of the dehydration reaction rate.

[0058] The solvent or the aqueous solvent used in the aqueous suspension liquid is preferably water from the standpoint of the availability and the safety in the preparation process of the catalyst.

Ex. 1044, ¶¶56-58.

Dr. Wright's Testimony



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106. What is more, Dr. Taber provides no discussion as to why a POSITA reviewing *De Bruyn* in view of *Takamura* would be motivated to use a modified γ-alumina catalyst, such as a calcium-promoted catalyst. Indeed, the relevant experiments described by *De Bruyn* disclose a 1-alkene regioselectivity of 97.5% (Example 3), 96.5% (Example 4, Step 5), and 97% to 98% (Example 5, Step 4). *See* **Ex. 1008**, ¶ [0053], ¶¶ [0067]-[0068], Table 10; ¶¶ [0080]-[0083], Figure 2, Table 14. On cross examination, Dr. Taber testified that a POSITA would only look to other prior art or attempt to create a modified γ-alumina catalyst if he/she could not obtain a commercially available catalyst from a company such as SASOL. **Ex. 2007**, p. 16, l. 13 – p. 17, l. 12. As any POSITA working in the field of heterogeneous catalysis would know, there are numerous sources of commercially available catalysts—from SASOL as well as many other companies.

Ex. 2010, p. 62, ¶ 106; Paper 25 at 10.

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108. It is only through hindsight that a POSITA reviewing *De Bruyn* would land on *Takamura* as a reference having any relevance to preparing base-treated or promoted alumina catalysts suitable for gas phase catalytic dehydration of bio-1-alkenes. That is, *Takamura*'s disclosure of acid-treatment of an alumina catalyst is entirely at odds with *De Bruyn*'s disclosure that low-acidic catalysts are preferred. Entirely ignoring this dichotomy, Dr. Taber nonetheless concludes that a POSITA would have relied on *Takamura*'s teachings concerning the preferred levels of acidic oxide sites on an alumina catalyst useful in a heterogeneous solid-liquid phase reaction in order to modify a calcium promoted catalyst of *De Bruyn* for use in a solid-gas phase reaction.

Ex. 2010, p. 62, ¶ 106; Paper 25 at 10-11.

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109. In sum, a POSITA would not have combined *De Bruyn* with *Takamura*, and even if such a combination were made, *Takamura* does not remedy any of the deficiencies of *De Bruyn*. *Takamura* would not have informed a POSITA on the appropriate levels of calcium promoter to be used, or at what temperature a calcium-promoted γ-alumina catalyst should be calcined, let alone at a temperature of about 420 °C to about 480 °C. Thus, the combination of *De Bruyn* in view of *Takamura* provides 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, and in view of any relevant secondary indicia of non-obviousness discuss *infra* in Sections VIII.B.2-4, in my opinion substitute claim 29 is patentable over *De Bruyn* in view of *Takamura*.

Ex. 2010, p. 65, ¶ 109; Paper 25 at 10.

Petitioners' Ground III – § 103



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OPPOSITION TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND



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SUR-REPLY TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND

(Paper 20 at 5, 11-15; Paper 29 at 1-5.)

- Substitute claims 29-44 are allegedly obvious over PCT Pub No. WO 2015/130451 (Ex. 1039, *"Wright '451"*)

Paper 20 at 5, 11-15; Paper 29 at 1-5.

Wright '451 – Ex. 1039



Ex. 1039 – Wright '451

BRIEF SUMMARY OF THE INVENTION

[13] A catalyst system and process are provided that can convert a feed of terminal alcohol containing from about 0.1 wt% to about 90 wt% water to a product stream containing the respective terminal olefin and water. In some embodiments, the terminal olefin is produced with about 90% to about 99% regiochemical selectivity. In some embodiments, a single pass over the catalyst system affords a chemical conversion of greater than about 95%. In some embodiments, the terminal olefin is produced employing WHSV values greater than 1 hr^{-1}, *e.g.*, greater than 1 and less than 10 h^{-1}, or about 1.01 hr^{-1} to about 10 hr^{-1}. In some embodiments of the methods disclosed herein, at least one solid phase catalyst is maintained at a temperature of about 220 °C to about 440 °C, with the feed a solution containing terminal alcohol and water at a pressure of about 1 psig to about 1000 psig to produce a 1-alkene in high regioselectivity at high terminal alcohol conversion. In one embodiment, bio-*n*-butanol that is obtained from a fermentation broth (*e.g.*, after removal of minor acidic contaminants and addition of water) can be dehydrated to bio-1-butene in a yield of greater than about 90%, or greater than 95%, or greater than 98%.

Ex. 1039, p. 5, ¶13.

Wright '451 – Ex. 1039



Ex. 1039 – Wright '451

[15] Promoter (*e.g.* Group 2 metal) modified and heat-treated (*e.g.*, calcined) catalysts can be further modified by treatment with at least one organosilane to create new catalysts that are especially useful for the dehydration of 1-alcohols mixed with high wt% of steam. The promoter-organosilane-modified alumina catalysts afford high regioselectivity in forming 1-alkene and can perform for extended periods of time on stream with little loss in regioselectivity.

Ex. 1039, p. 5, ¶15.

Ex. 1039 at ¶[15]. Thus, *Wright '451* teaches that silanized γ-alumina catalysts provided longer time-on-stream.

Ex. 2010, pp. 49-50, ¶89.

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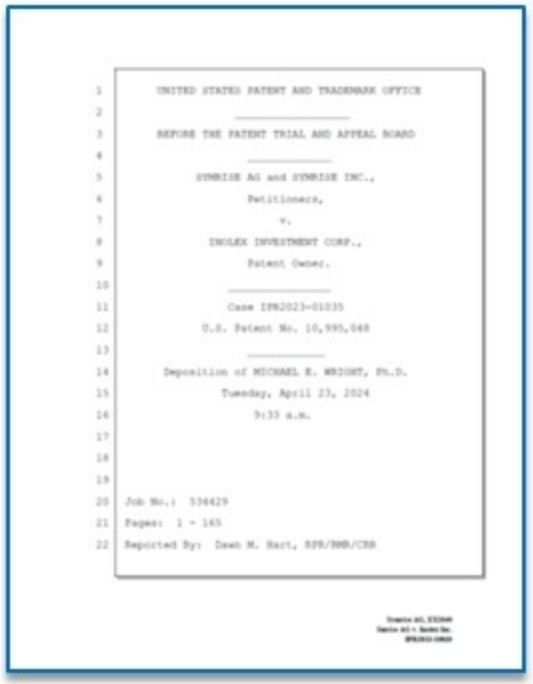
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pp. 23-24. I stand by my testimony that a POSITA would view *Wright '451* and *Wright '788* as the closest prior art to the substitute claims because both teach the considerable benefit of using silanized γ-alumina catalysts to improve time-on-stream, which is reflected in the specification of the '048 patent. What is more, with respect to *Wright '451* specifically, I reiterate my oral and written testimony that *Wright '451* discloses general parameters and specific examples that work very well for 1-butanol, but just don't work for 1-octanol. Specifically, at my

Ex. 2010, p. 54, ¶97.

Dr. Wright's Testimony



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Deposition of MICHAEL E. WRIGHT, Ph.D.
Tuesday, April 23, 2024
9:33 a.m.

Job No.: 534429
Pages: 1 - 165
Reported By: Dawn M. Hart, RPR/RMR/CSR

Ex. 1040 – Wright Dep. Tr. 04/23/2024

Q So if we were not worried about time-on-stream, is there any advantage to including the silanization?

* * *

I have to admit I sort of looked at this. I knew this was coming. It's no surprise. This has been out in the literature. You can find it on Google. So I knew this would come up. Well, gee, this has already been invented. Well, it really hasn't because it gives you specifications which just don't work. And you can't operate in that range of 200 to 600 degrees, and the amount of loading of calcium is different, and so on, so on.

I'd have to look back, but -- we explored different calcium loadings, but I'm not sure, I have to be careful, I -- I'd have to take the Inolex patent and look at this exactly and see if we loaded it exactly the same calcium loading, but I don't think so. I think Inolex was a bit higher.

Ex. 1040, p. 130:4 – 131:10 (discussing Ex. 1039).

U.S. Patent No. 10,995,048



Ex. 1001 – The '048 Patent

Bio-1-butanol is known as a source to make biofuels and provides a pathway for converting straight-chain, linear, primary four-carbon alcohols to bio-1-alkenes at very high yield to improve biofuel production and can also be used, for example in production of eco-friendly tires. In this method, bio-1-alcohols are dehydrated to bio-1-alkenes with high selectivity and chemical yield. The resulting bio-1-alkenes are useful in preparing high flashpoint diesel and jet biofuels useful for civilian and military applications. The bio-1-butanol is dehydrated using a solid phase dehydration catalyst that includes an inorganic support, such as γ-alumina or zinc aluminate ($ZnAl_2O_4$), and that has been treated/modified with a basic aqueous solution. The support may be further treated with an organosilane diluted in at least one hydrocarbon solvent. The process can convert bio-1-butanol from a fermentation process that contains 0.1 to about 90 wt % water and produces a corresponding 1-alkene at a 92-99% regioselectivity, with only a single pass over the solid-phase catalyst, produing a reaction conversion of greater than 95% and a chemical yield of greater than 90% of the bio-1-butene. However, even with such a result for bio-1-butene, it is noted that the conversion of 1-octanol to 1-octene is not simply a homolog of a conversion of 1-butanol to bio-1-butene. In fact, comparative examples described herein below, illustrated that silanization of the catalyst leads to a loss of regioselectivity for the dehydration chemistry of 1-octanol.

Ex. 1001, Col. 7:19-45.

Ex. 2004



Ex. 2004

The silanized γ-alumina catalyst was loaded into a 75 mL reactor, heated to a temperature of 325-335 °C. Bio-1-octanol was flowed into the reactor and the products were collected. A series of runs were conducted in which the bio-l-octanol pump rate was adjusted to optimize conversion and/or 1-octene regioselectivity. The pump rate, temperature, conversion, and 1-octene regioselectivity for the 16 runs are summarized in the following Table:

Run	Pump rate (mL/min)	Conversion (%)	Regioselectivity (%)
1	0.10	98	61
2	0.20	97	73
3	0.50	95	85
4	0.50	93	89
5	0.52	89	92
6	0.52	87	90
7	0.52	85	94
8	0.52	62	97
9	0.55	76	96
10	0.62	44	98
11	0.62	43	98
12	0.75	33	98
13	0.75	32	98
14	0.75	29	98
15	1.0	63	97
16	2.0	11	97

Ex. 2004.

Dr. Wright's Testimony



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conversion." **Ex. 1039** at ¶[14]. Having led this work, I can unequivocally state that, as reflected in the Examples of *Wright '451*, 1-butanol is the only alcohol that was dehydrated, and no attempts were made to dehydrate longer chain alcohols using the catalysts described in *Wright '451*.

Ex. 2010, p. 49, ¶88.

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90. After modification with a Ca^{+2} compound, *Wright '451* discloses that the modified γ-alumina catalysts

> [C]an be further heated … to provide drying or calcination of the catalyst. The drying or calcination can be carried out in vessel **203** or **205** at temperatures of about 100 °C to about 900 °C, or about 200 °C to 600 °C for a time period of about 1 h to about 8 h, or about 2 h to 6 h.

Ex. 1039 at ¶[67]. Further, *Wright '451* discloses Examples 1-26 in which drying and/or calcination of various catalysts is performed at 120 °C, 200 °C, 500 °C, 550 °C, and/or 600 °C for various amounts of time. **Ex. 1039** at pp. 24-33 (Examples 2-4, 8-24).

Ex. 2010, p. 50, ¶90 (discussing Ex. 1039 at pp. 24-33).

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111. I disagree with Dr. Taber's conclusions that *Wright '451* teaches or suggests each and every limitation of substitute claim 29. **Ex. 1046**, ¶¶62-66. Further, contrary to Dr. Taber's assertions, the claimed calcination temperature range of "about 420 °C to about 480 °C" for the specifically claimed "a calcium-promoted γ-alumina catalyst" provides unexpected results. Ex. 1046, ¶¶51-61.

112. *Wright '451* also discloses that calcium-promoted γ-alumina catalysts calcined at 500 °C, 550 °C, or 600 °C dehydrated 1-butanol to provide butene with an alcohol conversion of greater than 95% and a 1-butene regioselectivity of 95% to about 99%. *See* **Ex. 1039**, p. 31 (Table 2). I disagree with Dr. Taber's conclusion that a POSITA reviewing *Wright '451* would understand that these results were obtained for conversion of a C6 to C8 1-alcohol to 1-alkene. *See* **Ex. 1046**, ¶¶65-66.

Ex. 2010, p. 66, ¶¶111-112; Paper 25 at 11.

Wright '451 – Ex. 1039



Ex. 1039 – Wright '451

Table 2

Bio-1-butanol Dehydration Results.

	WHSV (h^{-1})	T (°C)	Aging time (h)	1-Butanol/DBE conversion	1-Butene selectivity
Example 6	1.26	380	no	89.4	95.9
			60	42.7	94.6
			120	41.1	93.6
Example 7	1.26	380	no	78.4	96.1
			60	77.3	95.5
			120	78.5	95.1
Example 8	1.26	380	no	82.8	96.9
			60	93.6	95.5
			120	95.1	94.9
Example 9	1.26	380	no	81.0	97.2
			60	92.6	96.1
			120	94.8	95.2
Example 10	1.26	380	no	95.6	95.1
			60	98.4	93.9
			120	98.8	93.2
Example 11	1.26	380	no	81.8	96.1
			60	87.4	95.4
			120	88.9	94.8
Example 12	1.26	360	no	98.8	96.4
Example 13	1.26	360	no	96.8	97.2
Example 14	1.26	360	no	99.0	93.5
Example 15	1.26	380	no	99.9	93.4
Example 16	1.26	380	no	99.9	95.3
Example 17	1.26	380	no	99.3	95.0
Example 18	1.26	380	no	99.4	94.6
			60	99.9	92.2
			120	100.0	91.0
Example 19	1.26	380	no	98.0	95.9
			60	99.6	94.1
			120	99.8	92.6
Example 20	1.26	380	no	93.0	96.2
Example 21	1.26	380	no	90.4	96.4
Example 22	1.26	380	no	95.1	95.5
Example 23	1.26	360	no	85.9	97.5
			60	98.1	95.1
			120	98.4	94.0

Ex. 1039, p. 32, Table 2.

Dr. Wright's Testimony



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

THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

94. In his Second Declaration, Dr. Taber repeatedly pointed to the presence of heptane in the dehydration of bio-1-butanol as a reason a POSITA would have relied on *Wright '451* because "the POSITA would know that adding the heptane to the 1-butanol would drive the water out of the 1-butanol." **Ex. 2007**, p. 141, l. 19 – p. 143, l. 9; p. 145, l. 15 – 147, l. 10; p. 150, ll. 9-14. This rationale was notably dropped from Dr. Taber's Third Declaration. Specifically, the addition of 2 wt-% heptane in *Wright '451* had no impact whatsoever on the miscibility of water in bio-1-butanol, as the heptane was completely miscible with the reactant solution of 88 wt% 1-butanol, 2 wt% heptane, and 10 wt% water. *See* **Ex. 1039**, ¶¶ [0042], [0043], [0045], [0046]. What is more, as noted above, during the dehydration reaction, the reactants are entirely in the vapor-phase as they pass into and through the reactor. A POSITA would understand that any γ-alumina catalyst, whether modified, base-treated, calcium-promoted, silanized, or treated in some other way, will cause no reaction of the heptane, and therefore heptane was present solely as an internal reference standard in order to better quantify the amount of 1-butene (and other by-products) formed by the dehydration reactions using gas chromatography (GC).

Ex. 2010, p. 52, ¶94.

Dr. Wright's Testimony



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IPR2023-01035
U.S. Patent No. 10,993,048

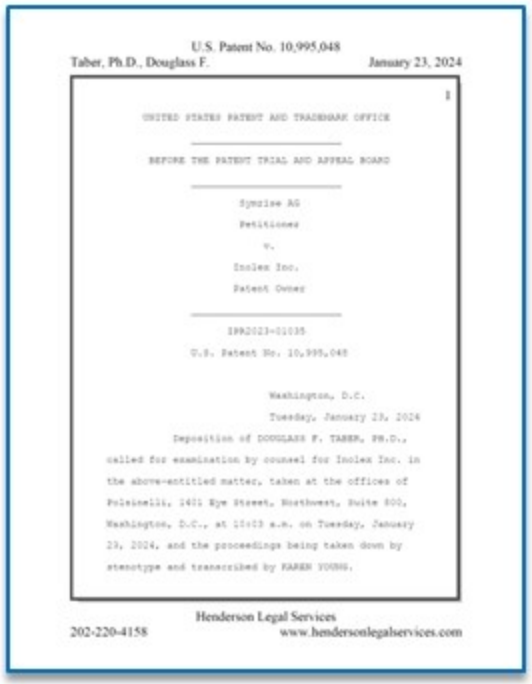
THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

processing of catalyst leading to a truly unexpected result. As I testified at my deposition, there is no way to predict how the treatment of a catalyst with, for example, a particular base, promoter, or other chemical will affect its performance for conversion and regioselectivity of a given substrate without conducting experiments. *See* **Ex. 1040**, p. 43, l. 18 – p. 45, l. 16; p. 139, ll. 1-20. I also made it very clear that 1-butanol does not behave as 1-octanol in heterogeneous catalytic dehydration, nor would a POSITA expect it to. *See* **Ex. 1040**, p. 81, l. 15 – p. 83, l. 10; p. 96, l. 17 – p. 99, l. 2; p. 100, l. 5 – p. 102, l. 3; p. 122, ll. 2-22; p. 124, l. 7 – p. 126, l. 17; p. 128, l. 13 – p. 130, l. 3; p. 133, l. 3 – p. 134, l. 9. What is more, Dr. Taber testified extensively that a POSITA in this field would need to conduct experiments in order to see "what worked." *See* **Ex. 2005**, p. 30, l. 13 – p. 31, l. 10; p. 44, l. 7 – p. 45, l. 11; p. 54, l. 13 – p. 55, l. 7; p. 71, ll. 2-14; 92, l. 4 – p. 93, l. 15; p. 103, l. 10 – p. 104, l. 1.

Ex. 2010, p. 27, ¶47.

Dr. Taber's Testimony


U.S. Patent No. 10,995,048
Taber, Ph.D., Douglass F. January 23, 2024

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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

Syncrise AG
Petitioner
v.
Inolex Inc.
Patent Owner

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

Washington, D.C.
Tuesday, January 23, 2024

Deposition of DOUGLASS F. TABER, PH.D., called for examination by counsel for Inolex Inc. in the above-entitled matter, taken at the offices of Polsinelli, 1401 Eye Street, Northwest, Suite 800, Washington, D.C., at 10:03 a.m. on Tuesday, January 23, 2024, and the proceedings being taken down by stenotype and transcribed by KAREN YOUNG.

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Ex. 2005 (Taber Dep. Tr. 01/23/2024)

Q. Okay. And is it -- is it your understanding that a person of ordinary skill in the art would understand that increasing the temperature of a dehydration reaction leads to a predictably higher yield of the desired alpha-olefin?

A. No, the -- raising the temperature may increase the yield, it may decrease the yield. Depends how much you raise the temperature. Clearly there's -- a lot of experimentation would have to go on to optimize --

Q. Okay.

A. -- the yield of the desired product.

Q. So for a given catalyst, you'd have to actually go and do the experiment to find out if it's going to increase the yield as you increase temperature; is that right?

A. Yes.

Ex. 2005, p. 54:13 – 55:7.

Dr. Wright's Testimony



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SECOND DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Symrise v. Inolex
IPR2023-01035
Inolex Ex. 2006-1

Ex. 2006 – Second Wright Declaration

38. In addition to the level of ordinary skill in the relevant art, I understand that the level of predictability in the art can significantly impact an analysis of whether a particular claimed process is deemed non-obvious over the prior art. Here, the field of organic synthesis using heterogeneous catalysis has been an active area of research for more than 70 years. However, organic synthesis using heterogeneous catalysis remains a highly unpredictable field. As I testified at my deposition, there is no way to predict how the treatment of a catalyst with, for example, a particular base, promoter, or other chemical will affect its performance for conversion and regioselectivity of a given substrate without conducting experiments. *See* **Ex. 1040** at 43:18 – 45:16; 139:1-20.

Ex. 2006 at ¶38 (citing Ex. 1040).

Dr. Wright's Testimony



UNITED STATES PATENT AND TRADEMARK OFFICE

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SYMRISE AG and SYMRISE INC.,
Petitioners,

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Patent Owner.

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

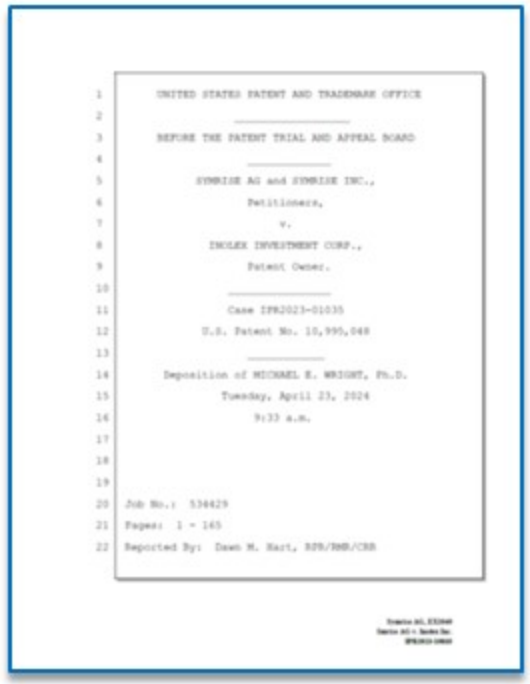
THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

114. In my opinion, *Wright '451*'s disclosure of a calcination temperature range of about 200 °C to 600 °C for a time period of about 1 h to about 8 h, or about 2 h to 6 h (**Ex. 1039**, ¶[67]) with of drying and calcination at 120 °C, 200 °C, 500 °C, 550 °C, and/or 600 °C for times of 30 min to 24 hours (**Ex. 1039** at pp. 24-33 (Examples 2-4, 8-22, and 24)) does not disclose, let alone teach or suggest to a POSITA to specifically perform a calcination of any catalyst "at a temperature of about 420 °C to about 480 °C." A POSITA reading *Wright '451* would have no reason to lower the calcination temperature below 500 °C because the catalysts already provide high conversion, high regioselectivity and long TOS for 1-butanol.

Ex. 2010, p. 67, ¶114; Paper 25 at 11.

Dr. Wright's Testimony re. Wright '451



UNITED STATES PATENT AND TRADEMARK OFFICE

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SYMRISE AG and SYMRISE INC.,
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v.
INCLEX INVESTMENT CORP.,
Patent Owner.

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

Deposition of MICHAEL E. WRIGHT, Ph.D.
Tuesday, April 23, 2024
9:33 a.m.

Job No.: 534429
Pages: 1 - 165
Reported By: Dawn M. Hart, RPR/RMR/CRR

Ex. 1040 – Wright Dep. Tr. 04/23/2024

The drying or calcination can be carried out in vessel 203 or 205 at temperatures of about 100 to about 900 C or about 200 C to 600 C for a period of time of one hours to eight hours or two hours to six hours.

Do you see that?

A Yes, correct.

Q And is that an accurate representation of how to calicine a gamma alumina catalyst after an incipient wetness impregnation process?

A Can you find a combination of those parameters that would, you know, work? In other words, can you be at 600 degrees C and go for four hours and consider that complete calcination? One would have to test it, but that was the range that we called out. Was it physically tested, proved? No.

Q Would a person of ordinary skill in the art understand that within this range they would be able to achieve calcination?

A Well, you've got two variables. So you're talking about could they find a set of variables between 100 degrees and 900 degrees and one hours and eight hours to calicine? Potentially, yes.

Q And then the narrower range, 200 to 600, would they be able to utilize that smaller range to calicine the gamma alumina catalyst?

A Yes, it's possible. Again, you know, did we in this work run a catalyst at 600 degrees for six hours and prove that it was calcined? These are ranges that were taught, or expected to be able to teach, but -- I'm trying to think of the best way to say this.

It depends on scale. And I know these

Ex. 1040, p. 112:3 – 113:15.
Paper 20 at 12.

Dr. Wright's Testimony



UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

SYMRISE AG and SYMRISE INC.,
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IPR2023-01035
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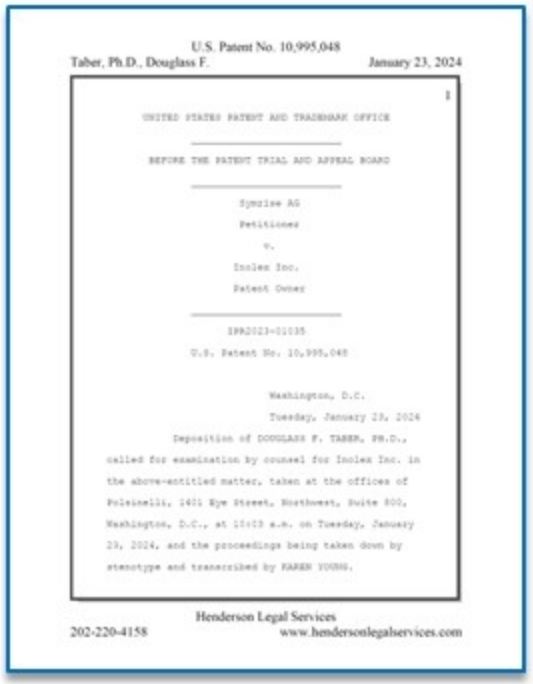
THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

115. In attempting to rebut Inolex's unexpected results associated decreasing the calcination temperature from 500 °C in *Wright '451*'s examples to "about 420 °C to about 480 °C," Dr. Taber first points to broad disclosures in the specification of *Wright '451*. Ex. 1046, ¶52. However, recognizing the gap in *Wright '451*, Dr. Taber then points to other prior art of record such as *Takamura* (**Ex. 1044**), *Chokkaram* (**Ex. 1041**), *Berteau* (**Ex. 1043**), and *Cortez* (**Ex. 1042**) that concern entirely different catalysts than those in *Wright '451* and purportedly would have provided "background" that would motivate a POSITA to modify the teachings of *Wright '451* to calcine a calcium-promoted γ-alumina catalyst at temperatures about 420 °C to about 480 °C and about 440 °C to about 460 °C. **Ex. 2010**, ¶¶55-60. I disagree with Dr. Taber's analysis.

Ex. 2010, p. 68, ¶115; Paper 25 at 11.

Dr. Taber's Testimony


U.S. Patent No. 10,995,048
Taber, Ph.D., Douglass F. January 23, 2024

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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

Synzise AG
Petitioner
v.
Inolex Inc.
Patent Owner

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

Washington, D.C.
Tuesday, January 23, 2024

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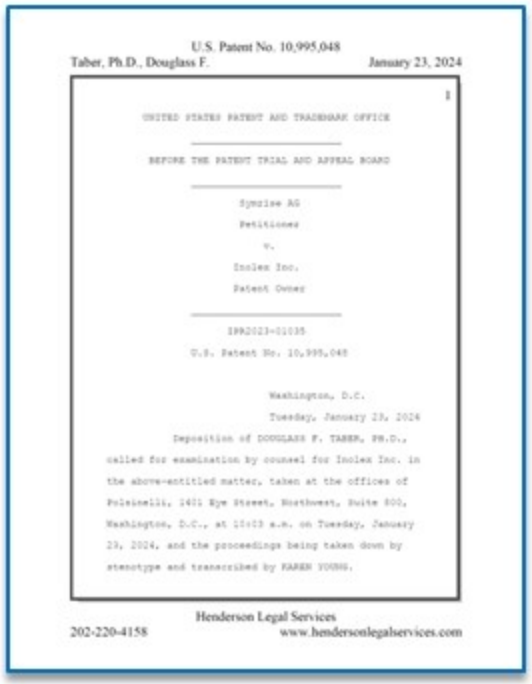
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Ex. 2005 (Taber Dep. Tr. 01/23/2024)

4 Q. Okay. Would you need to do that experiment
5 on any new batch of alumina that you were using to
6 ensure that your time and temperature was sufficient
7 to activate it?
8 **A. Probably.**
9 Q. Would a person of ordinary skill in the art
10 understand that?
11 **A. Yes.**

Ex. 2005, p. 45:4-11.

Dr. Taber's Testimony


U.S. Patent No. 10,995,048
Taber, Ph.D., Douglass F. January 23, 2024

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Ex. 2005 (Taber Dep. Tr. 01/23/2024)

Q. When you look at some of the art around catalysts and alumina catalysts, there's also discussion of a process called calcination. Are you familiar with that?

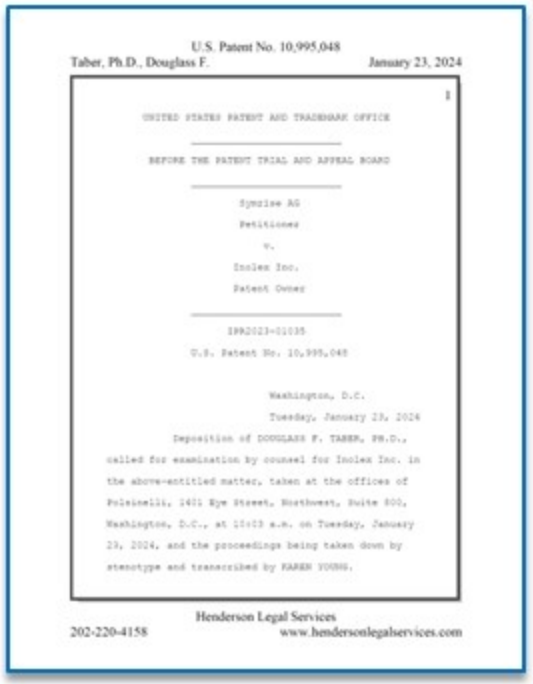
A. Not really. My recollection is that calcination is just heating, but I could be wrong.

Q. Right, so I guess my question is is when you're talking about activation versus calcination, are they the same or different?

A. I really don't know. The -- my sense of things is that calcination is sort of an archaic term, but it probably means something, heating to a certain degree of dryness and that sort of thing.

Ex. 2005, p. 69:13 – 7:3.

Dr. Taber's Testimony



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Taber, Ph.D., Douglass F. January 23, 2024

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Ex. 2005 (Taber Dep. Tr. 01/23/2024)

Q. Would a person of ordinary skill in the art understand that an alumina catalyst is typically calcined prior to being used?

A. **They would certainly understand that it's activated. The -- as I say, my sense of things is that calcined might be a bit of an archaic term, and so they might operate without ever hearing the word "calcined" but still be successful.**

Q. Okay. Would a person of ordinary skill in the art understand that calcination results in a predictable change to the alumina catalyst?

A. **Let us define calcine -- calcination as being heated for some time or temperature, and of course, it would depend on the time and temperature. At some temperature, it would probably be possible to destroy the alumina catalyst, so there would be some optimal time and temperature for the activation.**

Q. Okay, and in order to know what that is for a particular catalyst, would you have to do the experiment?

A. **Yes.**

Ex. 2005, p. 70:16 – 71:14.

Dr. Wright's Testimony re. The '048 Patent



Ex. 1001 – The '048 Patent



Ex. 2010 – Third Wright Declaration

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. 27:1-10 (Table 2); see also Col. 15 (Table 1).

118. Turning back to the experiments at issue, the '048 patent discloses that 1-octanol was dehydrated with γ-alumina catalyst that was first treated with a calcium acetate (i.e., promoter) 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%[4] and 93.1% (per Table 2). **Ex. 1001**, col. 26, l. 10 – col. 27, l. 9 (Examples 1-3). Thus, the '048 patent demonstrates that the higher calcination temperature of 500 °C (as promoted catalyst calcined at the lower temperature of 440 °C exhibited markedly better regioselectivity for 1-octene and also exhibited higher conversion compared to the same catalyst calcined at 500 °C. Nothing in *Wright '451* (or any of the other prior art cited by Dr. Taber) could have predicted this result.

Ex. 2010, p. 70, ¶118; Paper 25 at 11-12.

Dr. Wright's Testimony



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THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

119. Further, a POSITA would view the results provided by the lower calcination temperature of 440 °C as a difference in kind and not one of degree. Indeed, in this field obtaining even a 1-2% improvement in either conversion or 1-alkene regioselectivity can provide significant benefit from the perspective of reactor design and operating costs—for example, by eliminating a recycling stream. Here, 1-octene regioselectivity increased from 93.1% to 96.7% and conversion increased from 95.5% to 97.2%. A POSITA would view these improvements as significant for either variable, but to see improvements in both is very significant and highly unexpected

Ex. 2010, p. 71, ¶119; Paper 25 at 11-12.

Petitioners' Opposition Cites *In re Peterson*



In re Peterson,
315 F.3d 1325 (Fed. Cir. 2003)

[T]he addition of 1% rhenium increased stress rupture life from 34 hours to only 57 hours) is supported by substantial evidence, and there are no data to show that the addition of rhenium in the uppermost portion of the claimed range (i.e., 3% rhenium) would lead to unexpected results. … Example IV includes no rhenium and resulted in an average rupture life of about 148 hours. Example V, which contains 2% rhenium, resulted in an average rupture life of about 275 hours. Example VI, which contains 3% rhenium but less titanium than Examples IV and V, resulted in an average rupture life of only about 130 hours. Thus, the only data for an alloy containing 3% rhenium actually show a decrease in average rupture life as compared with alloys having 0% or 2% rhenium.

315 F.3d at 1331.

Substitute Claims 34, 37, 40, and 43



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SECOND DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Symrise v. Inolex
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Inolex Ex. 2006-1

Ex. 2006 – Second Wright Declaration

	Element(s) of Substitute Claims	Citation to Support in Non-Provisional Priority Application (Ex. 1006), and As-Filed Specification (Ex. 1005)
[34a]	34. The method according to claim 33, wherein prior to the dehydrating, the calcium-promoted catalyst has been calcined at a temperature of about 440 °C to about 460 °C.	**Ex. 1006** (pp. 23-79) at [0035], [0042], [0076], Table 1, [0084], [0123], [0125], Table 2, [0126], claims 21 and 41. **Ex. 1005** (pp. 4-54) at [0035], [0042], [0076], Table 1, [0084], [0123], [0125], Table 2, [0126], claim 19.

Paper 18 at 9, 22-23.

to about 460 °C. Finally, *Wright '451* discloses calcination of a calcium-promoted γ-alumina catalyst at a preferred temperature range of 200 °C to 600 °C, with specific calcination conditions disclosed at 500 °C, 550 °C, and 600 °C. Thus, the closest prior art to the revised substitute claims is *Wright '451*'s disclosure of calcination of a calcium-promoted γ-alumina catalyst at 500 °C.

Ex. 2006, pp. 73-74, ¶130; Paper 25 at 12.

Dr. Wright's Testimony


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THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

120. Lastly, Dr. Taber purports to take issue with these results because they do not show that there is a negative impact from calcination at 500 °C. Ex. 1046, ¶54. But that is not required. Rather, the data show something unexpected compared to the higher calcination temperatures that are used in Wright '451 and many other prior art references. Nor is there any need for a POSITA to be able to make a "blanket determination" based on this result, as Dr. Taber would have it. Ex. 1046, ¶54. Rather, this result is limited to the claimed "calcium-promoted γ-alumina catalyst" that has not been silanized. Finally, Dr. Taber cites no support for his "additional sources of data" that contradict the experiments of the '048 patent and this statement can be ignored.

Ex. 2010, pp. 71-72, ¶120; Paper 25 at 11-12.

Dr. Wright's Testimony re. The '048 Patent



Ex. 1001 – The '048 Patent



Ex. 2010 – Third Wright Declaration

Example 10

In a comparative example, bio-1-octanol was fed into a 75 mL reactor packed with a catalyst prepared as follows: gamma-alumina powder was modified with a sodium hydroxide solution (5 wt-% NaOH) to create a second catalyst, the second catalyst was dried in an oven and then treated with diethoxydiphenylsilane (1 wt-% based on alumina) dissolved in ethanol. The solvent was removed, the catalyst washed with ethanol, and dried in an oven to create a third catalyst. This third catalyst was calcined at 400° C. for 12 h. The catalyst was loaded into a 75 mL reactor, heated to a target temperature of 325° C. and bio-1-octanol feed rates were adjusted to optimize both conversion and regioselectivity.

Ex. 1001, Col. 28:51-67.

122. As set forth in my Second Declaration, the '048 patent also discloses unexpected results over a prior art 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. 1006** at pp. 61-62 of 719; **Ex. 1005** at pp. 42-43 of 236. As I testified at length in my deposition (and as disclosed in the '048 patent), the closest prior art to the catalyst developed by Inolex were the silanized catalysts developed from my research at the U.S. Navy and Cobalt Technologies.

Ex. 2010, p. 72, ¶122; Paper 25 at 11-12.

U.S. Patent No. 10,995,048 – Ex. 1001



Ex. 1001 – The '048 Patent

Example 4

γ-alumina in the form of 1/16" extrudate, Porocel® Cat-Guard® was cut to have length to diameter ratios in the range of 2 to 4. The extrudate was modified with an aqueous solution of calcium acetate. The weight of solution to alumina was 0.55 by weight and the calcium acetate concentration was at level to deliver 1.50% of CaO (after calcination). This catalyst was subjected to calcination in air at 440° C. for a period of 12 h.

Example 5

The 48 g of the catalyst prepared in EXAMPLE 4 was loaded into a 75 mL (Sigma Aldrich Part No. Z173592) reactor, wrapped with heating tape, and covered with fiber-glass insulation. The temperature was controlled by affixing a thermocouple to the outside skin of the reactor, this input was used for a temperature controller. Bio-1-octanol was fed at 0.2 mL/min into the reactor maintained at 315° C. After >1,400 h of TOS, that included regular shut-downs and start-ups, catalyst performance with regard 1) chemical conversion of the bio-1-octanol, 2) product selectivity, 3) and regioselectivity for bio-1-octene was unchanged. A GC chromatogram of the crude product collected from the reactor is shown in FIG. **3** with an octene selectivity of 96% and with chemical yield of bio-1-octene of 94%.

Ex. 1001, Col. 27:10-35.

U.S. Patent No. 10,995,048 – Ex. 1001



United States Patent
Burge et al.

(10) Patent No.: US 10,995,048 B2
(45) Date of Patent: May 4, 2021

(54) NATURAL 1,2-ALKANEDIOLS, COMPOSITIONS HAVING NATURAL 1,2-ALKANEDIOLS AND PROCESSES FOR MAKING THE SAME

(71) Applicant: Indes Investment Corporation, Wilmington, DE (US)

(72) Inventors: Bruce V. Burge, Mullica Hill, NJ (US); Michael E. Wright, Hurricane, UT (US); Gary B. Mosser, Tabernacle, NJ (US); Michael J. Ferola, Delle Mead, NJ (US)

(73) Assignee: INDES INVESTMENT CORPORATION, Wilmington, DE (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 16/682,699

(22) Filed: Nov. 13, 2019

(65) Prior Publication Data

US 2020/0339595 A1 Oct. 29, 2020

Related U.S. Application Data

(63) Continuation of application No. 16/262,902, filed on Jan. 30, 2019.

(60) Provisional application No. 62/623,995, filed on Jan. 30, 2018.

(51) Int. Cl.
C07C 29/04 (2006.01)
C07C 33/20 (2006.01)
C07C 29/60 (2006.01)
B01J 19/06 (2006.01)
B01J 37/08 (2006.01)
B01J 21/04 (2006.01)
C12P 7/20 (2006.01)
C07C 1/24 (2006.01)
C07C 29/48 (2006.01)
C07C 45/82 (2006.01)
C07C 45/85 (2006.01)
C07B 41/02 (2006.01)
C07B 43/00 (2006.01)
B01J 2/19 (2006.01)
B01J 41/02 (2006.01)
B01J 4/04 (2006.01)
B01J 3/00 (2006.01)
B01J 21/00 (2006.01)
B01J 19/00 (2006.01)
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(52) U.S. Cl.
CPC C07C 29/04 (2013.01); B01J 21/04 (2013.01); B01J 19/06 (2013.01); B01J 37/08 (2013.01); C07C 1/24 (2013.01); C07C 33/20 (2013.01); C07C 45/82 (2013.01); C07C 45/85 (2013.01); C07C 29/48 (2013.01); C07C 29/60 (2013.01)

(58) Field of Classification Search
None
See application file for complete search history.

(56) References Cited

U.S. PATENT DOCUMENTS

FOREIGN PATENT DOCUMENTS

OTHER PUBLICATIONS

(Continued)

Primary Examiner — Natasha Hellman
(74) Attorney, Agent, or Firm — Cozen O'Connor

(57) ABSTRACT

Syncrude AG, EX1001
Syncrude AG v. Indes Inc.
IPR2023-01035

28 Claims, 7 Drawing Sheets

Ex. 1001 – The '048 Patent

Example 11

Petro-1-hexanol was fed into a 75 mL reactor packed with the catalyst prepared in EXAMPLE 4. The catalyst used in this example had greater than 1,400 h of TOS used for bio-1-octanol dehydration to bio-1-octene. For this series of dehydration reaction examples, the temperature was varied from 330° C. to 370° C. and the feed rate was adjusted in order to achieve between 1 and 2% of hexanol passing through the reactor. The results for the hexanol dehydration are shown 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. 29:1-21 (Table 5).

Dr. Wright's Testimony re. The '048 Patent



Ex. 1001 – The '048 Patent



Ex. 2010 – Third Wright Declaration

124. As discussed in the '048 patent, dehydration reactions conducted at 330 °C and 350 °C provided conversion (to 1-, 2-, and 3-hexenes) of 96.3% and 96.9%, respectively, with a regioselectivity for the formation of 1-hexene of 95.8%[5] and 96.8% (as reported 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). Surprisingly, increasing the reactor temperature to 370 °C reduced conversion (to hexenes) to 93.3% (from 96.9% at 350 °C), while maintaining a regioselectivity of 96.6% (as shown in Table 5, above). This temperature sensitivity was unexpected, particularly in view of prior art calcium-promoted catalysts such as those disclosed in *Wright '451* that provide conversion in excess of 95% at 380 °C. *See* **Ex. 1039**, Figs. 10-12.

Ex. 2010, p. 73, ¶124; Paper 25 at 11-12.

Dr. Wright's Testimony re. The '048 Patent



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126. As discussed in my Second Declaration, a POSITA would also view this result as unexpected based on *Wright '451*'s teaching that the lowest 1-butanol conversion is always at 340 °C and *conversion increases with temperature*." **Ex. 1039**, ¶¶ [13], [42]. Nothing in *Wright '451* would have taught or suggested to a POSITA that conversion of 1-hexanol would decrease at 370 °C. A POSITA would view increase in conversion at lower temperatures as a difference in kind and not one of degree. As discussed above, even a 1-2% improvement in conversion can provide significant design and operational benefits. Here, 1-hexene conversion increased from 93.3% to 96.9% while maintaining regioselectivity. A POSITA would view this improvement as significant.

Ex. 2010, pp. 74, ¶126; Paper 25 at 11-12.

Dr. Wright's Testimony re. Long-Felt Need



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4. The substitute claims satisfy a long-felt need and show success where others have failed

128. There can be no dispute that the dehydration of 1-alcohols has been an area of active research for more than 70 years, and in the current age of sustainable chemistry and processing technology there has always been a need for improved methods that reduce energy requirements and waste emissions (such as CO2 and used catalyst). To my knowledge, Inolex is the first company to have developed a specific catalyst and process suitable for dehydrating C_6 to C_{10} bio-1-alcohols with a conversion of at least 95% and a bio-1-alkene regioselectivity of 95% to about 99% from a single-pass reaction after at least 20 hours time-on-stream.

Ex. 2010, p. 75, ¶128; Paper 25 at 12.

Dr. Wright's Testimony re. Long-Felt Need



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129. Others in the past have achieved higher overall "net conversion" of alcohol by isolating the unreacted alcohol and ethers (i.e. a purification step(s)) and reintroducing (and heating) those materials back into the reactor (i.e., using a recycle stream). *See, e.g.,* **Ex. 1008**, ¶ [0053] (Example 3). However, this comes with consequences, some quite severe: An increase of catalyst amount is needed, much higher energy inputs are required, strong potential of decreased catalyst lifetime (due to impurities in recycle stream), increased equipment cost and added failure points in the process, and an overall more complicated process to monitor and maintain a continuous 24/7 process. And even after recycling the unreacted starting materials (and by-products such as ethers), *De Bruyn* claims to achieve a 94% conversion of 1-hexanol to alkenes. **Ex. 1008**, ¶ [0053],

130. Thus, the ability of a catalyst to maintain regioselective dehydrogenation of 1-alcohols to 1-alkenes at high and steady conversion rates *and* for extended duration of time-on-stream, was and is a crucial consideration for any commercial and sustainable bio-1-alcohol dehydration process.

Ex. 2010, pp. 75-76, ¶¶129-130; Paper 25 at 12.

Dr. Wright's Testimony re. Long-Felt Need



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IPR2013-01005
U.S. Patent No. 10,995,048

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Ex. 2010 – Third Wright Declaration

131. While my prior work, as described for example, in *Wright '788* and *Wright '451* provided processes for the conversion of 1-butanol to 1-butene, it cannot be overstated that 1-butanol does not behave as 1-octanol in heterogeneous catalytic dehydration, nor would a POSITA expect it to. *See* **Ex. 1040**, p. 81, l. 15 – p. 83, l. 10; p. 96, l. 17 – p. 99, l. 2; p. 100, l. 5 – p. 102, l. 3; p. 122, ll. 2-22; p. 124, l. 7 – p. 126, l. 17; p. 128, l. 13 – p. 130, l. 3; p. 133, l. 3 – p. 134, l. 9.

Therefore, in my opinion the substitute claim satisfy a long-felt need.

Ex. 2010, p. 76, ¶131; Paper 25 at 12.

Dr. Wright's Testimony re. Failure of Others



Ex. 2008 – *Bugdahn*



Ex. 2010 – Third Wright Declaration

(51) International Patent Classification:
C07C 1/20 (2006.01) *C07C 11/02* (2006.01)
C07C 1/24 (2006.01)

(21) International Application Number:
PCT/EP2020/085177

(22) International Filing Date:
09 December 2020 (09.12.2020)

(71) Applicant: SYMRISE AG [DE/DE]; Mühlenfeldstr. 1, 37603 Holzminden (DE).

(57) Abstract: The present invention relates to a process for the efficient, sustainable and gentle preparation of bio-based alkanediols, especially 1,2-alkanediols, at moderate temperatures based on a recycling of side products and non-reacted educts. The present invention also relates to a composition comprising at least two alkanediols obtainable by said process and to the use of said composition in consumer product formulations. In addition, the present invention relates to consumer products as such comprising the inventive composition.

Ex. 2008, Abstract.

132. Further, many other researchers are still very active in this field and are still attempting to develop processes that are competitive with Inolex's. It is noteworthy that Petitioners attempted (and apparently have failed) to develop their own catalytic dehydration process, which, far from achieving anywhere near the high-degree of both conversion and regioselectivity, instead relies on "moderate temperatures" and "recycling of side products and non-reacted educts." *See* *Bugdahn* (**Ex. 2008**), Abstract.

Ex. 2010, pp. 76-77, ¶132; Paper 25 at 12.

Ground V: Estoppel

Petitioners' Ground V – Estoppel



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PATENT OWNER.

IPR2021-01015
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OPPOSITION TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND



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SUR-REPLY TO PATENT OWNER'S REVISED NON-CONTINGENT MOTION TO AMEND

(Paper 20 at 4; Paper 29 at 8-10.)

IX. Patent Owner Estoppel

PO has disclaimed claims 1-7, 9-11, 24-26, 28, 29, 35, and 36 of US Pat. No. 10,882,803, the "Disclaimed Claims". Ex. 1049; Ex 1047, 1048. The Board has entered an adverse judgment against PO based on the disclaimer. PO is "precluded from taking action inconsistent with the adverse judgment". 37 CFR 42.73(d)(3).

Paper 20 at 19.

Petitioners' Opposition Cites *AbbVie v. Mathilda & Terrence Kennedy*



AbbVie Inc. v. Mathilda & Terrence Kennedy Inst. Of Rhematology Trust, 764 F.3d 1366 (Fed. Cir. 2014)

Estoppel applies when a claim is not "patentably distinct" from a finally refused or canceled claim. 37 CFR 42.73(d)(3)(i). The test for patentably distinct looks at the "differences in subject matter between the claims" and then undertakes an "analysis [that] is analogous to an inquiry into the obviousness of a claim under 35 USC §103." *Abbvie Inc. v. Mathilda & Terence Kennedy Inst. of Rheumatology Trust*, 764 F.3d 1366, 1378-79 (Fed. Cir. 2014); *see also Apple Inc. Requester &*

Paper 20 at 20.

To be sure, obviousness is not demonstrated merely by showing that an earlier expiring patent dominates a later expiring patent. Nor do we think that the district court here relied on any such principle. It is well-settled that a narrow species can be non-obvious and patent eligible despite a patent on its genus.

See Eli Lilly & Co. v. Bd. of Regents of Univ. of Wash., 334 F.3d 1264, 1270 (Fed. Cir. 2003); *In re Kaplan*, 789 F.2d 1574, 1577-78 (Fed. Cir. 1986); *In re Sarett*, 327 F.2d 1005, 1014 (CCPA 1964); 3A Donald S. Chisum, Chisum on Patents § 9.03[2][b][ii].

764 F.3d at 1379.

Dr. Wright's Testimony



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Patent Owner.

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U.S. Patent No. 10,993,848

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52. Referring to **Ex. 2011**, I note that claims 1-7, 24-26, 28, 29, 35, and 36 of the '803 patent merely require a process that utilizes "a catalyst"—without any limitation as to what the catalyst is or how it is prepared. Further, claims 9-11 of the '803 patent merely require that "the catalyst is selected from $ZnAl_2O_4$ and γ-alumina catalyst." I readily acknowledge that $ZnAl_2O_4$ and γ-alumina catalysts were known in the prior art, but strongly disagree that the catalysts required by substitute claims 29-44 are patentably indistinct from the generic categories of "$ZnAl_2O_4$ and γ-alumina catalysts," which cover thousands of different possible catalysts that can be, for example, doped or undoped, treated or untreated, calcined or not calcined. Dr. Taber takes an entirely unsupported and baseless view that selecting a catalyst is akin to choosing features on a new car, whereby a chemical treatment process or calcination temperature useful for one specific catalyst in the prior art is readily applicable to other, entirely different catalysts to yield predictable results. *See, e.g.,* **Ex. 1046**, ¶¶57, 59, 60 (alleging without any evidence

Ex. 2010 at 30, ¶52.

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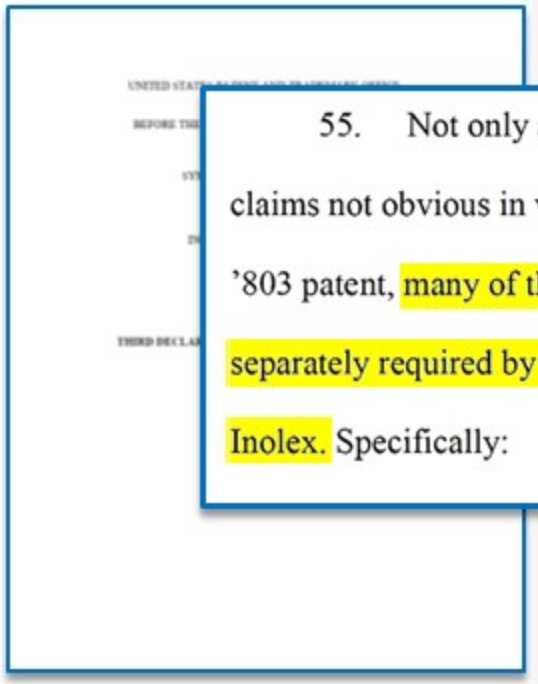
THIRD DECLARATION OF MICHAEL E. WRIGHT, Ph.D.

Ex. 2010 – Third Wright Declaration

53. Substitute claims 29-33, 36, 39, and 42 require "a calcium-promoted γ-alumina catalyst calcined at a temperature of about 420 °C to about 480 °C, wherein the catalyst has not been silanized." In my opinion, nothing in any of the disclaimed claims of the '803 patent teaches or suggests the catalyst required by the substitute claims, as there is no reference to a calcium-promoted γ-alumina catalyst, let alone a calcium-promoted γ-alumina catalyst that has been calcined at a temperature of about 420 °C to about 480 °C. What is more, substitute claims 34, 25, 37, 38, 40, 41, 43, and 44 require that "the calcium-promoted catalyst has been calcined at a temperature of about 440 °C to about 460 °C."

Ex. 2010 at 31, ¶53.

Dr. Wright's Testimony



55. Not only are these process parameters required by the substitute claims not obvious in view of claims 1-7, 9-11, 24-26, 28, 29, 35, and 36 of the '803 patent, many of the process parameters required by the substitute claims are separately required by claims of the '803 patent that were *not disclaimed* by Inolex. Specifically:

Ex. 2010 – Third Wright Declaration

- Claim 15 of the '803 patent requires "wherein the γ-alumina catalyst is treated with a calcium promoter to provide a modified γ-alumina catalyst";
- Claim 16 of the '803 patent (which depends from claim 15) requires "wherein the modified γ-alumina catalyst is calcined to provide a calcined γ-alumina catalyst";
- Claim 18 of the '803 patent (which indirectly depends from claim 16) requires "wherein the calcium promoter is used in an amount of about 1 weight percent to about 2 weight percent based on the weight of CaO determined after calcination";
- Claim 20 of the '803 patent (which indirectly depends from claim 15) requires "wherein the temperature during calcination is about 420° C to about 480° C"; and
- Claim 21 of the '803 patent (which depends from claim 20) requires "wherein the temperature during calcination is about 440° C. to about 460° C."

See **Ex. 2011**.

Ex. 2010 at 32-33, ¶55.

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56. Further, none of claims 1-7, 9-11, 24-26, 28, 29, 35, and 36 of the '803 patent require a continuous flow reactor heated "at a temperature of 240 °C to 360 °C." This limitation of substitute claim 29 is not recited by any claims of the '803 patent but instead only appears in the specification. Similarly, none of claims 1-7, 9-11, 24-26, 28, 29, 35, and 36 of the '803 patent require a continuous flow reactor "heated at a temperature of 315 °C to 350 °C." This limitation of substitute claims 35, 38, 41, and 44 is also not recited by any claims of the '803 patent and only appears in the specification.

Ex. 2010 at 33-34, ¶56.

General Foods v. Studiengesellschaft Kohle mbH



General Foods v. Studiengesellschaft Kohle mbH, 972 F.2d 1272 (Fed. Cir. 1992)

Our precedent makes clear that the disclosure of a patent cited in support of a double patenting rejection cannot be used as though it were prior art, even where the disclosure is found in the claims. *See, e.g., Braat*, 937 F.2d at 594 n. 5, 19 USPQ2dat 1293 n. 5 ("The patent disclosure must not be used as prior art"); …

In re Sutherland, 347 F.2d 1009, 1015, 146 USPQ 485, 491 (CCPA1965) ("Nor is obviousness invariably involved in 'double patenting' rejections. Claims relied on in such [double patenting] rejections often disclose or name the *very thing* being claimed [in the rejected claims]. Furthermore, the words of such claims cannot be treated as 'prior art,' ... but are looked to solely for the purpose of determining what has already been patented. They are not treated as prior art for the simple reason they are no more 'prior art' under the statue than the specification") (citation omitted); *In re Sarett*, 327 F.2d 1005, 1013, 140 USPQ 474, 481 (CCPA1964) ("We are not here concerned with what one skilled in the art would be aware [of] from reading the claims but with what inventions the claims define.")

972 F.2d at 1281-82 (cited in Paper 25 at 3-4).

Dr. Wright's Testimony



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57. Nor have I (or Inolex) admitted that any prior art discloses, let alone teaches or suggests, the claimed calcination temperatures or reactor temperatures used in combination with the catalyst required by substitute claims 29-44. **Ex. 1046**, ¶26. As discussed above, I acknowledge that prior art processes (such as *Chokkaram*) utilized similar calcination temperatures or reactor temperatures *with other catalysts and other reagents*. As I have attempted to explain at length in my three Declarations, the mere presence of a calcination or reactor temperature in the prior art would not provide a POSITA any motivation to utilize those same temperatures for the catalysts required by the substitute claims, let alone with any reasonable expectation of success.

Ex. 2010 at 34, ¶57.

Dr. Wright's Testimony



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58. Dr. Taber attempts to brush aside the fact that the substitute claims also require that the bio-1-alkene is "produced in a conversion of at least 95% and a bio-1-alkene regioselectivity of 95% to about 99% from a single-pass reaction after at least 20 hours time-on-stream" by arguing that disclaimed claims 6 and 7 at least require a single pass. Ex. 1046, ¶22. This completely ignores that a POSITA would have no understanding, let alone any reasonable expectation that disclaimed claims 6 and 7 could achieve the required regioselectivity or conversion in a single pass through the continuous flow reactor.

Ex. 2010 at 34, ¶58.

General Foods v. Studiengesellschaft Kohle mbH



General Foods v. Studiengesellschaft Kohle mbH, 972 F.2d 1272 (Fed. Cir. 1992)

Our precedent makes clear that the disclosure of a patent cited in support of a double patenting rejection cannot be used as though it were prior art, even where the disclosure is found in the claims. *See, e.g., Braat*, 937 F.2d at 594 n. 5, 19 USPQ2dat 1293 n. 5 ("The patent disclosure must not be used as prior art"); …

* * *

In re Sutherland, 347 F.2d 1009, 1015, 146 USPQ 485, 491 (CCPA1965) ("Nor is obviousness invariably involved in 'double patenting' rejections. Claims relied on in such [double patenting] rejections often disclose or name the *very thing* being claimed [in the rejected claims]. Furthermore, the words of such claims cannot be treated as 'prior art,' ... but are looked to solely for the purpose of determining what has already been patented. They are not treated as prior art for the simple reason they are no more 'prior art' under the statue than the specification") (citation omitted); *In re Sarett*, 327 F.2d 1005, 1013, 140 USPQ 474, 481 (CCPA1964) ("We are not here concerned with what one skilled in the art would be aware [of] from reading the claims but with what inventions the claims define.")

972 F.2d at 1281-82 (discussed in Paper 25 at 3-4).

Aspex Eyewear, Inc. v. Zenni Optical Inc.



Aspex Eyewear, Inc. v. Zenni Optical Inc., 713 F.3d 1377 (Fed. Cir. 2013)

Collateral estoppel "precludes a plaintiff from relitigating identical issues by merely switching adversaries" and precludes a plaintiff "from asserting a claim that the plaintiff had previously litigated and lost against another defendant." *Parklane Hosiery Co. v. Shore*, 439 U.S. 322, 329 (1979).

No changed circumstances are here shown, nor any new evidence nor materially different argument that was unavailable to Aspex in the Altair litigation. The Aspex claims now in suit contain the same "retaining mechanism" term that was construed in Altair as requiring rims. This construction is determinative of noninfringement by the Zenni eyewear; it is irrelevant whether the additional claims now in suit contain additional terms that were not previously construed. *See Transocean Offshore Deepwater Drilling, Inc. v. Maersk Contractors USA, Inc.*, 617 F.3d 1296, 1311-12 (Fed. Cir. 2010) (applying collateral estoppel when "the infringement issue in [the second] case is identical to the one in [the first case]," for the "differences in the facts ... do not change the fact that the modified rig does not infringe"); …

713 F.3d at 1381-82 (discussed in Paper 25 at 5).

Ohio Willow Wood Co. v. Alps South, LLC



Ohio Willow Wood Co. v. Alps South LLC, 735 F.3d 1333 (Fed. Cir. 2013)

If the differences between the unadjudicated patent claims and adjudicated patent claims do not materially alter the question of invalidity, collateral estoppel applies. *Bourns, Inc. v. U.S.*, 537 F.2d 486, 493 (Ct. Cl. 1976).

Re-exam claim (found invalid)

1. A tube sock-shaped covering for enclosing an amputation stump, said amputation stump being a residual limb, said covering having an open end for introduction of said residual limb and a closed end opposite said open end, said covering comprising fabric in the shape of a tube sock, said fabric having a coating of a foamed or non-foamed block copolymer and mineral oil gel composition residing on only an interior surface thereof.

Claim at issue

1. A cushion liner for enclosing an amputation stump, said liner comprising a fabric covering having an open end for introduction of said stump and a closed end opposite said open end, said fabric coated seamlessly on only an inside surface thereof with a polymeric cushioning gel that substantially conforms to the shape of said amputation stump when said liner is worn; wherein said liner is configured such that said polymeric cushioning gel is in contact with the skin of said amputation stump when said liner is worn by a user thereof.

735 F.3d at 1342 (discussed in Paper 25 at 5).

Maxlinear, Inc. v. CF Crespe LLC



Maxlinear, Inc. v. CF Crespe LLC, 880 F.3d 1373 (Fed. Cir. 2018)

The preclusive effect of the prior adjudications, and subsequent affirmations, has finally resolved the issue of the unpatentability of independent claims 1 and 17 and dependent claim 20 in this proceeding. Thus, the sole remaining question at issue is whether the dependent claims 4, 6-9, and 21, not addressed in the earlier IPRs, are unpatentable.

* * *

On remand, the Board must consider whether the dependent claims 4, 6-9, and 21 can survive the unpatentability of claims 1 and 17 from which they depend in view of the prior art cited in the '728 IPR. The Board must decide whether the remaining claims present materially different issues that alter the question of patentability, making them patentably distinct from claims 1 and 17.

880 F.3d at 1377-78 (discussed in Paper 25 at 5-6).

Amgen Inc. v. Hoffman-La Roche Ltd.



Amgen Inc. v. Hoffman-La Roche Ltd., 580 F.3d 1340 (Fed. Cir. 2009)

We agree with the district court that claims 1 and 2 of the '868 patent and claims 6-9 of the '698 patent are not invalid for obviousness-type double patenting over claim 27 of the '008 patent. The obviousness-type double patenting analysis entails two steps: (1) construction of the claims in the earlier patent and the claim in the later patent to identify any differences, and (2) determination of whether the differences in subject matter between the claims render the claims patentably distinct. *See Eli Lilly & Co. v. Barr Labs., Inc.*, 251 F.3d 955, 968 (Fed. Cir. 2001).

* * *

[The claims at issue] recite processes of producing EPO that involve (a) growing mammalian cells transfected with DNA encoding EPO and (b) isolating from those cells glycosylated EPO having the stated biological properties.

In essence, claim 27 of the '008 patent recites the starting materials necessary to execute the processes recited in the asserted claims of the '868 and '698 patents.

580 F.3d at 1361 (discussed in Paper 29 at 11).

Softview LLC v. Apple Inc.



Softview LLC v. Apple Inc.,
108 F.4th 1366 (Fed. Cir. 2024)

[T]he Board issued a final written decision in the IPR proceeding, finding each of the 18 challenged claims of the '353 patent unpatentable.

* * *

To the extent they differ more than trivially from the canceled claims, the amended claims merely combine limitations from multiple canceled claims.

* * *

Thus, absent strong evidence to the contrary, the PTO's use of the term "patentably distinct" in section 42.73(d)(3)(i) should be read to invoke the term's established meaning in the double patenting and interference contexts. That is particularly so because the term "patentably distinct" serves a similar purpose in all three settings: to
prohibit a patentee from exploiting patent claims that are not materially distinguishable from claims that have either expired or been invalidated in prior proceedings.

* * *

SoftView's core complaint is that the Board applied estoppel based on the IPR decision canceling claims from the '353 patent rather than comparing the claims before it to the prior art.

108 F.4th at 1369-70, 1371, 1372 (cited in Paper 29 at 11).

CERTIFICATE OF SERVICE

I hereby certify that on September 16, 2024, a true and correct copy of Patent Owner Inolex Investment Corp.'s Demonstratives was served on counsel of record for Petitioner by filing this document through the P-TACTS System as well as delivering a copy via electronic mail to the following addresses:

James P. Murphy
Polsinelli PC
1000 Louisiana Street
Suite 6400
Houston, TX 77002
jpmurphy@polsinelli.com

Ryan Murphy
R. James Balls
Polsinelli PC
1401 I St. N.W.
Suite 800
Washington, DC 20005
rmurphy@polsinelli.com
jballs@polsinelli.com

/Aaron S. Lukas/
Aaron S. Lukas
Reg. No. 59,443
COZEN O'CONNOR
1200 19th Street, N.W.
Washington, DC 20036

Lead Counsel for Patent Owner
Inolex Investment Corporation