

Symrise AG and Symrise Inc. v Inolex Investment Corp.

IPR2023-01035

Petitioner's Oral Hearing Demonstratives

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

September 19, 2024

Overview of Issues

- Background of Technology
- Ground 1: Wright '451
 - Wright '451 teaches all elements of Substitute Claims 29-44
 - No Unexpected Results
- Ground 2: De Bruyn in view of Takamura
 - Combination teaches all elements of Substitute Claims 29-44
 - Motivation to combine provided by the references, no hindsight necessary
- Ground 3: Written Description
 - No Flow Rate Claimed
- Ground 4: Indefiniteness-
 - Multiple Interpretations Possible
- Ground 5: Estoppel over Disclaimed '803 Claims

U.S. Patent No. 10,995,048 and Background of Technology

Proposed Substitute Claim 29 of '048 Patent

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 - RM TA at A-1

Background of Technology

- Dr. Taber (Petitioner expert):

23. Accordingly, before the Priority Date of the '048 Patent, a POSITA would expect to successfully obtain bio-1-alkenes with high regioselectivity by simply dehydrating bio-1-alcohol using well-known methods, as described in detail

33. As of the Priority Date of the '048 Patent, in my opinion, all of the chemical products and reactions claimed in the '048 Patent were well known, and had been well known for decades. For example, the chemical reactions described in the '048 Patent are taught in entry-level organic chemistry textbooks. The '048 Patent does not explain or identify how or why its chemical products and reactions differ from those known in the art.

Background of Technology

- Dehydration Process was Known:

- 1943-Heptanol dehydrated over γ -alumina – 98% conversion, 97% selectivity (Appleby)
- 1945-Octanol dehydrated over γ -alumina – 98% conversion, 98% selectivity (Komarewsky)
- 1980-1-alcohol dehydrated over base-treated γ -alumina – >94% conversion, 97.7% selectivity (Wu)
- 1989- study of 1-alcohol dehydrated over γ -alumina treated with different ions and calcined (Berteau)
- 1990-1-alcohol dehydrated over γ -alumina with Ammonia – 98% conversion, 95% selectivity (similar results to treatment with alkali earth metals) (Voste)
- 1992- study of 1-alcohol dehydrated over γ -alumina impregnated and calcined with various alkali metals (Cortez)

Ex. 1002 at 42-45;
Ex. 1038 at 22-28;
Paper 1- Pet. at 19;
Paper 14 - Opp to MTA at 13

Proposed Substitute Claim 29 of '048 Patent

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 - RM TA at A-1

Background of Technology

- Dr. Wright deposition(Patent Owner expert):

example is an operator would generally know that to get the highest conversion, they would, you know, and selectivity, they would try and meter between feed rate and temperature and try and find that, I'll call it a sweet spot.

Ex. 1040 at 42:16-22;
Paper 14 - Opp. to MTA at 7

Ground 1

Wright '451

Renders Obvious Claims 29-44

Ground 1 Overview

- Wright '451 teaches each element of the Substitute Claims
- Wright '451 has overlapping disclosure to the claimed ranges, thereby presenting a presumption of obviousness
 - Patent Owner fails to present evidence of unexpected results

"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 "

- Wright '451 teaches "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"

amount. The output of 401 is transferred to vessel 403 which contains the dehydration catalyst (*e.g.*, 206 or 304 prepared above) at an elevated temperature, for example, about 200 °C to about 440 °C, or about 300 °C to about 420 °C. Optionally, a purge gas (*e.g.*, an

"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 "

- Dr. Wright

Q Then at paragraph 27 it says:

In some embodiments, the reaction mixture is heated to a temperature of about 200 to about 440 C.

A Yes.

Q Is that teaching that dehydration reactions can take place at those temperatures?

A Yes.

"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 "

- Wright '451 teaches examples at 360 °C:

Table 2

Bio-1-butanol Dehydration Results.

	WHSV (h ⁻¹)	T (°C)	Aging time (h)	1-Butanol/DBE conversion	1-Butene selectivity

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

Ex. 1039 at Table 2;
Paper 20 - Opp. to RMTA at 13

"a calcium-promoted γ -alumina catalyst calcined at a temperature of about 420 °C to about 480 °C

- Wright '451 teaches a calcium-promoted γ -alumina catalyst:

[14] Catalyst compositions disclosed herein contain an inorganic support modified by a Ca^{+2} compound as one preferred embodiment. In one embodiment modification may be accomplished via treatment of the support with the calcium compound dissolved in a solvent. In some embodiments Ca^{+2} is the modifier and the Ca^{+2} compound is calcium acetate ($\text{Ca}(\text{OAc})_2$). The solvent may be water, or an organic solvent, or a mixture of the support) at about 0.8 to about 3.0 wt%. In one embodiment, γ -alumina is used as the inorganic support. In some embodiments, γ -alumina is modified with binder, physically

"a calcium-promoted γ -alumina catalyst calcined at a temperature of about 420 °C to about 480 °C

- Wright '451 teaches that following surface modification, "typically" calcination takes place

the solvent(s) is then removed, *e.g.*, with heat and/or reduced pressure. Those experienced in the methods of surface modification know that one technique to accomplish this is referred to as an incipient wetness impregnation process (IWP). IWP is typically followed by a drying or calcination process for some time, as shown schematically in FIG 2. The

heated to remove some or all of the IWP solvent. This material 204 can be further heated in a second apparatus (205) 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. The removal of the solvent can be done in air or under an inert atmosphere. In one

Ex. 1039 at [0067];
Paper 20 - Opp. to RMTA at 12

"a calcium-promoted γ -alumina catalyst calcined at a temperature of about 420 °C to about 480 °C

- Dr. Wright

Q And then the narrower range, 200 to 600, would they be able to utilize that smaller range to calcine 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

Alleged Unexpected Results

- Revised Non-Contingent Motion to Amend

As shown in the '048 patent, a calcium-promoted γ -alumina catalyst calcined at 440 °C provides unexpectedly better 1-octene regioselectivity and higher conversion compared to the same catalyst calcined at 500 °C. Ex. 1001, col.

- Requirements for Unexpected Results

Paper 18 - RMTA at 19

“Although those data show that alloy strength improved with the addition of rhenium, they do not evidence unexpected results for the entire claimed range of about 1-3% rhenium”

In re Peterson, 315 F.3d
1325, 1331 (Fed. Cir. 2003)

Alleged Unexpected Results

- Relied Upon Evidence of Better 1-Octene Regioselectivity:

26, l. 10 – col. 27, l. 9 (Examples 1-3); Ex. 2006, ¶¶20, 123. Specifically, the calcium-promoted γ -alumina catalyst calcined at 500 °C provided a 1-octene regioselectivity of 93.1% at a conversion of 95.5%, while calcination at 440 °C provided a 1-octene regioselectivity of 96.7% at a conversion of 97.2%. *See id.*

Paper 18 - RMTA at 19

- Wright '451 Disclosure:

[22] In some embodiments, the regioselectivity of 1-alkene production is greater than about 95%. In some embodiments, the regioselectivity of 1-alkene production is about 90%

Ex. 1039 at [0022];
Paper 20 - Opp. to RMTA at 13

Alleged Unexpected Results

- No Evidence that Regioselectivity is Unexpected:

This is consistent with the teachings by Wright '451. Ex. 1039 at ¶¶13, 14, 20, 22, 67, 78, 82; *see also* RMTA at 18; Ex. 2006 at ¶¶ 22, 66, 72, 76. There is nothing unexpected about the results as they align with prior art teachings. Ex. 1046 at ¶52.

Paper 20 - Opp. to RMTA at 8

As set forth in the '048 patent specification, base-treatment followed by calcination at a temperature of about 400 °C to about 480 °C, provides a bio-1-alkene regioselectivity of 97% and conversion of 97%, whereas calcination at a

Paper 12 - MTA at 23

Alleged Unexpected Results

- Relied Upon Evidence of Better Conversion:

increases with temperature.” Ex. 1039, [42]-[46]. Nothing in *Wright '451* would have taught or suggested to a POSITA that conversion of 1-hexanol would decrease at 370 °C. Ex. 2006, ¶¶125-127. What is more, a POSITA would view

Paper 18 - RMTA at 20

Alleged Unexpected Results

- Definition of Conversion

Q So is this saying that 95 percent of the bio-1-alcohol is converted to an alkene? Is that what "95 percent conversion" refers to?

A No, that doesn't say that. It just says 95 percent of the alcohol is no longer alcohol. That's been converted to something else.

Q So as long as 95 percent of the alcohol undergoes a reaction to a different product, then it's been converted?

A Correct.

- Dr. Wright

Ex. 1040 at 24:8-17;
Paper 29 - Sur-Reply at 5

Alleged Unexpected Results

- Underlying Data from '048 Patent: Conversion is Constant

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

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:

Alleged Unexpected Results

- Underlying Data from '048 Patent: Conversion is Constant

In EXAMPLE 11C where the reactor is maintained at 370° C. a new peak in the GC chromatogram appears have an area-% of 3.6; thus, indicating a lower chemical yield of 1-hexene and formation of another product.

Ground 1 Conclusion

- Wright '451 teaches each element of the Substitute Claims
- Patent Owner's Evidence fails to Show Unexpected Results

Ground 2

**De Bruyn in view of Takamura
Renders Obvious Claims 29-44**

Ground 2 Overview

- De Bruyn teaches a dehydration reaction for 1-hexanol and 1-octanol involving a calcium modified γ -alumina catalyst
- De Bruyn can be combined with Takamura for the specifics of the preparation of the calcium modified γ -alumina catalyst along with expectations on results of the preparation.

"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 "

- De Bruyn teaches “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”

[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

Ex. 1008 at [0037];
Paper 20 - Opp. to RMTA at 17

"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 "

De Bruyn:

[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-Al₂O₃ 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 at [0053];
Paper 20 - Opp. to RMTA at 17

"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 "

De Bruyn:

subjected to dehydration. The experimental procedure employed during dehydration, was as described in example 4. The results presented here are for a total of 8 hours on-stream. The conditions employed were a reaction temperature of 280° C. and an MHSV of 10 h⁻¹. The results obtained are shown in Table 14 and FIG. 2.

Ex. 1008 at [0081];
Paper 20 - Opp. to RMTA at 17-18

which is free of branched alcohols, is dehydrated. Therefore, with proper purification of the 1-octanol feed it could be possible to produce 1-octene fractions with purities higher than 95% at high conversions of the alcohol.

Ex. 1008 at [0083];
Paper 20 - Opp. to RMTA at 17-18

"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 "

Dr Wright Compiled these teachings from multiple examples of De Bruyn:

	<i>De Bruyn</i> (Example 3)	<i>De Bruyn</i> (Example 4, Step 5)	<i>De Bruyn</i> (Example 5, Step 4)
Alkene	1-hexene	1-hexene	1-octene
Catalyst	γ -alumina	γ -alumina Pural KR1	γ -alumina Pural KR1
Temp.	315 °C	290 °C	280 °C
Flow Rate (HSV) ^a	5 h ⁻¹	8 h ⁻¹	10 h ⁻¹
1-alkene regioselectivity	97.5%	96.5%	97%-98%
Conversion	94% ¹	76.5%	73.2%-59.4%

Ex. 2001 at [0045] (Excerpt);
Paper 12 - MTA at 15

"a calcium-promoted γ -alumina catalyst calcined at a temperature of about 420 °C to about 480 °C

- De Bruyn can be combined with Takamura to show the specifics of catalyst preparation.

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. $\text{CaO} \cdot \text{Al}_2\text{O}_3$, $\text{Ca}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3$.

Ex. 1008 at [0037];
Paper 20 - Opp. to RMTA at 17

[0062] The calcining temperature after the impregnation is preferably 300°C or more, more preferably 400°C or more, and further preferably 450°C or more, from the standpoint of the enhancement of the dehydration reaction rate through the formation of the oxide of the element, and is preferably 900°C or less, more preferably 850°C or less, further preferably 800°C or less, and still further preferably 550°C or less, from the standpoint of the enhancement of the dehydration reaction rate through the prevention of the reduction of the surface area of the catalyst and the maintenance of the degree of dispersion of the supported element.

Ex. 1044 at [0062];
Paper 20 - Opp. to RMTA at 16

Patentability of Claim 29-44 over De Bruyn in view of Takamura

- PO Only Provides Two Sentences of argument Re Patentability over De Bruyn in view of Takamura:

With respect to Ground 4, there would have been no motivation for a POSITA to modify De Bruyn because a POSITA would have simply used one of the commercially available catalysts described there, a problem that is compounded by

2010, ¶¶77, 81, 85, 102-109. Dr. Taber also impermissibly relies on hindsight in picking and choosing selected teachings of Takamura without any reasonable scientific basis or expectation of success. **Ex. 2010**, ¶¶102-109.

"a calcium-promoted γ -alumina catalyst calcined at a temperature of about 420 °C to about 480 °C

- Motivation Provided by the References:

[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 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. $\text{CaO} \cdot \text{Al}_2\text{O}_3$, $\text{Ca}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3$.

Ex. 1008 at [0037];
Paper 20 - Opp. to RMTA at 17

"a calcium-promoted γ -alumina catalyst calcined at a temperature of about 420 °C to about 480 °C

- Motivation Provided by the References:

[0060] The temperature at which the aluminum oxide is impregnated with the oxide of the element is preferably 0°C or more, and more preferably 20°C or more, and is preferably 100°C or less, more preferably 95°C or less, further preferably 90°C or less, still further preferably 50°C or less, and still further preferably 30°C or less, from the standpoint of the enhancement of the supporting rate and the standpoint of the enhancement of the dehydration reaction rate through the uniform support of the oxide of the element in the resulting catalyst.

[0061] The time of the impregnation is preferably 0.1 hour or more, more preferably 0.2 hour or more, and further preferably 0.5 hour or more, and is preferably 10 hours or less, more preferably 5 hours or less, and further preferably 2 hours or less, from the standpoint of the enhancement of the supporting rate and the standpoint of the enhancement of the dehydration reaction rate through the uniform support of the oxide of the element in the resulting catalyst.

[0062] The calcining temperature after the impregnation is preferably 300°C or more, more preferably 400°C or more, and further preferably 450°C or more, from the standpoint of the enhancement of the dehydration reaction rate through the formation of the oxide of the element, and is preferably 900°C or less, more preferably 850°C or less, further preferably 800°C or less, and still further preferably 550°C or less, from the standpoint of the enhancement of the dehydration reaction rate through the prevention of the reduction of the surface area of the catalyst and the maintenance of the degree of dispersion of the supported element.

Ex. 1044 at [0060]-[0062];
Paper 20 - Opp. to RMTA at 16

"a calcium-promoted γ -alumina catalyst calcined at a temperature of about 420 °C to about 480 °C

- No Hindsight Required, Promoted γ -Alumina Catalyst Explicitly Disclosed:

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. $\text{CaO} \cdot \text{Al}_2\text{O}_3$, $\text{Ca}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3$.

Ex. 1008 at [0037];
Paper 20 - Opp. to RM TA at 17

Ground 2 Conclusion

- De Bruyn in view of Takamura Teaches all Elements of the Substitute Claims
- Combination is Proper, does not Require Hindsight

Ground 3

**Claims 29-44 Have no Written
Description Support**

Ground 3-Overview

- The Revised Substitute Claims do not include Flow-Rate.

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 - RMTA at A-1

- Dr. Wright Opines that Flow-Rate is a Necessary Variable

~~overlapping with a reactor temperature of 325 °C to 335 °C. 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. ~~Therefore, because Example 10~~

Ex. 2006 at 115;
Paper 20 - Opp. to RMTA at 3

Ground 3-No Written Description

- The '048 is largely silent as to Flow-Rate.

33. Furthermore, after a review of the '048 patent itself, a POSITA would understand that it is largely silent as to flow-rates for the reactions. For example, Examples 3, 10, and 11 merely state that flow rates are adjusted to obtain the desired conversion. Ex. 1001 at 26:55-67, 28:55-67, and 29:3-11. However, with no indication of what amount of alcohol needs to be introduced or how the amount of alcohol is introduced relative to the amount of catalyst present, a POSITA would not be able to recreate the process claimed. Based on the '048

- Dr. Taber

Ex.1046 at [0033];
Paper 20 – Opp. to RMTA at 3

Ground 3-Alleged Written Description Support

- PO's Only Identified Support – Example 5 of the '048 Patent

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 at 27:20-35;
Paper 29 - Sur Reply at 7

Ground 3-Alleged Written Description Support

- PO's Only Identified Support – Example 5 of the '048 Patent
- Claim 1 recites:

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 - RMTA at A-1

- Example 5 only recites “octene selectivity”

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 at 27:20-35;
Paper 29 - Sur Reply at 7

Ground 3-Alleged Written Description Support

- PO's Only Identified Support – Example 5 of the '048 Patent
- Claim 1 recites:

~~20~~ carbon atoms ~~and~~ is produced in a conversion of at least 95% % ~~and has~~

Paper 18 - RMTA at A-1

- Example 5 does not address conversion

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 at 27:20-35;
Paper 29 - Sur Reply at 7

Ground 4

Claims 29-44 are Indefinite

Ground 4-Overview of Indefiniteness

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 -

Paper 18 - RMTA at A-1

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 - Opp. to RMTA at 4

Ground 4-Indefiniteness

- PO's Provides an Implicit Claim Construction

Contrary to Petitioner's argument, a POSITA reviewing the specification of the '048 patent would readily understand that the phrase at issue merely requires that the continuous flow reactor be heated at a temperature within the specified range.

Paper 25 - Reply at 8

Ground 4-Indefiniteness

- PO argues, through Dr. Wright's Declaration, that dependent claims provide evidence of definiteness.

- Dr. Wright

72. Finally, I note that claims 35, 38, 41, and 44 use nearly identical words as the phrase in substitute claim 29 that is allegedly indefinite, but Dr. Taber did not identify any uncertainty or ambiguity as to the scope of those dependent claims. *See, Ex. 1046, ¶¶34-38.*

Ex. 2010 at [0072];
Paper 25 - Reply at 8

35. (Proposed Substitute for Claim 8) The method according to claim ~~4~~34, wherein the reactor is heated at a temperature of 315 °C to 350 °C catalyst

Paper 18 - RMTA at A-1

Ground 5

Claims 29-44 are Subject to Estoppel

Ground 5- Estoppel Background

- PO disclaimed claims 1-7, 9-11, 24-26, 28, 29, 35, and 36 of US Pat. No.10,882,803

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).

Ex. 1045 at 29:24-32;
Paper 20 - Opp. to RMTA at 19

Ground 5- Estoppel Disclaimed Claims

- Claim 1 of US Pat. No.10,882,803

1. A process for the synthesis of bio-1,2-alkanediols, comprising:
providing a bio-alkene having a carbon chain of about 5 to about 20 carbon atoms and a bio-1-alkene regioselectivity of at least about 80%; and
converting the bio-alkene to a bio-1,2-alkanediol having a carbon chain length of about 5 to about 20 carbon atoms.

Ex. 1045 at 29:24-32;
Paper 20 - Opp. to RMTA at 21

Ground 5- Estoppel Disclaimed Claims

- Claims 6, 7, and 9-11 of US Pat. No.10,882,803

6. The process according to claim 1, wherein the bio-alkene results from a process wherein a bio-1-alcohol is dehydrated by heating the bio-1-alcohol in a reactor with a catalyst.

7. The process according to claim 6, wherein the reactor is a fixed bed reactor.

9. The process according to claim 6, wherein the catalyst is selected from ZnAl_2O_4 and a γ -alumina catalyst.

10. The process according to claim 9, wherein the bio-alkene has a bio-1-alkene regioselectivity of about 92% to about 99%.

11. The process according to claim 10, wherein the bio-alkene have a bio-1-alkene regioselectivity of about 95% to about 99%.

Ex. 1045 at 29:44-53;
Paper 20 - Opp. to RMTA at 21

Ground 5- Alleged Distinctions

- Dr. Wright identifies the alleged distinctions are within the disclaimed subject matter, i.e., doped and calcined γ -Alumina Catalysts

“ 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

Ex. 2010 at [0052];
Paper 25 - Reply at 5

Ground 5- Estoppel Background

- '803 Discussion of Calcination

treatments typically used in prior catalyst processes. Instead, the temperature for calcination is kept at about 400° C. to about 500° C., and preferably about 420° C. to about 480° C., and most preferably about 440° C. to about 460° C.

Ex. 1045 at 17:22-24;
Paper 20 - Opp. to RMTA at 21

- '803 Discussion of Reaction Temperature

In one embodiment the process is carried out in a fluidized bed reactor operated in the temperature range of about 240° C. to about 360° C. Other continuous flow reactor designs

Ex. 1045 at 15:39-41;
Paper 20 - Opp. to RMTA at 21

Ground 5- Estoppel Background

- '803 Example

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

Ex. 1004 at 59 ([0125]);
Paper 12 - MTA at 8

Ground 5- Alleged Distinctions

- Patent Owner Identifies 4 Alleged Distinctions

process parameters including the requirements of i) a calcium-promoted γ -alumina catalyst, ii) calcined at a temperature of about 420 °C to about 480 °C, iii) wherein the catalyst has not been silanized, iv) in a reactor at a temperature of 240 °C to 360 °C. **Ex. 2010**, ¶¶52-59. Further, substitute claims 34, 25, 37, 38, 40, 41, 43, and 44

Ground 5- Alleged Distinctions

- Alleged Distinction 1: Prior Art Teaches Calcium Promoted γ -Alumina Catalysts

117. As discussed above, *Wright '451* discloses the use of calcium-promoted γ -alumina catalysts that are prepared by modifying a γ -alumina catalyst with a Ca^{+2} compound. **Ex. 1039** at ¶[14]. Whereas *Wright '788* only includes a

Ex. 2006 at [0117];
Paper 20 - Opp. to RMTA at 22

Ground 5- Alleged Distinctions

- Alleged Distinction 1: Prior Art Teaches Calcium Promoted γ -Alumina Catalysts

De Bruyn states that “many different dehydration processes are known, and are accordingly not disclosed in any detail in this specification,” and also discloses that “the catalyst may comprise a gamma-alumina catalyst or a promoted alumina catalyst e.g. $\text{CaO} \cdot \text{Al}_2\text{O}_3$, $\text{Ca}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3$.” (Ex. 1008, ¶ [0037].) However, *De Bruyn*

Ex. 2001 at [0041];
Paper 12 - MTA at 23

Ground 5- Alleged Distinctions

- Alleged Distinction 2: The Prior Art Calcined Catalysts at a temperature of about 420 °C to about 480 °C

Wright '451 discloses calcination of the modified γ -alumina catalysts “at temperatures of about 100 °C to about 900 °C, or about 200 °C to 600 °C” (**Ex. 1039** at ¶[67]) and in the examples teaches drying and/or calcination of various catalysts at 120 °C, 200 °C, 500 °C, 550 °C, and/or 600 °C (**Ex. 1039** at pp. 24-33, examples 2-4, 8-24).

Ex. 2006 at [0120];
Paper 20 - Opp. to RMTA at 22

Ground 5- Alleged Distinctions

- Alleged Distinction 2: The Prior Art Calcined Catalysts at a temperature of about 420 °C to about 480 °C

discussion of calcination time, not temperature)). I note that *Takamura* at ¶[0062] discloses that the calcination temperature for the impregnated catalyst is “preferably 450 °C or more, from the standpoint of the enhancement of the dehydration reaction through the formation of the oxide of the element,” but, as

Ex. 2010 at [0105];
Paper 25 - Reply at 10

Ground 5- Alleged Distinctions

- Alleged Distinction 3: Prior Art Catalysts are “Unsilanized”.

be silanized. Ex. 1039, ¶[68]-¶[72]. However, unlike *Wright '788*, *Wright '451* discloses examples in which the modified γ -alumina catalysts are both unsilanized or silanized. See Ex. 1039 at 24-33 (Examples 4, 7, and 20-22 utilize silanized catalysts).

Ex. 2006 at [0118];
Paper 20 - Opp. to RMTA at 22

Ground 5- Alleged Distinctions

- Alleged Distinction 4: Prior Art Dehydration Reactions take place at about 240 °C to about 360 °C

26. For example, Inolex admits that the claimed dehydration temperature range is known in the art. See e.g., RMTA at 20 (reactor temperatures of 220 °C -440 °C); Ex. 2001 at ¶45 Table (De Bruyn and Le teach reactor temperatures of 280 °C to 345 °C) Ex. 2006 at ¶42 (Wu teaches reactor temperatures of 200 °C to 500 °C); ¶48 (Chokkaram teaches reactor temperatures of 280 °C); ¶120 (Wright 451 teaches reactor temperatures of 220 °C-440 °C). The Board has found this temperature range is known in the art. Paper 7 at 11, 13; Paper 16 at 11, 12, 14, 15. Inolex also admits that this calcination temperature range was known.

Ex. 1046 at [0026];
Paper 20 - Opp. to RMTA at 22

Ground 5- Alleged Distinctions

- Alleged Distinction 4: Prior Art Dehydration Reactions take place at about 240 °C to about 360 °C

alkene regioselectivity, and time-on-stream could be obtained. Second,

Wright '451 teaches a reactor temperature of about 220 °C to about 440 °C, and

Ex. 2006 at [0120];
Paper 20 - Opp. to RMTA at 22

42. For the conditions of the dehydration reaction, *Wu* teaches that

“temperatures in the range of from about 200° to 500° C. are suitable for the

Ex. 2006 at [0042];
Paper 20 - Opp. to RMTA at 22

Ground 6

**Claims 29-44 are Procedurally
Improper**

Ground 6- Summary

- PO Filed Knowingly Obvious Claims

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 reactor with a base-treated and calcined γ -alumina catalyst, wherein the catalyst has not been silanized, and

wherein the resulting bio-alkene has a carbon chain of 6 to 14 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 92% to about 99% at least about 80%.

Paper 12 - MTA at A-1

Ground 6- Summary

- Wright '451's Admitted Teachings

66. Similar to *Wright '788*, *Wright '451* discloses the methods of preparing bio-1-alkenes from bio-1-alcohols, and in particular bio-1-butene from bio-1-butanol, by catalytic dehydration with a 1-alkene regioselectivity of greater than about 90%, about 90% to about 99%, or greater than about 95% using a γ -alumina catalyst that is 1) treated with a calcium promoter, 2) calcined, and 3) optionally reacted with an organosilane, washed, and dried. Ex. 1039, ¶[18], ¶[22].

Ex. 2006 at [0066];
Paper 20 - Opp. to RMTA at 22

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

Ex. 2010 at [0112];
Paper 25 - Reply at 10

Ground 6- Procedurally Improper

- Rejected
Claims in '803
Prosecution
History

35. (original) A method for treating a catalyst for use in dehydration of alcohols, comprising providing a γ -alumina catalyst;
treating the γ -alumina catalyst with a base comprising a Group I or a Group II metal; and
heating the γ -alumina catalyst to a temperature of about 400°C to about 500°C.

36. (original) The method according to claim 35, wherein the base comprises calcium.

37. (original) The method according to claim 36, wherein the base is a calcium promoter and the heating occurs during calcination.

38. (original) The method according to claim 37, wherein the calcium promoter includes is used in an amount of about 0.01 weight percent to about 4 weight percent based on the weight of CaO determined after calcination.

39. (original) The process according to claim 38, 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.

40. (original) The process according to claim 35, temperature during calcination is about 420°C to about 480°C.

41. (original) The process according to claim 40, wherein the temperature during calcination is about 440°C to about 460°C.

Ex. 1004 at 4-5;
Paper 20 - Opp. to RMTA at 24

Ground 6-Procedurally Improper

- Examiner's Rejection

21. However, Wright discloses a method for treating catalyst for use in the dehydration of alcohols to diene products. ([22] & [32]). In the teachings of Wright a gamma-alumina catalyst is provided. The gamma-alumina catalyst is treated with a base comprising a Group 1 or Group 2 metal. ([33] – [34]). Calcination of the catalyst can be carried out at temperatures of about 100°C to about 900°C. ([122]). In Wright the base comprises calcium. ([33]). Also, in Wright the calcium base is a promoter and heating is conducted during calcination. ([122]). Further, the Ca-modified catalyst comprised about 0.8wt-% to about 4.0 wt.-% or about 1.0 wt.-% to 1.7 wt.-% of the total catalyst (weight of Ca^{+2} /(weight of inorganic support + weight of Ca^{+2}) (consistent with 1.1 – 5.6 weight percent based on the weight of CaO, provided the molar mas of Ca is 40g/mol; and Cao is 56g/mol) . ([122]). The temperature during calcination is about 100°C to about 900°C.

Ex. 1004 at 127;
Paper 20 - Opp. to RMTA at 24

Conclusion

Conclusion

- The Prior Art renders obvious Revised Substitute Claims 29-44
- Patent Owner's only argument is that they achieved unexpected results
 - The results relied on are not unexpected and include improper interpretations of the evidence
- There is no Written Description Support for all flow rates
- The Claim Language is Indefinite
- Patent Owner is Estopped from arguing for patentability
- The claims are Procedurally Improper

Appendix

CERTIFICATE OF SERVICE

The undersigned hereby certifies that a copy of the Petitioner's Oral Hearing Demonstratives was served September 16, 2024, via email directed to the following email addresses of record:

Aaron S. Lukas
alukas@cozen.com

W. Blake Coblentz
wcoblentz@cozen.com

Mark Boezi
mboezi@cozen.com

/Ericka McNeil/
Ericka McNeil
Polsinelli PC
1000 Louisiana Street, 64th Floor
Houston, Texas 77002