

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

DAICEL CORPORATION,
Petitioner,

v.

CELANESE INTERNATIONAL CORPORATION,
Patent Owner.

Case IPR2014-01515
Patent 7,223,886 B2

Before LINDA M. GAUDETTE, CHRISTOPHER L. CRUMBLEY, and
JON B. TORNQUIST, *Administrative Patent Judges*.

TORNQUIST, *Administrative Patent Judge*.

DECISION
Denying Institution of *Inter Partes* Review
37 C.F.R. § 42.108

I. INTRODUCTION

Daicel Corporation (“Petitioner”) filed a Petition (Paper 3, “Pet.”) requesting institution of *inter partes* review of claim 1 of U.S. Patent No. 7,223,886 B2 (Ex. 1001, “the ’886 patent”). Celanese International Corporation (“Patent Owner”) timely filed a Preliminary Response (Paper 7, “Prelim. Resp.”) to the Petition.

We have jurisdiction under 35 U.S.C. § 314(a), which provides that an *inter partes* review may not be instituted “unless . . . there is a reasonable likelihood that the petitioner would prevail with respect to at least 1 of the claims challenged in the petition.” For the reasons given below, we determine that Petitioner has not demonstrated a reasonable likelihood of prevailing with respect to claim 1 of the ’886 patent. Accordingly, we do not authorize an *inter partes* review to be instituted as to the challenged claim.

A. *Related Matters*

Petitions for *inter partes* review of related U.S. Patent No. 8,076,507 are pending in IPR2014-01514, IPR2015-00170, IPR2015-00171, and IPR2015-00173. Pet. 1–2; Paper 10, 2.

B. *The ’886 Patent*

The ’886 patent is directed to an improved process for the removal of permanganate reducing compounds (“PRCs”) and alkyl iodides “formed by the carbonylation of methanol in the presence of a Group VIII metal carbonylation catalyst.” Ex. 1001, 1:9–12. In particular, “the invention relates to an improved process for reducing and/or removing precursors of permanganate reducing compounds and alkyl iodides from intermediate streams during the formation of acetic acid.” *Id.* at 1:12–17.

In the disclosed process, carbon monoxide is bubbled through methanol in the presence of water, methyl acetate, methyl halide, and rhodium to produce acetic acid. *Id.* at 1:43–53, 6:42–49. PRC impurities, such as acetaldehyde, are generated in this process and may react with iodide catalyst promoters to form alkyl iodides. *Id.* at 2:36–39. Even in small amounts, these alkyl iodides are undesirable because they “tend to poison the catalyst used in the production of vinyl acetate, the product most commonly produced from acetic acid.” *Id.* at 2:39–44.

The ’886 patent discloses that, “[b]ecause many impurities originate with acetaldehyde,” a focus in the art was to remove acetaldehyde from the production process. *Id.* at 2:46–49. The ’886 patent further discloses that many different techniques were known in the art for removing acetaldehyde from the final product, including treating the product with oxidizers and using a series of distillation steps. *Id.* at 2:50–55. These methods of treating the final acetic acid product were not economically or commercially feasible, however, because the impurities to be removed “have boiling points close to that of the acetic acid product.” *Id.* at 3:7–10. Thus, methods of removing impurities elsewhere in the process were needed. *Id.* at 3:11–14.

The ’886 patent discloses that several successful methods for removing acetaldehyde from various points in the production process were known in the art, including those disclosed in U.S. Patent Nos. 5,625,095 (Ex. 1010, “the ’095 patent”) and 6,339,171 (Ex. 1011, “the ’171 patent”). *Id.* at 3:31–67. According to the ’886 patent, the ’095 patent discloses a successful method of removing acetaldehyde by distilling the light phase stream, heavy phase stream, or both, but does not “identify which of these streams possesses the greatest concentration of acetaldehyde.” *Id.* at 3:31–

Figure 1 illustrates “the prior art process, as disclosed in” the ’171 patent, and Figure 2 “illustrates a preferred embodiment” of the ’886 patent. *Id.* at 5:10–15.

In the prior art process of Figure 1, a first vapor phase acetic acid stream 28, containing methyl iodide, methyl acetate, acetaldehyde and other carbonyl components, exits splitter column 14. *Id.* at 8:29–32. This stream is condensed in vessel 16 to separate a heavy phase, containing the catalytic components, and light phase 30, containing acetaldehyde, water, and acetic acid. *Id.* at 8:32–36. Either phase may then be directed to column 18 for distillation, but distillation of the light phase is preferred. *Id.* at 8:37–46.

From column 18, second vapor phase 36, which is enriched in aldehydes and alkyl iodides, is condensed in vessel 20 to form a second liquid phase product. *Id.* at 8:45–50. This second liquid phase product is then directed to second distillation column 22, where acetaldehyde is separated from the other components of the liquid. *Id.* at 8:50–53. Stream 52, exiting from the top of column 22, is directed first to a condenser and then to overhead receiver 24 and extractor 27. *Id.* at 10:38–56. In the extractor, PRCs and alkyl iodides are extracted with water. *Id.* at 10:56–59. PRC-rich aqueous extract stream 64 exits the extractor from the top and is directed to waste treatment. *Id.* at 10:63–67. Raffinate stream 66, containing methyl iodide, exits the condenser from the bottom and is returned to the reaction system. *Id.*

In Figure 2, an additional extraction step is performed. Specifically, instead of returning raffinate stream 66 to the reaction system, it is directed to extractor 25 to remove additional PRCs and alkyl iodides. *Id.* at 11:1–5. Aqueous extract stream 70 exits from the top of extractor 25 and is directed

to waste treatment. *Id.* at 11:8–14. Raffinate stream 72, exiting from the bottom of extractor 25, is “recycled to the reaction system and ultimately to the reactor.” *Id.*

The ’886 patent discloses that, although the multistage extraction set forth in Figure 2 removes acetaldehyde, it also may remove a measurable amount of methyl iodide, “an especially costly component of the reaction system.” *Id.* at 11:27–32. The ’886 patent discloses, however, that loss of methyl iodide in the extraction step can be reduced when dimethyl ether (“DME”) is present in the extractor, which can be produced in “[t]he required quantity” by introducing water upstream of the extractor, for example, in feed 40 or reflux 50. *Id.* at 11:35–50. The ’886 patent postulates that this water reacts with methyl acetate and/or methyl iodide to form methanol, “which is then dehydrated in the presence of an acid catalyst (such as HI) to form DME.” *Id.* at 11:51–56.

C. Illustrative Claim

Independent claim 1 is reproduced below:

1. An improved method for reduction and/or removal of permanganate-reducing compounds (PRC’s)[,] C₃₋₈ carboxylic acids and C₂₋₁₂ alkyl iodide compounds formed in the carbonylation of a carbonylatable reactant selected from the group consisting of methanol, methyl acetate, methyl formate and dimethyl ether and mixtures thereof to an acetic acid product, the products of said carbonylation including a volatile [p]hase that is distilled to yield a purified acetic acid product and a first overhead comprising methyl iodide, water and at least one PRC, wherein the improvement comprises the steps of:

(a) distilling at least a portion of the first overhead to produce a second overhead stream comprising methyl iodide, dimethyl ether, and said at least one PRC;

(b) extracting the second overhead stream with water to form a first raffinate and a first aqueous extract stream containing said at least one PRC; and

(c) extracting the first raffinate with water to form a second raffinate and a second aqueous extract stream containing said at least one PRC,

wherein the improvement further comprises introducing at least a portion of the second raffinate directly or indirectly into the reaction medium.

Ex. 1001, 12:5–27; *see also* Ex. 1007, claim 1 (indicating typographical errors in published claims).

D. The Prior Art

Petitioner relies upon the following references, as well as the Declarations of Jeremy Cooper (Ex. 1003) and Hiroyuki Miura (Ex. 1005):

Reference	Document	Date	Exhibit
Miura ("the '095 patent")	US 5,625,095	Apr. 29, 1997	1010
Singh ("the '171 patent")	US 6,339,171 B1	Jan. 15, 2002	1011
Akinori	<i>Acetic Acid Synthesis from Methanol</i> , 20 J. JAPAN PETROLEUM INSTITUTE 379–462 (1977) (English translation)	1977	1013 ¹

¹ The original Japanese language document is provided as Exhibit 1012.

E. The Asserted Grounds of Unpatentability

Petitioner challenges the patentability of claim 1 of the '886 patent based on the following grounds:

Reference(s)	Basis	Claim Challenged
The '095 patent	§ 102	1
The '171 patent	§ 103	1
The '095 patent and Akinori	§ 103	1
The '095 patent and the '171 patent	§ 103	1

II. ANALYSIS

A. Claim Construction

Claims of an unexpired patent are interpreted using the broadest reasonable construction in light of the specification of the patent. *See* 37 C.F.R. § 42.100(b); *see also In re Cuozzo Speed Techs., LLC*, No. 2014-1301, 2015 WL 448667, at *7 (Fed. Cir. Feb. 4, 2015) (“Congress implicitly adopted the broadest reasonable interpretation standard in enacting the AIA.”). For purposes of this Decision, we determine no express construction of the claim language is required.

B. Anticipation by the '095 Patent

Petitioner contends that claim 1 is anticipated by the '095 patent. Pet. 20–29. In support of its argument, Petitioner relies upon the Declaration testimony of Mr. Cooper and Mr. Miura. *Id.*

1. The '095 Patent

The '095 patent is directed to “a process for producing acetic acid by continuously reacting methanol with carbon monoxide in the presence of water using a rhodium catalyst and methyl iodide.” Ex. 1010, 1:14–18. One

object of the '095 patent is "to provide a process for producing high purity acetic acid, wherein carbonyl compounds or organic iodides . . . are reduced." *Id.* at 2:45-49.

The process and equipment for accomplishing the method of the '095 patent are depicted in Figures 1 and 2, set forth below:

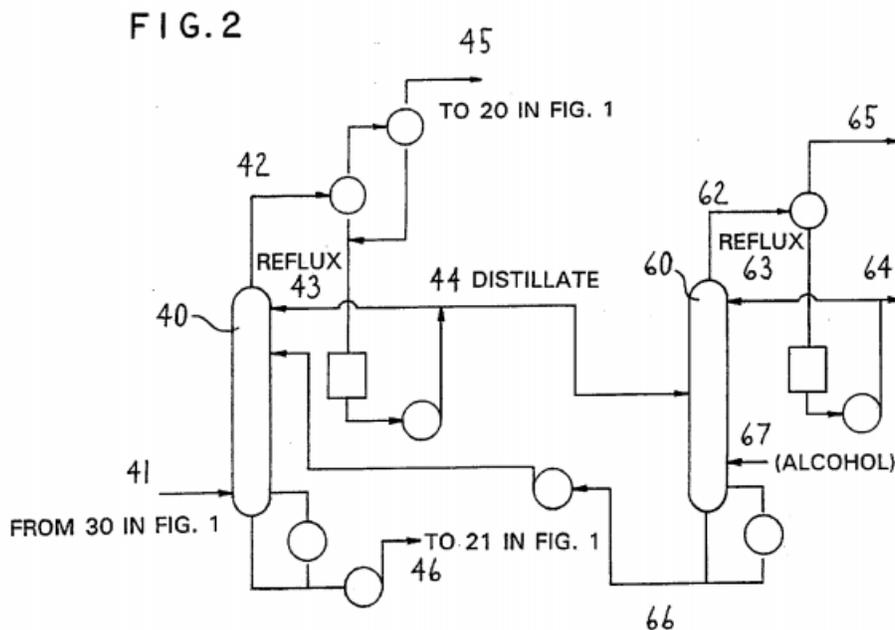
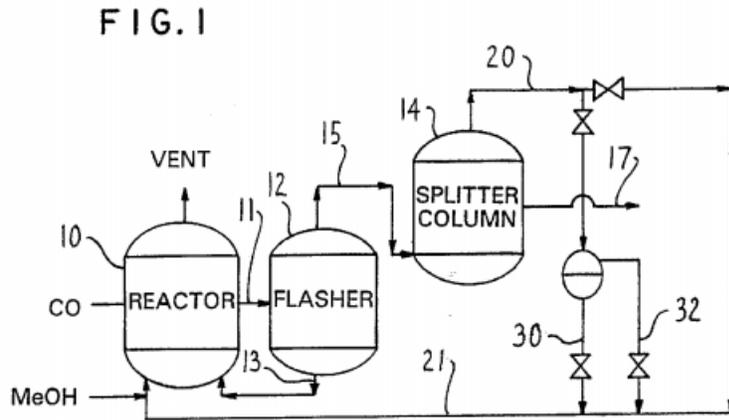


Figure 1 is a process flow diagram showing a reaction and recovery system "used for rhodium-catalyzed carbonylation of methanol to acetic acid." *Id.*

at 5:3–5. Figure 2 shows “one example of a distillation system for separating methyl iodide from acetaldehyde.” *Id.* at 2:34–35.

Referring to Figure 1, liquid products are withdrawn from the reactor 10 via line 11 and introduced into flasher 12. *Id.* at 5:30–34. A catalyst liquid—containing primarily acetic acid, rhodium catalyst, and iodide salts—is withdrawn from the flasher as bottom stream 13 and returned to reactor 10. *Id.* at 5:34–39. Overhead 15, which “contains mainly product acetic acid together with methyl iodide, methyl acetate, and water,” is directed to splitter column 14. *Id.* at 5:39–41. Splitter column 14 separates acetic acid from methyl iodide, methyl acetate, and water, and these products are then transferred as overhead 20 and separated into two liquid phases: lower phase 30 containing primarily methyl iodide and small amounts of methyl acetate and acetic acid, and upper phase 32 containing “mainly water, acetic acid, and a small amount of methyl acetate.” *Id.* at 5:50–55.

The lower phase, the upper phase, or both, are then introduced into 80 plate distillation column 40. *Id.* at 8:52–58. From the bottom of distillation column 40 a methyl iodide stream is recirculated into the reactor via line 46, whereas distillate 44, obtained from the top of the distillation column, is extracted with water in one or two stages. *Id.* at 8:57–58, 12:56–61, 15:30–40 (noting that extraction using a theoretical stage of one stage resulted in acetaldehyde extractability of 68% and extraction using a theoretical stage of two stages resulted in an acetaldehyde extractability of 95%). After extraction, the methyl iodide-rich raffinate is directed into the 80 plate distillation column, “to thereby recirculate it into the reactor as a bottom withdrawn liquid from the above 80 plates distillation column.” *Id.* at 13:3–

8. The aqueous phase may then be supplied to subsequent distillation column 60, wherein acetaldehyde is withdrawn as a distillate and water is withdrawn as a bottom product and recirculated to the extractor as a solvent. *Id.* at 13:9–20.

2. Analysis

Petitioner asserts that the '095 patent expressly discloses each and every element of claim 1, except the presence of DME in the second overhead. Pet. 24–25. Petitioner asserts, however, that “DME is inherently produced in the process and therefore it must be present in the overhead as described in the examples of the '095 patent.” *Id.* at 25.

To show that the '095 patent discloses a process that inherently results in the presence of DME, Petitioner relies upon an experiment conducted by Mr. Miura, one of the inventors identified on the face of the '095 patent. *Id.*; Ex. 1005. Petitioner asserts that this experiment confirms that when the '095 patent process is operated according to the “Example” and “Example 1” of the '095 patent, DME is present in the first and second overheads. Pet. 25–26; Ex. 1005 ¶¶ 5, 7, 8, 10, 13.

Patent Owner contends that Petitioner’s experimental evidence is insufficient to establish that DME is inherently present in the second overhead stream of the '095 patent, because Mr. Miura’s experiment does not “*come remotely close to replicating* the conditions and compositions” of the examples in the '095 patent.² Prelim. Resp. 28–29. Patent Owner

² Compare Ex. 1005 ¶ 11 (describing the reaction liquid of the Miura experiment as containing “8.2 wt% methyl iodide, 2.7 wt% water, 1.6 wt% methyl acetate, 73.5 wt% acetic acid, 12.3 wt% lithium iodide, and 910 ppm rhodium”) with Ex. 1010, 12:9–12 (describing the Example reaction liquid

contends that: (1) Mr. Miura's use of increased levels of lithium iodide would be expected to increase side reactions; (2) Mr. Miura's use of increased rhodium catalyst content (400 ppm to 910 ppm) would be expected to produce dramatically different reaction kinetics; and (3) Mr. Miura's use of a lower water content (2.7 wt% vs. 8 wt%) would be expected to result in a markedly different impurity profile. *Id.* at 30–31 (citing Ex. 2014,³ 2:15–20, 5:38–41 (noting that because lithium iodide increases side reactions, its “content must . . . be carefully set”), 8:21–25).

Mr. Miura concedes that his experiments do not match those used in the '095 patent exactly, but asserts “it was not feasible” to do so. Ex. 1005 ¶¶ 10, 21. Mr. Miura also asserts that the differences in reaction liquid composition would not be expected to affect the determination of whether DME is necessarily present in the process disclosed in the '095 patent, stating:

The *slight difference* in weight % of methyl iodide, water, acetic acid, lithium iodide, and rhodium in Experiment 1 as compared to ‘Example’ from the '095 Patent is inconsequential to the outcome of the experiment relating to the presence of DME in the first and second overheads and that methanol is found in the liquid charged into the 80-plate distillation column. This is because Experiment 1 shows that a reaction liquid *having the same constituents* as set forth in the Example of the '095 Patent and subjected to similar conditions results in the

as containing 14 wt% methyl iodide, 8 wt% water, 1.6 wt% methyl acetate, 70.9 wt% acetic acid, 5 wt% lithium iodide, and 400 ppm rhodium).

³ U.S. Patent No. 7,683,212 B2 issued March 23, 2010 and is directed to a method for producing “acetic acid by continuously reacting methanol with carbon monoxide in the presence of a rhodium catalyst, an iodide salt, methyl iodide, methyl acetate, and water.” Ex. 2014, Abstract.

presence of DME being formed from the reaction liquid. Because these reactants are known to produce DME under these conditions, at least some DME will always be present under these conditions, regardless of any slight differences in the concentrations of the reactants. Furthermore, even if not exactly the same as in the Example, the composition (proportion of the components) of the reaction liquid used in Experiment 1 is *within the scope of the reaction liquids expressly taught in the '095 Patent* at, for example, column 4, lines 25, 42-45, 48, 52, and 55-57[].

Id. ¶ 21 (emphasis added).

“To establish inherency, the extrinsic evidence ‘must make clear that the missing descriptive matter is necessarily present in the thing described in the reference, and that it would be so recognized by persons of ordinary skill.’” *In re Robertson*, 169 F.3d 743, 745 (Fed. Cir. 1999) (quoting *Cont’l Can Co. USA, Inc. v. Monsanto Co.*, 948 F.2d 1264, 1268 (Fed. Cir. 1991)). Inherency “may not be established by probabilities or possibilities. The mere fact that a certain thing may result from a given set of circumstances is not sufficient.” *Cont’l Can*, 948 F.2d at 1269.

We agree with Patent Owner that Petitioner’s experimental evidence is insufficient to conclude that the process of the ’095 patent necessarily will result in a “second overhead stream comprising methyl iodide, dimethyl ether, and said at least one PRC,” as required in claim 1. Ex. 1001, 12:16–18. First, Mr. Miura does not explain sufficiently why one of ordinary skill in the art would have considered the differences in weight percent of methyl iodide, water, acetic acid, lithium iodide, and rhodium between the Miura experiment and the Example of the ’095 patent to be “slight” or “inconsequential.” See 37 C.F.R. § 42.65(a) (“Expert testimony that does not disclose the underlying facts or data on which the opinion is based is

entitled to little or no weight.”); *In re Am. Acad. of Sci. Tech. Ctr.*, 367 F.3d 1359, 1368 (Fed. Cir. 2004) (noting that the Board is entitled to weigh declarations and conclude that the lack of factual corroboration warrants discounting the opinions expressed).

Second, Mr. Miura provides no credible support for his conclusion that as long as the two processes use the same constituents—even if at significantly different concentrations—the same products necessarily will be obtained in the second overhead. Nor does Mr. Miura provide a credible explanation as to why “it was not feasible” to run the experiment using reaction conditions identical to, or at least closer to, the conditions set forth in the examples of the ’095 patent.

Finally, Mr. Miura does not explain sufficiently the basis for his apparent conclusion that, because his experiment was run within the broad range of reaction conditions disclosed in the ’095 patent, any variations in operating conditions between the ’095 patent examples and his experiment would not influence whether DME is found in the second overhead.

Although an experiment need not recreate a disclosed embodiment precisely to establish inherency, the proponent of the experimental evidence must demonstrate sufficiently that the variations would not affect the relevant outcome, in this case the presence of DME in the second overhead. Upon review of the record as a whole, we are not persuaded that Mr. Miura provides sufficient explanation for why the variations in his experiment would not affect the formation of DME in the second overhead of the ’095 patent. Accordingly, we are not persuaded that Mr. Miura’s experimental evidence shows that DME is necessarily present in the second overhead when run under the conditions disclosed in the Example and Example 1 of

the '095 patent. Thus, we are not persuaded that Petitioner has demonstrated a reasonable likelihood that claim 1 is anticipated by the '095 patent.

C. Obviousness of Claim 1 Over the '171 Patent

Petitioner asserts that claim 1 would have been obvious over the '171 patent. Pet. 29–35. Patent Owner responds that the '171 patent does not render claim 1 obvious because, *inter alia*, the '171 patent does not disclose or suggest the presence of DME in the second overhead. Prelim. Resp. 43.

To demonstrate that DME is necessarily present in the second overhead, Petitioner again relies upon Mr. Miura's experimental evidence based on the examples of the '095 patent. Pet. 30 (“DME is present in the second overhead stream of Figure 1 of the '171 patent inherently, as shown above with respect to Ground 1.”). For the reasons discussed above, we are not persuaded that Mr. Miura's experiment confirms that DME is necessarily present in the second overhead of the '095 patent. Moreover, even if the experiment were sufficient to show that DME is necessarily present in the second overhead of the '095 patent, Petitioner presents no credible evidence to show that these experimental results apply equally to the '171 patent process and compositions. *See* Pet. 30; Ex. 1003 ¶ 95. Thus, we are not persuaded that Mr. Miura's experimental results establish that the process of the '171 patent discloses or suggests the presence of DME in the second overhead.

Petitioner also asserts—in its claim chart—that, during prosecution, the Applicants admitted that DME was formed when water was added in the distillation step of the '171 patent. Pet. 33. Based on this alleged admission, Petitioner concludes that the addition of water in the distillation step of the '171 patent “was the equivalent of increasing DME.” *Id.* (citing Ex. 1009,

20; Ex. 1003 ¶¶ 32, 73). This argument, however, takes Applicants' statement in the Response to Office Action out of context. Specifically, Petitioner omits the final two sentences of the section upon which it relies, which assert that "formation of DME is not favored," "would be unexpected," and is "not an inherent feature of the process." Ex. 1009, 20–21. In addition, in the same Response to Office Action, Applicants stated that a "sufficient amount" of water must be added to produce DME. *Id.* at 22, 23 ("[The '171 patent] fails to suggest adding water to the distillation column in an amount effect[ive] to produce DME."). Petitioner does not explain why this assertion is incorrect or why *the amount* of water added in the '171 patent was sufficient to form DME.⁴ Ex. 1001, 8:28–29. Thus, on this record, we are not persuaded that Applicants' statements during prosecution are tantamount to an admission that DME necessarily would be found in the second overhead of the '171 patent.

Based on the foregoing, we are not persuaded that Petitioner has established that DME is necessarily present in the overhead of the '171 patent or that it would have been obvious—in view of the '171 patent disclosure or knowledge in the art—to add DME (directly, or via reaction through addition of a sufficient amount of water) in the process of the '171 patent. Accordingly, Petitioner has not demonstrated a reasonable likelihood that claim 1 would have been obvious over the '171 patent.

⁴ Mr. Cooper concludes that the '171 patent discloses adding the "required amount" of water to produce DME. Ex. 1003 ¶ 73. Mr. Cooper does not explain, however, what the required amount of water would be, or how he arrived at this conclusion. Moreover, Mr. Cooper's analysis and conclusions are based on an assumption that Applicants had admitted that any addition of water would result in DME formation. *Id.* ¶ 72.

D. Obviousness of Claim 1 Over the '095 Patent and Akinori

Petitioner contends that claim 1 would have been obvious over the combination of the '095 patent and Akinori, a review paper directed to existing processes for the production of acetic acid from methanol. Pet. 35–36; Ex. 1013, 1. Akinori discloses that, during the production process of acetic acid, in addition to the primary conversion of methanol and carbon monoxide to acetic acid, several side reactions occur, including the production of DME. Ex. 1013, 8; Ex. 1003 ¶¶ 87–88. Akinori notes that “these side reactions are equilibrium reactions” and, therefore, the intermediates “ultimately change into acetic acid.” Ex. 1013, 8.

Patent Owner asserts that claim 1 is not rendered obvious by the '095 patent and Akinori because Akinori does not disclose, expressly or inherently, that DME is present in the second overhead of the '095 patent, and Petitioner provides no articulated reasoning explaining why one of ordinary skill in the art would have combined the '095 patent and Akinori. Prelim. Resp. 49–50, 52. We agree.

Although Akinori discloses that DME is produced as a side reaction, Petitioner provides no credible evidence to demonstrate that Akinori's intermediates necessarily would travel to, or be generated in, the second overhead of the '095 patent. Nor does Petitioner provide articulated reasoning with rational underpinning to explain why or how one of ordinary skill in the art would have sought to combine Akinori and the '095 patent to arrive at the method of claim 1.

Accordingly, Petitioner has not demonstrated a reasonable likelihood that claim 1 would have been obvious over the '095 patent and Akinori.

E. Obviousness of Claim 1 Over the '095 Patent and the '171 Patent

Petitioner further asserts that claim 1 would have been obvious over the '095 and '171 patents in combination. Pet. 36–39. This combination, however, relies upon the '171 patent process to disclose DME inherently in the second overhead. *Id.* at 37. As noted above, we are not persuaded that Petitioner has demonstrated that the presence of water in the distillation step of the '171 patent necessarily results in the presence of DME in the second overhead. Accordingly, we are not persuaded that Petitioner has demonstrated a reasonable likelihood that claim 1 would have been obvious over the combination of the '095 and '171 patents.

III. CONCLUSION

For the foregoing reasons, we determine that the information presented in the Petition does not demonstrate a reasonable likelihood that claim 1 of the '886 patent is unpatentable.

IV. ORDER

Accordingly, it is:

ORDERED that the Petition is denied and no trial is instituted.

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PETITIONER:

Jeffrey R. Lomprey
George C. Beck
Foley & Lardner LLP
daicel_ipr@foley.com

PATENT OWNER:

Justin Krieger
Troy Petersen
Joshua Pond
KILPATRICK TOWNSEND & STOCKTON LLP
jkrieger@kilpatricktownsend.com
tpetersen@kilpatricktownsend.com
jpond@kilpatricktownsend.com