UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

DAICEL CORPORATION,
Petitioner,

v.

CELANESE INTERNATIONAL CORPORATION,
Patent Owner.

Case IPR2015-00170
Patent 8,076,507 B2


GAUDETTÉ, Administrative Patent Judge.

DECISION

Denying Institution of Inter Partes Review
37 C.F.R. § 42.108
II. BACKGROUND

A. Related Matters

7,223,886 (Case No. IPR2014-01515), to which the ’507 patent claims priority. *Id.*

**B. The ’507 Patent (Ex.1002)**

The ’507 patent relates to a process for removing permanganate reducing compounds (“PRCs”) and alkyl iodides from intermediate streams during production of acetic acid by the carbynylation of methanol in the presence of a Group VIII metal carbynylation catalyst. Ex. 1002, 1:13–21; see also Pet. 4 (“[T]he process is a variation on the Monsanto process, … [which is] the production of acetic acid by the metal-catalyzed carbynylation of alkyl alcohols, such as methanol.”) (citing Ex. 1003 ¶ 18). PRCs include “acetaldehyde, acetone, methyl ethyl ketone, butyraldehyde, crotonaldehyde, 2-ethyl crotonaldehyde, and 2-ethyl butyraldehyde and the like, and the aldol condensation products thereof.” *Id.* at 2:32–35. According to the ’507 patent, even small amounts of carbonyl impurities, such as acetaldehyde, tend to poison the catalyst used in the production of vinyl acetate, the product most commonly produced from acetic acid. *Id.* at 2:42–45.

The ’507 patent discloses that, at the time of the invention, various prior art processes were available for removing PRCs produced during methanol carbynylation. *Id.* at 3:11–12. The ’507 patent describes U.S. Patent No. 5,625,095 (“the ’095 patent,” Ex. 1010, relied on by Petitioner in its patentability challenges) as directed to “a process for producing high purity acetic acid in which . . . an acetaldehyde concentration of 400 ppm or less is maintained in the reactor by using a single or multi-stage distillation process to remove acetaldehyde.” *Id.* at 3:33–37. According to the ’507 patent, a drawback of the ’095 patent process is that the reaction conditions increase formation of propionic acid, an undesirable by-product. *Id.* at 3:51–
The '507 patent also provides a detailed discussion of the process disclosed in commonly assigned U.S. Patent No. 6,339,171 ("Singh," Ex. 1011, also relied on by Petitioner in its patentability challenges), which is described as "a purification process in which the light ends overhead is distilled twice, in each case taking the acetaldehyde overhead and returning a methyl iodide rich residuum to the reactor," thereby "significantly reduc[ing] the undesirable impurities in the acetic acid product." Id. at 3:55–61. The Singh process is described in connection with Figure 1, below. Id. at 8:25–26.

In Figure 1, above, carbonylation reaction products enter splitter column 14 as stream 26. Id. at 7:54–59. First vapor phase acetic acid stream 28, containing methyl iodide, methyl acetate, acetaldehyde and other carbonyl components, exits splitter column 14. Id. at 7:60–61, 8:29–32. Stream 28 is directed to receiver decanter 16. Id. at 7:66–67. Exiting light
ends phase, stream 30 contains acetaldehyde, water, and acetic acid. *Id.* at 8:32–33. A portion of stream 30 is directed back to light ends column 14 as reflux stream 34, and the remainder enters distillation column 18 as stream 32 in about the middle of the column. *Id.* at 9:10–13. Distillation column 18 concentrates the aldehyde components of stream 32 into overhead stream 36 by separating water and acetic acid from the lighter components. *Id.* at 9:13–15. Stream 38, containing approximately 70% water and 30% acetic acid, is generally recycled to overhead decanter 16. *Id.* at 9:20–24. A portion of stream 38 is also used to form stream 50, discussed in more detail below. *Id.* at 10:16–19.

Second vapor phase 36 is condensed in overhead receiver 20 to form stream 40 containing “acetaldehyde, methyl iodide, methyl acetate, and methanol.” *Id.* at 9:33–37. A portion of stream 40 is returned to column 18 as reflux stream 42, and the remainder of stream 40 enters second distillation column 22 close to the bottom of the column. *Id.* at 9:37–40. Distillation column 22 separates the majority of the acetaldehyde from the methyl iodide, methyl acetate, and methanol in stream 40. *Id.* at 9:40–42.

The ’507 patent notes Singh’s disclosure that “during the reaction, and with the heating of column 22, higher molecular weight polymers of acetaldehyde form” and “tend to harden and adhere to the walls of the tower where their removal is cumbersome.” *Id.* at 9:66–10:7. “[T]o reduce the formation of these impurities, i.e., metaldehyde and paraldehyde and higher molecular weight polymers of acetaldehyde,” an inhibitor, such as methanol, water, and acetic acid are used individually or in combination, preferably in column 22. *Id.* at 10:9–15. According to the ’507 patent, “small amounts of water, methanol, acetic acid, or a combination thereof, do not interfere with
the reaction chemistry and practically eliminate the formation of polymers of acetaldehyde. Stream 50 is also preferably employed as an inhibitor since this material does not change the reactor water balance.” *Id.* at 10:24–29.

Stream 44, containing methyl iodide, methanol, methyl acetate, methanol, and water, exits the bottom of distillation column 22. *Id.* at 10:38–40. Stream 52 exits the top of distillation column 22, and is directed to a condenser, and then overhead receiver 24, where it exits as stream 54. *Id.* at 10:33–35. Part of stream 54 is used as reflux for distillation column 22, and the remainder is condensed and sent to first extractor 27 as stream 62. *Id.* at 10:37–38, 49–51. In the extractor, PRCs and alkyl iodides are extracted with water, preferably from an internal stream. *Id.* at 10:51–52. PRC-rich aqueous extract stream 64 then leaves extractor 17 from the top and is directed to waste treatment. *Id.* at 10:59–60. Raffinate stream 66, containing methyl iodide, exits the bottom of extractor 27. *Id.* at 10:60–62.

As depicted in Figure 1, in the prior art process of Singh, raffinate stream 66 is recycled to the reaction system, and ultimately to the reactor. Ex. 1011, 12:1–3. However, in the '507 patent process, raffinate stream 66 is directed to extractor 25 (shown in Figure 2) to extract additional PRCs and alkyl iodides using water, preferably from an internal stream. Ex. 1002, 10:63–66. PRC-rich aqueous extract stream 70 leaves extractor 25 from the top and is directed to waste treatment. *Id.* at 10:59–60. Raffinate stream 72, containing methyl iodide, exits extractor 25 from the bottom and “is normally recycled to the reaction system and ultimately to the reactor.” *Id.* at 11:6–9. As depicted in Figure 2, a portion of raffinate stream 72 is also combined with stream 40 entering second distillation column 22.
Appellants are said to have discovered that:

adding dimethyl ether (DME) to the feed to extractor 27 limits
the loss of methyl iodide in the extraction steps. The presence
of DME reduces the solubility of methyl iodide in water,
thereby reducing the amount of methyl iodide extracted into
aqueous extract streams 64 and 70 and lost in wastewater
treatment.

Id. at 11:31–37. “The required quantity of DME in stream 62 can be
obtained by adding water to column 22, for example to the feed 40 or reflux
50.” Id. at 11:45–47. According to the ’507 patent, “it is believed that this
water reacts with methyl acetate and/or methyl iodide in column 22 to form
methanol, which is then dehydrated in the presence of an acid catalyst (such
as HI) to form DME.” Id. at 11:49–52. “Any DME that is not extracted into
the aqueous extract streams 64 and 70 is recycled directly or indirectly to the
reaction system, where it reacts with carbon monoxide and water to form
acetic acid.” Id. at 11:52–56.

C. Illustrative Claim

Of the challenged claims, claim 1 is independent, and reads as
follows:

1. An improved method for reduction and/or removal of
permanganate-reducing compounds (PRC’s), C_{3–8} carboxylic
acids and C_{2–12} 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
phase 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.

**D. Evidence Relied Upon**

Petitioner’s patentability challenges are based on the following references:

<table>
<thead>
<tr>
<th>References</th>
<th>Patents/Printed Publications</th>
<th>Exhibit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Miura (“the ’095 Patent”)</td>
<td>US 5,625,095</td>
<td>1010</td>
</tr>
<tr>
<td>Singh</td>
<td>US 6,339,171 B1</td>
<td>1011</td>
</tr>
</tbody>
</table>


**E. Asserted Grounds of Unpatentability**

Petitioner challenges the patentability of claims 1–4, 6–11, and 13 of the ’507 patent based on the following grounds:

<table>
<thead>
<tr>
<th>Reference(s)</th>
<th>Basis</th>
<th>Claims challenged</th>
</tr>
</thead>
<tbody>
<tr>
<td>The ’095 patent</td>
<td>§102(b)</td>
<td>1–4, 6, 7, 10, 11, and 13</td>
</tr>
<tr>
<td>Singh</td>
<td>§103(a)</td>
<td>1–4, 7–11, and 13</td>
</tr>
<tr>
<td>Singh, the ’095 patent, and Akinori</td>
<td>§103(a)</td>
<td>1–4, 6–11, and 13</td>
</tr>
</tbody>
</table>
III. 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); In re Cuozzo Speed Techs., LLC., 113 U.S.P.Q.2d 1613, 1621 (Fed. Cir. 2015). At this stage of the proceeding, for purposes of this Decision, we determine no express construction of the claim language is required.

IV. ANALYSIS

A. Anticipation of Claims 1–4, 6, 7, 10, 11, and 13 by the ’095 Patent

1. The ’095 Patent (Ex. 1010)

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; see also Ex. 1002, 3:46–51 (describing the ’095 patent as teaching that managing reaction conditions to control formation of acetaldehyde in the reactor reduces formation of aldehyde by-products). The ’095 patent states that “[i]n the present invention, it is important in the reaction–acetic acid recovery system to carry out the reaction while keeping an acetaldehyde concentration in the reaction liquid at 400 ppm or less” to avoid formation of impurities. Id. at 5:56–59.

The ’095 patent discloses that acetaldehyde concentration in the reaction liquid can be controlled by managing reaction conditions, e.g., “increasing the hydrogen partial pressure, water concentration, and rhodium
catalyst concentration.” *Id.* at 5:62–6:3. However, a drawback is that by-production speed of propionic acid is increased. *Id.* at 6:9–11. The ’095 patent further notes that, at lower water contents, production of by-products such as carbon dioxide and propionic acid is reduced, however, trace impurities increase, and deteriorate the quality of the acetic acid product. *Id.* at 1:26–31. “These impurities include . . . carbonyl compounds such as acetaldehyde, butylaldehyde, crotonaldehyde, and 2-ethylcrotonaldehyde, aldol condensation products thereof, and alkyl iodides such as ethyl iodide, butyl iodide, and hexyl iodide. *Id.* at 1:44–49 (citing EP-A 487284).

The ’095 patent thus controls acetaldehyde concentration in the carbonylation reactor by using a combination of methods for controlling reaction conditions and methods for removing acetaldehyde from process liquid circulated into the carbonylation reactor. *Id.* at 6:12–19. Methods for removing acetaldehyde from the process liquid include distillation and/or extraction. *Id.* at 6:25–28. According to the ’095 patent,

> [i]nvestigations intensively made by the present inventors have resulted in finding that the generation and deposition of paraldehyde and metaldehyde which are the condensation products of acetaldehyde can be controlled, and methyl iodide can efficiently be separated from acetaldehyde by controlling top temperature, reflux tank temperature, and pressure or controlling top temperature and reflux tank temperature in the presence of alcohol in distilling a mixed liquid containing acetaldehyde and methyl iodide, and completing the present invention.

*Id.* at 8:12–21.

The ’095 patent describes the system and method for removing acetaldehyde and recovering the reaction products in connection with Figures 1 and 2, reproduced 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, the ’095 patent describes an example (“Example”) in which a reaction liquid having the following composition is
introduced into reactor 10: 14 wt% methyl iodide; 8 wt% water; 1.6 wt% methyl acetate; 5 wt% lithium iodide; 400 ppm rhodium catalyst and 70.9 wt% acetic acid. Ex. 1010, 12:9–12. As further shown in Figure 1, liquid products are withdrawn from 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 flasher 12 as bottom stream 13 and returned to reactor 10. Id. at 34–39. Overhead stream 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 crude acetic acid liquid, withdrawn via line 17, from methyl iodide, methyl acetate, and small amounts of water and acetic acid, withdrawn via overhead 20. Id. at 5:46–48, 6:43–44. When a sufficient amount of water is present, overhead 20 is typically 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. Lower phase 30, upper phase 32, or overhead 20 can be recirculated to reactor 10 via line 21. Id. 8:32–34.

The lower phase, the upper phase, or both, are introduced into 80 plate distillation column 40. Id. at 8:52–58. A methyl iodide stream is withdrawn via line 46 from the bottom of column 40 and recirculated to reactor 10 via line 21. Id. at 8:55–57. In the embodiment illustrated in Figure 2, distillate 44, obtained from the top of distillation column 40, is introduced into second distillation column 60. Id. at 8:59–60. However, the ’095 patent states that any number of distillation columns may be used. Id. at 8:45–48.

According to the ’095 patent, the inventors also found that the
undesirable deposition of metaldehyde, a condensation product of acetaldehyde, can be controlled by distilling in the presence of an alcohol, such as methanol. *Id.* at 10:3–7. In Example 5, for example, top withdrawn liquid 44 from distillation column 40 in Example 1 was introduced into distillation column 60 and methanol was charged via line 67 into distillation column 60. *Id.* at 17:46–47, 53–55. The compositions of the liquids charged to distillation column 60 and the exiting distillate and bottom liquid streams are shown in Table 9, reproduced below (*id.* at 18:1–19).

<table>
<thead>
<tr>
<th>TABLE 9</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Composition (wt %)</strong></td>
</tr>
<tr>
<td>Charged liquid</td>
</tr>
<tr>
<td>Methyl iodide</td>
</tr>
<tr>
<td>Methyl acetate</td>
</tr>
<tr>
<td>Water</td>
</tr>
<tr>
<td>Acetaldehyde</td>
</tr>
<tr>
<td>Paraldehyde</td>
</tr>
<tr>
<td>Metaldehyde</td>
</tr>
<tr>
<td>Alkane</td>
</tr>
<tr>
<td>Others</td>
</tr>
<tr>
<td>Methanol</td>
</tr>
</tbody>
</table>

2. **Miura Declaration (Ex. 1005)**

Mr. Miura, a named inventor of the '095 patent and employee of Petitioner, testified that, in 2011, he conducted an experiment (“Experiment 1”) to determine trace level contaminants present in the streams described in the '095 patent, and that “[t]he data from Experiment 1 was based upon [the] ‘Example’ and ‘Example 1’ as set forth in the '095 Patent.” Ex. 1005 ¶¶ 1, 3, 5, 7.

Mr. Miura testified that the volatile phase from the reactor was subjected to a splitter column to obtain a first overhead which was
condensed, and a part of the lower phase in the separator bath was distilled in a distillation column of 80 plates “in accordance with the ‘Distillation condition’ shown in ‘Example’ of the ’095 Patent.” *Id.* (citing Ex. 1002, 12:1–54). The top withdrawn liquid from the 80 plate distillation column was subjected to water extraction, and the extract was supplied to a subsequent distillation column. *Id.* ¶¶ 11–12.

The results of Mr. Miura’s analyses of the various Experiment 1 process streams are presented in Table 1 in his Declaration (*see id.* ¶ 13), reproduced below:
Mr. Miura testified that "as a result of analyzing the chemical constituents present in Experiment 1, [he] was able to determine that the process set forth in the '095 Patent necessarily results in 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." *Id.* ¶ 9.

Mr. Miura acknowledged "slight difference[s] in weight % of methyl iodide, water, acetic acid, lithium iodide, and rhodium" in the reaction liquid of Experiment 1 as compared to the '095 patent Example, but stated that because Experiment 1 showed the same reactants under similar conditions to

<table>
<thead>
<tr>
<th>Table 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Condensed and Separated Liquid Phases of the Overhead 20</td>
</tr>
<tr>
<td>The Upper Phase 32</td>
</tr>
<tr>
<td>Methyl Iodide (McI)</td>
</tr>
<tr>
<td>Methyl Acetate (MA)</td>
</tr>
<tr>
<td>H₂O</td>
</tr>
<tr>
<td>Acetic Acid (AC)</td>
</tr>
<tr>
<td>Acetaldehyde (AD)</td>
</tr>
<tr>
<td>Methanol</td>
</tr>
<tr>
<td>Dimethyl Ether (DME)</td>
</tr>
<tr>
<td>others</td>
</tr>
</tbody>
</table>

"ND" means "not detectable" by the quantitative analysis

the symbol "-" shows that the amount of the component was not measured
those used in the ’095 patent Example produced DME, “at least some DME will always be present under these conditions, regardless of any slight differences in the concentrations of the reactants.” *Id.* ¶ 21. Mr. Miura also acknowledged differences in extraction and distillation conditions, but explained why he believes these “few minor deviations” from the conditions used in the ’095 patent Example would not have affected the outcome with respect to the presence of DME in the overheads and methanol in the liquid charged to the 80 plate distillation column. *See id.* ¶¶ 21–24.

3. *Analysis*

Petitioner contends the ’095 patent describes the process as recited in independent claim 1 with the exception of explicitly stating that DME is present in second overhead stream. *See Pet.* 22. Petitioner notes the preamble of independent claim 1 is drafted in Jepson format and, as such, is a description of elements or steps that were known in the prior art. Pet. 10–11, 25–26. Petitioner contends dependent claim 10 of the ’507 patent recites that the first overhead comprises DME, and suggests that because the “first overhead” is recited in the Jepson preamble of claim 1, claim 10 amounts to an admission that DME is present in the feed directed to the distillation step in a Monsanto process. *See Pet.* 26. As acknowledged by Petitioner, the ’507 patent describes “a variation on the Monsanto process.” Pet. 4. Petitioner has not provided adequate evidence or explanation to support a contention that the elements or steps recited in the claim 1 preamble describe the particular variation of the Monsanto process described in the ’095 patent.

Petitioner also relies on the Miura Declaration to establish DME is present in second overhead stream of the ’095 patent process. *Id.* at 26–27. Patent Owner contends the experiments discussed in the Miura Declaration
“do not come remotely close to replicating the conditions and compositions of the Example and Example 1 of the ’095 patent,” and, therefore, are insufficient to support Petitioner’s position that DME is present inherently in the second overhead stream, as recited in claim 1. Prelim. Resp. 29. With respect to the reaction liquid, Patent Owner contends: (1) Miura’s use of a higher lithium iodide content would have been expected to increase side reactions; (2) Miura’s use of a higher rhodium catalyst content would have been expected to produce dramatically different reaction kinetics; and (3) Miura’s use of a lower water content would have been expected to result in a markedly different impurity profile. Id. at 30–31 (citing Ex. 2014, 1 5:38–41, 2 2:15–20, 3 and 8:21–254). Patent Owner contends these differences in


2 “The increase in lithium iodide content stabilizes the rhodium complex catalyst and increases the production rate of acetic acid, but also increases side reactions.”

3 “The increased rhodium content in the system, however, increases not only the production rate of acetic acid but also that of by-produced acetaldehyde. Acetic acid and acetaldehyde increase proportionally with increase in rhodium content. Specifically, acetaldehyde increases substantially proportionally with increase in acetic acid.”

4 “At low water contents under conventional reaction conditions, the rate of a water gas shift reaction decreases, the hydrogen partial pressure decreases, and by-production of condensation reaction products of acetaldehyde increases to thereby impair the quality of acetic acid.”
the reaction liquids of Miura’s Experiment 1 and the ’095 patent Example would be expected to result in different liquid compositions being charged into the respective 80 plate distillation columns of Experiment 1 and the Example. *Id.* at 31–32. In the table reproduced below, Patent Owner provides a comparison of the liquid compositions charged to the 80 plate distillation columns in the ’095 patent Example and Miura’s Experiment 1. *Id.* at 32.

### Charged Liquid Compositions

<table>
<thead>
<tr>
<th>Component</th>
<th>’095 patent (Ex. 1010, 12:20:28)</th>
<th>Miura Declaration (Ex. 1005, ¶13)</th>
<th>% Relative to ’095 patent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetaldehyde</td>
<td>0.07 wt.%</td>
<td>0.14 wt.%</td>
<td>200%</td>
</tr>
<tr>
<td>Methyl iodide</td>
<td>89.4 wt.%</td>
<td>88.0 wt.%</td>
<td>98%</td>
</tr>
<tr>
<td>Water</td>
<td>0.5 wt.%</td>
<td>0.53 wt.%</td>
<td>106%</td>
</tr>
<tr>
<td>Methyl acetate</td>
<td>5.0 wt.%</td>
<td>9.3 wt.%</td>
<td>186%</td>
</tr>
<tr>
<td>Acetic acid</td>
<td>5.0 wt.%</td>
<td>1.8 wt.%</td>
<td>36%</td>
</tr>
<tr>
<td>Paraldehyde</td>
<td>0 wt.%</td>
<td>Unknown</td>
<td>Unknown</td>
</tr>
<tr>
<td>Alkanes</td>
<td>0.01 wt.%</td>
<td>Unknown</td>
<td>Unknown</td>
</tr>
<tr>
<td>Methanol</td>
<td>None reported</td>
<td>0.11 wt.%</td>
<td>Unknown</td>
</tr>
<tr>
<td>Others</td>
<td>0.02 wt.%</td>
<td>0.16 wt.%</td>
<td>800%</td>
</tr>
<tr>
<td>DME</td>
<td>None reported</td>
<td>0.0048 wt.%</td>
<td>100.0048 wt.%</td>
</tr>
<tr>
<td>Total</td>
<td>100.00 wt.%</td>
<td>100.0048 wt.%</td>
<td>Unknown</td>
</tr>
</tbody>
</table>

We agree with Patent Owner that Petitioner’s evidence is insufficient to support its contention that Miura’s process inevitably results in a second overhead stream comprising DME, as recited in independent claim 1.

With respect to differences in content of the reaction liquids in the ’095 patent Example and Miura’s Experiment 1,\(^5\) both the disclosure in U.S.

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\(^5\) *Compare* Ex. 1005 ¶11 (describing the Experiment 1 reaction liquid 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 as containing 14 wt% methyl iodide, 8 wt% water, 1.6 wt% methyl acetate, 70.9 wt% acetic acid).
Patent No. 7,683,212 B2, referenced by Patent Owner (Pet. 25–26; see
footnotes 1–4 supra), and disclosure in the ’095 patent (see Ex. 1010, 1:26–31, 44–49, 5:62–6:3, 6:9–11 supra) indicate that differences in water and
catalyst contents affect the impurity content of the reaction product. As
observed by Patent Owner, the reaction liquid of Miura’s Experiment 1
contained more than twice the amount of rhodium and about 34% of the
amount of water used in the ’095 patent Example. See Prelim. Resp. 25.
Mr. Miura has not provided support for his characterization of these
differences (as well as the acknowledged differences in methyl iodide, acetic
acid, and lithium iodide) as “slight difference[s] in weight %.” Ex. 1005 ¶
21. Mr. Miura has not explained why the reactor contents of Experiment 1
did not more closely approximate the reactor contents in the ’095 patent
Example, or why “it was not feasible to run the equipment in exactly the
same way as was used in the ’095 patent example.” Id. ¶ 10.

Mr. Miura testified that Experiment 1 was “based on the disclosure of
the reaction liquid described in the ’095 Patent at column 4,” id. ¶ 10, and
that “even if not exactly the same as in the Example, the composition
(proportion of the components) of the reaction liquid used in Experiment 1
[was] 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. Column 4 of the ’095 patent lists numerous components and ranges
therefor which could be used in the reaction liquid. Ex. 1010, 4:16–58.
Petitioner has not explained sufficiently why one of ordinary skill in the art,
upon considering the ’095 patent, would have chosen the combination of

acid, 5 wt% lithium iodide, and 400 ppm rhodium).
reaction components and specific concentrations used in Miura Experiment 1, i.e., the only combination shown to produce “a second overhead stream comprising . . . dimethyl ether” (Ex. 1002, claim 1).

In view of the above-noted disclosure in U.S. Patent No. 7,683,212 B2 and in the ’095 patent suggesting that differences in concentrations of reactants would affect the reactant product, we are not persuaded by Mr. Miura’s unsupported statements that at least some DME would be present in the reaction product of the ‘095 patent Example because the reaction liquid contains the same constituents used in Miura’s Experiment 1, Ex. 1005 ¶ 21. 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 expressing opinions as to fact and conclude that the lack of factual corroboration warrants discounting the opinions expressed in the declarations); cf. Ashland Oil, Inc. v. Delta Resins & Refractories, Inc., 776 F.2d 281, 294 (Fed. Cir. 1985) (“Lack of factual support for expert opinion going to factual determinations . . . may render the testimony of little probative value in a validity determination.”).

For the foregoing reasons, Mr. Miura’s declaration lacks persuasive merit in showing claim 1 is inherently anticipated by the ’095 patent.

In addition to the Miura Declaration, Petitioner also relies on the Cooper Declaration. Pet. 21. We agree with Patent Owner that Petitioner merely cites to the Cooper Declaration in support of conclusory statements regarding the presence of DME without discussing the underlying evidence in support thereof. Mr. Cooper’s more detailed analysis cannot be
incorporated by reference into the Petition. 37 C.F.R. § 42.6(a)(3)
(“Arguments must not be incorporated by reference from one document into
another document.”); see also Cisco Sys. v. C-Cation Techs., IPR2014-
00454 (Paper 12) (Aug. 29, 2014) at 7–10 (refusing to consider arguments
made in the declaration and not the petition). Moreover, we note that in
concluding that DME is present inherently in the second overhead of the
’095 patent process, Mr. Cooper relies on Miura Experiment 1 (see, e.g., Ex.
1003 ¶ 97), which we found unpersuasive for the reasons discussed above.

For the foregoing reasons, Mr. Cooper’s declaration lacks persuasive
merit in showing claim 1 is inherently anticipated by the ’095 patent.

“Inherent anticipation requires more than mere probabilistic
inherency.” Crown Packaging Technology, Inc. v. Ball Metal Beverage
Container Corp., 635 F.3d 1373, 1383 (Fed. Cir. 2011); see also Cont’l Can
Co. USA, Inc. v. Monsanto Co., 948 F.2d 1264, 1269 (Fed. Cir. 1991)
(Inherent anticipation “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.”); Glaxo Inc. v. Novopharm Ltd., 52 F.3d
1043, 1047 (Fed.Cir.1995) (holding that even though the defendant's experts
reproduced a prior art method “thirteen times and each time they made [the
claimed] crystals,” the patentee's chemists twice produced different crystals
from the same method, thus precluding inherency). For the above reasons,
Petitioner’s evidence falls short of demonstrating a reasonable likelihood
that it would prevail in showing that claim 1, as well as its dependent claims
2–4, 6, 7, 10, 11, and 13, are inherently anticipated by the ’095 patent.
B. Obviousness of Claims 1–4, 7–11, and 13 Over Singh

1. Singh (Ex. 1011)

Singh discloses a “process for reducing and/or removing precursors of permanganate reducing compounds and alkyl iodides from intermediate streams during the formation of acetic acid by carbonylation processes.” Ex. 1011, 1:18–22. Singh’s process is illustrated in Figure 1 of the ’507 patent, Ex. 1002, 1:10–13, discussed above.

2. Analysis

Petitioner contends the only difference between challenged claim 1 and Singh is that Singh does not expressly disclose the presence of DME in a second overhead stream and multiple extractions. Pet. 36.

As an initial matter, we note Petitioner states that the Miura Declaration establishes the presence of DME in the second overhead. Id. at 42. Mr. Miura, however, does not assert that his experiments apply equally to the process of the ’171 patent, nor has Petitioner provided credible evidence to explain why experiments conducted according to the teachings of the ’095 patent necessarily indicate the presence of DME in the second overhead of ’171 patent. Thus, we are not persuaded Mr. Miura’s experiments provide evidence that DME is necessarily present in the second overhead of Singh’s process.

Petitioner argues the Singh process “(as depicted in Figure 1 and described in columns 9-12) [of Singh] is identical to the system employed in the ’507 Patent.” Id. at 37 (citing Ex. 1003 ¶ 75). “Accordingly, if DME is produced in the acetic acid production of the ’507 Patent, so too must it be produced in [Singh].” Id. Petitioner argues further that Singh’s addition of water to the distillation column to suppress meta-and paraldehyde formation

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must produce DME, relying on an alleged admission made by Patent Owner in a response to office action filed during prosecution of the ’507 patent. *Id.* at 41, 43 (citing Ex. 1009, 20 (“DME is formed when water is added to the distillation column.”)).

We agree with Patent Owner that Petitioner mischaracterizes the statement made during prosecution, which, taken in context, requires the addition of “a sufficient amount of water” (Ex. 1009, 22 (emphasis added)). See Prelim. Resp. 48. We further agree with Patent Owner that, although Singh indicates small amounts of water can be added to eliminate formation of polymers of acetaldehyde, Singh states that “[w]ater as an inhibitor is the *least preferred* solvent of inhibition since large quantities are generally needed to be an effective inhibitor” (Ex. 1011, 11:37–40 (emphasis added)). *Id.* at 46–48.

Petitioner has not provided adequate argument or evidence to support its contention that the amount of water present in Singh’s process, e.g., through addition as an inhibitor to column 22, would provide a sufficient amount of water to produce “a second overhead stream comprising . . . dimethyl ether” as recited in claim 1. Accordingly, Petitioner has not demonstrated a reasonable likelihood that it would prevail in showing the unpatentability of at least one of claims 1–4, 7–11, and 13 as obvious over Singh.

**C. Obviousness of Claims 1–4, 6–11, and 13 Over Singh, the ’095 Patent, and Akinori**

Petitioner contends Singh and the ’095 patent “are directed to reducing PRCs within the product stream from the Monsanto process, and Akinori provides guidance to the person of ordinary skill in the art as to the
types of side reactions that may take place within the Monsanto process reactor(s).” Pet. 50. Petitioner has not explained how Akinori cures the above-noted deficiencies in its challenges as to independent claim 1 based on Singh and the ’095 Patent, individually. See, e.g., id. at 51 (“[T]o the extent [Singh and the ’095 Patent] fail to expressly acknowledge the presence of DME, Akinori provides a basis for the person of ordinary skill in the art to readily understand that DME is in equilibrium with other components of the systems.”). Accordingly, for the reasons stated above, Petitioner has not demonstrated a reasonable likelihood that it would prevail in showing at least one of claims 1–4, 6–11, and 13 would have been unpatentable over Singh and/or the ’095 Patent in combination with Akinori.

V. CONCLUSION

For the above reasons, Petitioner has not demonstrated there is a reasonable likelihood it would prevail in showing the unpatentability of at least one of the challenged claims.

VI. ORDER

For the reasons given, it is

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