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Paper 85
Entered: June 23, 2016

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

DAICEL CORPORATION,
Petitioner,

v.

CELANESE INTERNATIONAL CORPORATION,
Patent Owner.

Case IPR2015-00173
Patent 8,076,507 B2

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

GAUDETTE, *Administrative Patent Judge*.

FINAL WRITTEN DECISION
35 U.S.C. § 318(a) and 37 C.F.R. § 42.73

Daicel Corporation ("Petitioner") filed a Petition (Paper 2, "Pet.") requesting institution of an *inter partes* review of claims 58–60, 67, 69, and 71–73 of U.S. Patent No. 8,076,507 B2 (Ex. 1002, "the '507 patent"). Celanese International Corporation ("Patent Owner") filed a Preliminary Response (Paper 11, "Prelim. Resp."). On April 1, 2015, we entered a Decision Denying Institution of *Inter Partes* Review. Paper 13. We subsequently granted-in-part Petitioner's Request for Rehearing (Paper 14), and instituted an *inter partes* review of claims 58–60, 67, 69, and 71–73 on June 26, 2015 (Paper 15, "Dec. on Inst." or "Institution Decision"). We denied Patent Owner's Request for Rehearing (Paper 17) of our Institution Decision. Paper 24.

After institution, Patent Owner filed a Response (Paper 68, "PO Resp.")[1], and Petitioner filed a Reply to the Patent Owner Response (Paper 61, "Pet. Reply").

Patent Owner filed a Motion to Exclude Evidence. Paper 74; *see also* Paper 77 (Petitioner's Opposition) and Paper 79 (Patent Owner's Reply). Patent Owner also filed a Motion for Observation. Paper 73; *see also* Paper 78 (Petitioner's Response).

Oral argument was held on March 29, 2016. A transcript of the oral hearing ("Tr.") has been entered into the record. *See* Paper 84.

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[1] Pursuant to a motion filed by Petitioner, a modified protective order was entered (Ex. 1071), and certain papers and exhibits have been filed under seal. *See* Paper 64. Paper 68 is a redacted version of Patent Owner's Response, the complete version having been filed under seal as Paper 83. Citations in this Final Written Decision are to non-confidential portions of Patent Owner's Response. As such, all citations are to Paper 65.

We have jurisdiction under 35 U.S.C. § 6(c). This is a Final Written Decision, issued pursuant to 35 U.S. C. § 318(a) and 37 C.F.R. § 42.73.

For the reasons discussed below, we determine Petitioner has not met its burden to show by a preponderance of the evidence that claims 58–60, 67, 69, and 71–73 of the ’507 patent are unpatentable.

I. BACKGROUND

A. Related Matters

Petitioner also requested, and we instituted, *inter partes* reviews of claims 1–4, 6–11, and 13 (Case No. IPR2015-00170) and claims 42–44, 47–51, 53, and 55–57 (Case No. IPR2015-00171) of the ’507 patent. Oral arguments in IPR2015-00170 and IPR2015-00171 were consolidated with the oral argument in the present *inter partes* review. Final decisions in the two related *inter partes* reviews are issued concurrently with this final decision.

Petitioner previously filed a petition for *inter partes* review of the ’507 patent, challenging claims 25–28, 31–35, 37, and 39–41 (Case No. IPR2014-01514), and a petition for *inter partes* review of U.S. Patent No. 7,223,886 (Ex. 1001, “the ’886 patent”) (Case No. IPR2014-01515), to which the ’507 patent claims priority. *Id*. We denied these petitions.

B. The References

The pending grounds of unpatentability in this *inter partes* review are based on the following references:

References	**Patents/Printed Publications**	**Exhibit**
Miura (“the ’095 Patent”)	US 5,625,095	1010
JP ’712	Japanese Patent Application No. 2000-72712 (English Translation)	1020

C. The Instituted Ground of Unpatentability

We instituted the instant *inter partes* review of claims 58–60, 67, 69, and 71–73 of the ’507 patent based on the following grounds of unpatentability (Dec. on Inst. 12):

Grounds	Reference(s)	Basis[2]	Claims Challenged
1	The ’095 patent	§ 102(b)	58–60, 67, 69, 71, and 72
2	The ’095 patent and JP ’712	§ 103(a)	73

II. EXPERT TESTIMONY

Petitioner relies on the testimonies of William C. Jones and Hiroyuki Miura in support of its patentability challenge.[3] Patent Owner relies on the testimony of Dr. Richard Porcelli.

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[2] The Leahy-Smith America Invents Act, Pub. L. No. 112-29, 125 Stat. 284 (2011) (“AIA”), took effect on March 16, 2013, and amended 35 U.S.C. §§ 102 and 103. Because the ’507 patent has an effective filing date before the effective date of the applicable AIA amendments, we refer to the pre-AIA versions of 35 U.S.C. §§ 102 and 103.

[3] Petitioner previously relied on the declaration testimony of Jeremy Cooper (Ex. 1003). About two months after institution of trial, however, Petitioner advised the Board that Mr. Cooper had become gravely ill, and requested the Board’s approval to substitute a new expert for Mr. Cooper. *See* Paper 25, 2. The Board authorized the filing of a declaration by Mr. Jones (Ex. 1039), based on Petitioner’s showing that this declaration was substantially the same as Mr. Cooper’s declaration, with the only substantive differences being the respective credentials and qualifications of the two experts. *See* Paper 28, 2–3. Accordingly, we treat any citation to Exhibit 1003 in the Petition as a citation to Exhibit 1039.

To testify as an expert under Federal Rule of Evidence (FRE) 702, a person must be "qualified in the pertinent art." *Sundance, Inc. v. DeMonte Fabricating Ltd.*, 550 F.3d 1356, 1363–64 (Fed. Cir. 2008).

A. William C. Jones

Mr. Jones was cross-examined on the subject matter of his first declaration (Ex. 1039), and a transcript of the testimony was filed as Exhibits 2026 and 2027. Mr. Jones also executed a declaration in support of Petitioner's Reply (Ex. 1060, "reply declaration"). Mr. Jones was cross-examined on the subject matter of this reply declaration, and a transcript of the testimony was filed as Exhibit 2040.

Mr. Jones provided the following information and declaration testimony that is relevant to his qualifications to testify as an expert: He earned a Masters of Science in Chemical Engineering in 1969. Ex. 1038, 1. He also holds a Bachelor of Science in Chemical Engineering degree and a Masters of Science in Engineering Administration degree. *Id.* Mr. Jones is a chemical engineer and has "more than 40 years of experience in industrial-scale chemical processing, including the industrial-scale production of acetic acid and acetic anhydride. Ex. 1039 ¶¶ 1–2. He is retired from Eastman Chemical, where he worked "in R&D and operations management for production of acetic acid and acetyl derivatives[,] . . . includ[ing] pilot plant operation for production of acetic acid via methyl acetate carbonylation, and the design, construction, commissioning, and operation of acetic acid plants for Eastman and later for SIPCHEM (Saudi International Petrochemical Company)." *Id.* ¶¶ 1, 3. Mr. Jones also testified that he is a "named inventor on two patents/patent applications, both involving the process of methanol carbonylation." *Id.* ¶ 4.

Based on our review of Mr. Jones's education and work experience, we find Mr. Jones is qualified to testify as an expert under FRE 702.

B. Hiroyuki Miura

Mr. Miura executed a declaration in support of the Petition (Ex. 1005, "the Miura Declaration"). Mr. Miura was cross-examined on the subject matter of his declaration, and a transcript of the testimony was filed as Exhibit 1046.[4]

Mr. Miura provided the following declaration testimony that is relevant to his qualifications to testify as an expert: He earned a Masters of Science degree in 1983 from the Department of Industrial Organic Chemistry at Muroran Institute of Technology. Ex. 1005 ¶ 2. Mr. Miura is a senior manager and in charge of chemical process engineering at Daicel Corporation ("Daicel"). *Id.* ¶ 1. He has worked for Daicel since 1983 and his "research activities at Daicel have included the research of catalysis and process development for the manufacture of acetic acid." *Id.* ¶ 3. Mr. Miura is a named inventor on the '095 patent, as well as on numerous patent applications and issued patents. *Id.* ¶ 5.

Based on our review of Mr. Miura's education and work experience, we find Mr. Miura is qualified to testify as an expert under FRE 702.

[4] Exhibit 1046 is a redacted version of Mr. Miura's cross-examination testimony, the complete version having been filed under seal as Ex. 1045. Citations in this Final Written Decision are to non-confidential portions of Mr. Miura's cross-examination testimony. As such, all citations are to Exhibit 1046.

C. Richard Porcelli

Dr. Porcelli executed a declaration (Ex. 2029, "the Porcelli Declaration")[5] in support of Patent Owner's Response. Dr. Porcelli was cross-examined on the subject matter of his declaration, and a transcript of the testimony was filed as Exhibit 1065. [6]

Dr. Porcelli provided the following declaration testimony that is relevant to his qualifications to testify as an expert: He earned a Doctorate in Engineering Science in 1975, and he holds Masters and Bachelor of Science degrees in chemical engineering. Ex. 2029 ¶ 2. He has been an independent consultant for the worldwide chemical, refining and bioprocessing industries since 1986, and, during that time, has "been involved in evaluating various industrial R&D programs aimed at developing carbonylation and synthesis gas processes." *Id.* ¶¶ 4, 10. From 1970–1986, he worked full-time for the Halcon-Scientific Design Co. ("Halcon"), where his work focused on conceptualization, economic/technical evaluation, research and development, and commercialization of processes, including "rhodium-catalyzed carbonylation

[5] Patent Owner filed both complete and redacted versions of Dr. Porcelli's declaration testimony as Ex. 2029. Citations in this Final Written Decision are to non-confidential portions of Dr. Porcelli's declaration testimony. As such, all citations are to the public (redacted) version of Exhibit 2029.

[6] Exhibit 1065 is a redacted version of Dr. Porcelli's cross-examination testimony, the complete version having been filed under seal as Ex. 1064. Citations in this Final Written Decision are to non-confidential portions of Dr. Porcelli's cross-examination testimony. As such, all citations are to Exhibit 1065.

of dimethyl ether to acetic anhydride/acetic acid, rhodium-catalyzed carbonylation of methyl acetate to acetic anhydride/acetic acid, and nickel-catalyzed carbonylation of methanol to acetic acid." *Id.* ¶¶ 4–5. Dr. Porcelli is a named inventor on eight issued U.S. patents, six of which are related to carbonylation systems to form acetic acid/anhydride and other acetyls. *Id.* ¶ 7.

Based on our review of Dr. Porcelli's education and work experience, we find Dr. Porcelli is qualified to testify as an expert under FRE 702.

III. THE '507 PATENT (EX. 1002) AND THE PRIOR ART

A. Representative Claim

The '507 patent relates "to the production of acetic acid using a low water process with an iodide salt as a catalyst stabilizer." Ex. 2029 ¶ 26; *see also* Ex. 1039 ¶ 52 ("The '507 Patent does not specify a reactor water concentration, but . . . impl[ies] that . . . [low] water concentrations [are] envisaged."). Of the challenged claims, claim 58 is independent, and reads as follows:

> 58. A process for separating a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and at least one permanganate reducing compound (PRC), said process comprising the steps of:
>
> (a) distilling the mixture to separate the mixture into a plurality of streams, at least one of said streams being a PRC enriched overhead stream comprising dimethyl ether; and
>
> (b) extracting the PRC enriched overhead stream with water to form a first raffinate and a first aqueous extract stream comprising said at least one PRC; followed by extracting the first raffinate with water to fern [sic, form] a second

> raffinate and a second aqueous extract stream comprising said at least one PRC.

Ex. 1002, 16:13–26.

B. The Prior Art

In the background of the invention section, the ’507 patent discloses that one of the most useful processes for synthesizing acetic acid is the carbonylation of methanol with carbon monoxide using a rhodium catalyst and methyl iodide catalyst promotor, as taught in U.S. Patent 3,769,329, issued to Paulik et al. on October 30, 1973 (Ex. 2037, “Paulik”). Ex. 1002, 1:24–31. Paulik, filed in 1970, discloses that a disadvantage of prior art carbonylation processes “is their relatively low level of activity[,] . . . requir[ing] higher catalyst concentrations, longer reaction times, and higher temperatures.” Ex. 2037, 2:14–18. The higher temperatures, in turn, produce undesirable byproducts “comprising substantial amounts of ethers, aldehydes, higher carboxylic acids, carbon dioxide, methane and water.” *Id.* at 2:21–30. Paulik specifically notes that “significant quantities of the undesirable byproduct, dimethyl ether, are . . . formed in the presence of the cobalt catalyst system.” *Id.* at 12:51–53; *see id.* at 14:50–15:8 (Examples 6 and 7, wherein 50 mole percent and 60 mole percent, respectively, of methanol feedstocks were converted to dimethyl ether using a cobalt catalyst system). Paulik explains that dimethyl ether “attains a very high partial pressure at the reaction conditions employed, and necessitates the use of a high total pressure reaction system to maintain the necessary partial pressure of carbon monoxide for reaction to form carboxylic acids.” *Id.* at 12:54–58.

Paulik describes several exemplary embodiments of the invention wherein a methanol carbonylation using a rhodium catalyst was conducted

in a batch reactor. *See generally*, *id.* at 12:3–22:75. Paulik discloses the use of gas chromatography to detect the presence of "undesirable byproducts such as aldehydes, dimethyl ether, higher boiling carboxylic acids, methane, carbon dioxide, etc." *Id.* at 13:47–49, 15:32–34; *see id.* at 14:35–37, 16:71–74. In each of Examples 1–5, 8–18, and 22, Paulik discloses that these byproducts either were not detected or were detected in insubstantial amounts. *See id.* at 12:3–14:49, 15:9–18:75, 20:13–31. In Examples 20 and 21, Paulik describes simulating "a continuous reactor to produce acetic acid from methanol while maintaining equilibrium recycle streams of the ester (methyl acetate) and water, and resulting in a net conversion of the methanol feedstock to the desired acetic acid product while maintaining . . . equilibrium conditions." *Id.* at 19:65–71; *see id.* at 19:74–20:5. Paulik again reports that "[n]o substantial amounts of byproducts such as aldehydes, dimethyl ether, higher boiling carboxylic acids, methane or carbon dioxide [were] formed." *Id.* at 19:50–52. In Example 19, Paulik describes charging a batch reactor with a methanol feedstock containing 10 wt. percent dimethyl ether" (*id.* at 19:2–5) to "demonstrate[] the effect of a mixed feedstock in which an ether is present in the alcohol," e.g., "the commercial alcohol-ether product from a methanol plant" (*id.* at 19:24–27). Paulik reports "[n]o substantial amounts of byproducts such as aldehydes, dimethyl ether, higher boiling carboxylic acids, methane, or carbon dioxide [were] formed." *Id.* at 19:19–21. Paulik does not quantify the term "insubstantial" or "no substantial" amount as used in connection with the production of byproducts.

Dr. Porcelli testified that Paulik exemplifies a process commonly known as the "Monsanto process." Ex. 2029 ¶ 32. The Monsanto process

uses at least 14% water in the reaction liquid, and is considered a high water process. Ex. 2029 ¶ 27; Ex. 2026, 67:18–20; *see also* Ex. 1002, 1:54–56; Ex. 2038, 1:61–64. Akinori discloses that the main reaction in the Monsanto process is represented by the following formula (Ex. 1013, 7):

$$CH_3OH + CO \xrightarrow[\text{Co-catalyst}]{\text{Rhodium catalyst}} CH_3COOH \quad (1)$$

and that the following equilibrium side reactions occur, the intermediates ultimately changing into acetic acid (*id.* at 8):

$$2\,CH_3OH \rightleftarrows CH_3OCH_3 + H_2O \quad (2)$$

$$CH_3OH + CH_3\underset{\underset{O}{\|}}{C}OH \rightleftarrows CH_3\underset{\underset{O}{\|}}{C}OCH_3 + H_2O \quad (3)$$

$$CH_3OH + HI(aq) \rightleftarrows CH_3I + H_2O \quad (4)$$

Akinori does not mention specifically the production of dimethyl ether as a byproduct, but states, in general, that the amount of the byproduct produced in the Monsanto process is extremely small. *Id.* (stating that generation of byproducts "such as acetaldehyde, ethanol,[] [p]ropionaldehyde, propionic acid, butyl aldehyde, and butanol" is "almost negligible").

The '507 patent identifies several patents that are said to describe improvements to the conventional Monsanto process, e.g., U.S. Patent 5,001,259, issued to Smith et al. on March 19, 1991 (Ex. 2038, "Smith"). Ex. 1002, 1:40–47; *see also id.* at 2:11–12 (incorporating Smith by reference). Dr. Porcelli testified that Smith describes the Celanese "low water" carbonylation process. Ex. 2029 ¶¶ 32–33.

According to Smith, "[t]he general thrust of Paulik's teachings is . . . that a substantial quantity of water helps in attaining an adequately high reaction rate[, and] . . . that reducing the water content leads to the production of ester product as opposed to carboxylic acid." Ex. 2038, 1:52–57. Smith discloses that "about 14-15 wt% water is present in the reaction medium of a typical acetic acid plant using [Paulik's] technology" (*id.* at 1:61–63), and separating the acetic acid from the appreciable quantity of water used in the reaction medium of Paulik's process "involves substantial expenditure of energy in distillation and/or additional processing steps" (*id.* at 1:66–68). Smith also discloses that in Paulik's process, the catalyst tends to precipitate out of the reaction medium, especially during the course of distillation operations when carbon monoxide is reduced, and that this tendency increases as water content of the reaction medium is decreased. *Id.* at 2:10–17. Smith is said to have discovered that "catalyst stability and the productivity of the carbonylation reactor can be maintained at surprisingly high levels, even at very low water concentrations in the reaction medium . . . by maintaining in the reaction medium, along with a catalytically-effective amount of rhodium, at least a finite concentration of water" (*id.* at 4:25–34), i.e., "as low as about 0.1% wt" (*id.* at 4:59), methyl acetate and methyl iodide, and a specified concentration of iodide ion, preferably lithium iodide, present as a simple salt, that is over and above the iodide content which is present as methyl iodide or other organic iodide (*id.* at 4:35–41). *See also* Ex. 1002, 1:50–2:7. Smith discloses that in the inventive low-water method there is a substantial reduction in the rate of formation of the undesirable reaction byproducts propionic acid, hydrogen and carbon dioxide, as compared to the relatively high-water operating conditions of the prior art

carbonylation processes. Ex. 2038, 13:1–10. Smith does not discuss the presence or absence of dimethyl ether, or of any other byproducts. *See generally*, *id.* at 4:10–18:68.

According to the ’507 patent,

> although a low water carbonylation process for producing acetic acid reduces such by-products as carbon dioxide, hydrogen, and propionic acid, the amount of other impurities, present generally in trace amounts, is also increased, and the quality of acetic acid sometimes suffers when attempts are made to increase the production rate by improving catalysts, or modifying reaction conditions.

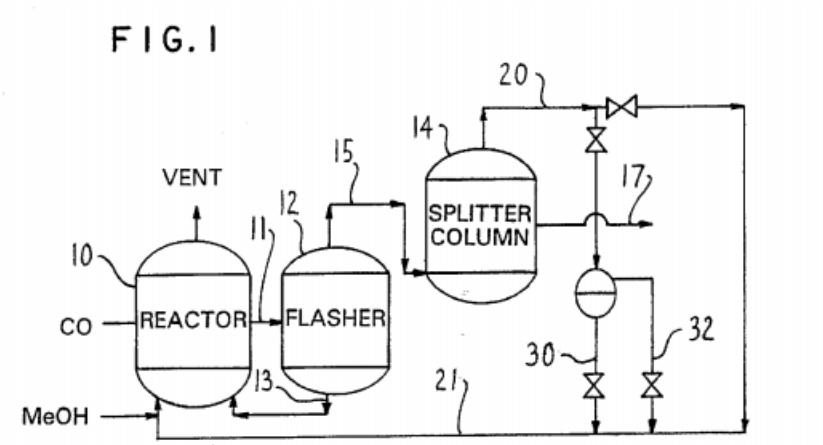
Ex. 1002, 2:13–19. The ’507 patent discloses that the presence of impurities that decrease permanganate time is objectionable because “a sufficient permanganate time is an important commercial test, which the acid product must meet to be suitable for many uses.” *Id.* at 3:2–6. According to the ’507 patent, “impurities that decrease the permanganate time of . . . acetic acid include carbonyl compounds and unsaturated carbonyl compounds” (*id.* at 2:22–24), such as “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). The ’507 patent discloses that acetaldehyde is particularly problematic because it can react with iodide catalyst promoters to form multi-carbon alkyl iodides, e.g., ethyl iodide, propyl iodide, butyl iodide, pentyl iodide, hexyl iodide and the like, that tend to poison the catalyst used in the production of vinyl acetate, the product most commonly produced from acetic acid.” *Id.* at 2:37–45.

The ’507 patent identifies several patents, including the ’095 patent, U.S. Patent No. 6,143,930 (Ex. 2006, “the ’930 patent”), and U.S. Patent No. 6,339,171 (Ex. 1011, “Singh”), a continuation-in-part of the ’930 patent (Ex. 1011, at [63]), which are said to describe carbonylation processes for producing an acetic acid product having reduced quantities of carbonyl impurities. *See* Ex. 1002, 3:11–67.

1. The ’095 Patent (Ex. 1010)

The ’095 patent, filed June 2, 1995, and issued April 29, 1997, identifies Petitioner as the assignee and Mr. Miura as one of the inventors. Ex. 1010, at [22], [45], [73], [75]. 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.” *Id.* at 1:14–18. The ’095 patent discloses that at lower water contents, production of byproducts such as carbon dioxide and propionic acid is reduced, however, trace impurities such as acetaldehyde increase, and deteriorate the quality of the acetic acid product. *Id.* at 1:26–31, 44–49. The ’095 patent does not describe dimethyl ether as a byproduct or impurity in the process, or otherwise mention its presence or absence.

The ’095 patent describes controlling 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. 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:



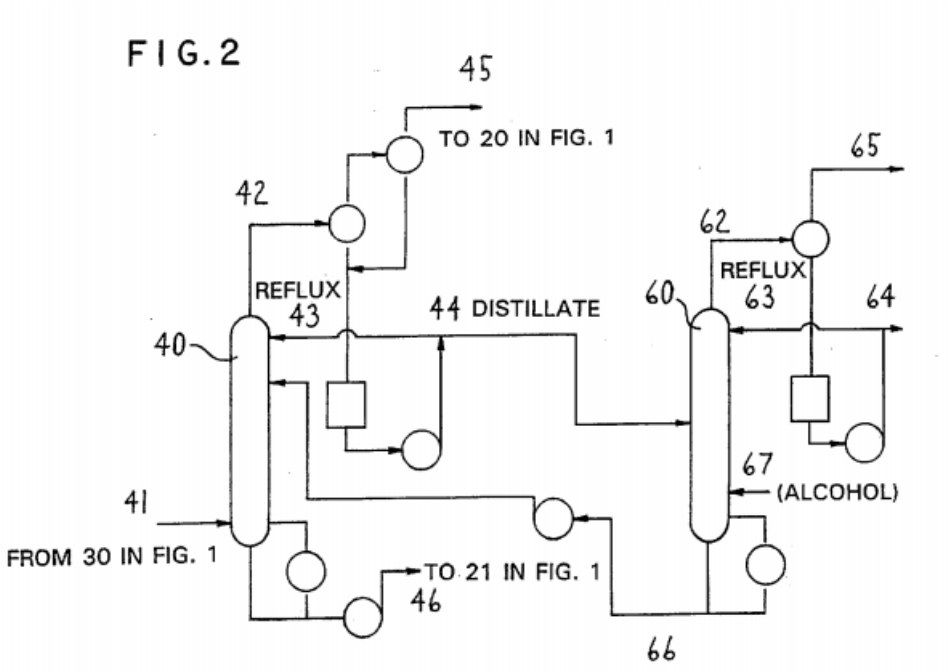


Figure 1 is a process flow diagram showing a reaction and recovery system "used for rhodium-catalyzed carbonylation of methanol to acetic acid." *Id.* at 5:3–5. Figure 2 shows "one example of a distillation system for separating methyl iodide from acetaldehyde." *Id.* at 2:34–35.

As shown in Figure 1, liquid products from a methanol carbonylation reaction are withdrawn from reactor 10 via line 11 and introduced into

flasher 12. Ex. 1010, 5:30–34. 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 5:39–44. 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), or introduced into 80 plate distillation column 40 (*id.* at 8:52–58).

The '095 patent discloses an Example and Example 1 (hereinafter referred to collectively as "the '095 patent Example") in which a methanol carbonylation was conducted and lower phase 30 was introduced into distillation column 40, as shown in Figure 2. *See id.* 12:1–13:4. The '095 patent discloses that "[w]hen distillation for separation is carried out in one distillation column, water may be charged into the distillation column, and/or distillation temperature and pressure may be elevated to control the generation of paraldehyde and metaldehyde." *Id.* at 7:25–29. Petitioner identifies lower phase 30 as meeting the claim 58 limitation of "a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and at least one permanganate reducing compound" and cites column 7, lines 14–19, of the '095 patent for a description of the claim 58, step (a) distillation of the mixture to obtain "a PRC enriched overhead stream

comprising dimethyl ether," i.e., distillate 44. *See* Pet. 28; Ex. 1005 ¶ 13. The '095 patent Example describes carrying out a water extraction with distillate 44 (*see* Ex. 1010, 12:56–58) and Petitioner identifies column 15, lines 30–32, describing the use of two extraction stages, as meeting the limitations of step (b) of challenged claim 58 (*see* Pet 28).

2. JP '712 (Ex. 1020)

Japanese Patent Application 2000-72712, filed August 31, 1998, and published March 7, 2000, identifies Petitioner as the assignee and Mr. Miura as the sole inventor. Ex. 1020, 1:1–7. JP '712 describes a low water carbonylation process wherein methanol, dimethyl ether, or methyl acetate is allowed to react with carbon monoxide in the presence of a rhodium catalyst, an iodide salt and methyl iodide (Ex. 1020, 3:4–6), and the reaction liquid is then separated into high and low volatile phases (*id.* at 1:18–19). The high volatile phase is separated by distillation into acetic acid product and an impurities stream comprising the carbonyl compound. *Id.* at 1:20–24. The impurities stream is contacted with water to separate the impurities into an organic phase free from the carbonyl compound and an aqueous phase comprising methyl iodide. *Id.* at 1:24–2:5. JP '712 discloses that the methyl iodide concentration in the process liquid was reduced by increasing the extraction temperature, e.g. 1.7 wt% vs. 0.1 wt% at contact temperatures of 13 °C and 47 °C, respectively. *Id.* at 21, Table 1.

According to JP '712, corrosion of the metal forming the distillation column can be induced by hydrogen iodide generated due to the presence of an excessively large amount of methyl iodide according to the reaction $CH_3I+H_2O \leftrightarrow CH_3OH+HI$. *Id.* at 14:20–15:3. JP '712 discloses that the generation of hydrogen iodide can be prevented by adding methanol to the

distillation column to shift the static reaction to the initial system. *Id.* at 15:3–5. JP ’712 cautions, however, that when methanol was added in a molar quantity of more than 55 times as much as that of iodide ion, it was assumed that the amount of hydrogen iodide was increased by the following side reaction, that also show production of DME:

$$CH_3I+CH_3OH \Rightarrow HI+CH_3OCH_3$$

Id. at 25:17–23.

3. Singh (Ex. 1011)

Singh, filed May 3, 1999, and issued January 15, 2002, identifies Patent Owner as the assignee. Ex. 1011, at [22], [45], [73]. The ’507 patent incorporates Singh by reference in its entirety. Ex. 1002, 3:66–67. 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 described in connection with Figure 1, below.

FIG.1

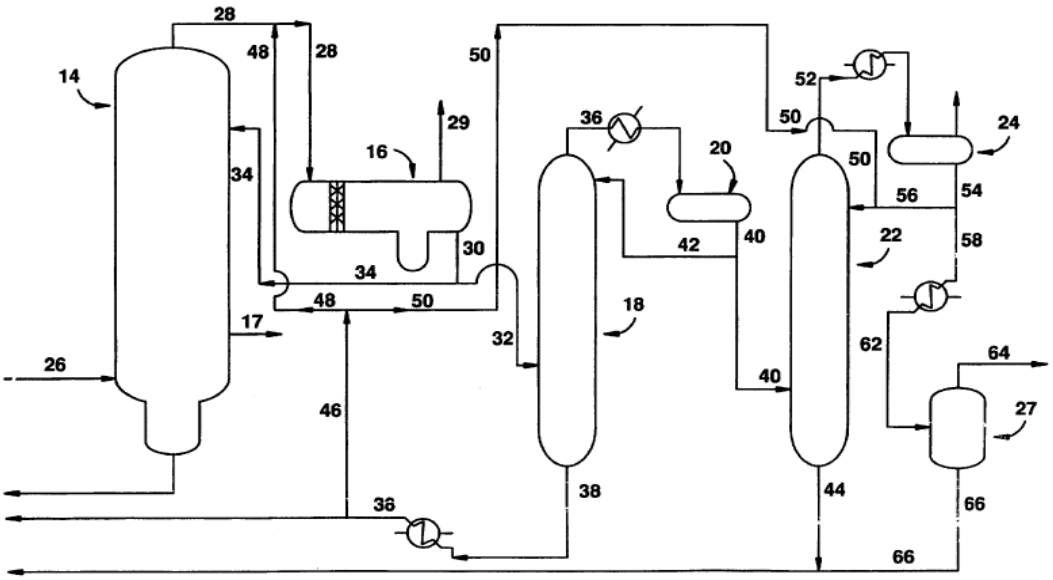


Figure 1 is a process flow diagram of "a preferred embodiment for the removal of carbonyl impurities from an intermediate stream of the carbonylation process for the production of acetic acid by a carbonylation reaction." *Id.* at 6:33–36. As discussed in connection with the '095 patent,

> [d]issolved gases exiting the [carbonylation] reactor as a side stream and entering the flasher consist of a portion of the carbon monoxide along with gaseous by[-]products such as methane, hydrogen, and carbon dioxide and exit the flasher as an overhead stream[, which then] are directed to the light ends or splitter column 14 as stream 26.

Id. at 8:53–57. First vapor phase acetic acid stream 28, containing methyl iodide, methyl acetate, acetaldehyde, and other carbonyl components, exits splitter column 14, and is condensed and separated in decanter 16 to form a light ends phase (stream 30) containing acetaldehyde, water, and acetic acid. *Id.* at 9:33–38. 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 10:1–3, 17–18. Distillation column 18 concentrates the aldehyde components of stream 32 into overhead stream 36 by separating water and acetic acid from the lighter components. Ex. 1011, 9:67–10:1.

Overhead stream 36 is condensed in overhead receiver 20 to form stream 40 containing "acetaldehyde, methyl iodide, methyl acetate, and methanol." *Id.* at 10:38–42. 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 10:42–45. Distillation column 22 separates the majority of the acetaldehyde from the methyl iodide, methyl acetate, and methanol in stream 40. *Id.* at 10:45–47. Singh discloses 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 11:5–7, 16–18; *see also id.* at 12:66–13:4 ("[I]t has been found that when shutting down the carbonylation system, in particular the distillation columns employed in the present process, polymers of acetaldehyde, both low and high molecular weight chain polymers, tend to form and build up in the base of the second column."). "[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 11:18–25. Singh discloses that acetic acid is the preferred solvent due to its abundance in an internal stream, and that the use of excess solvent is undesirable since this places a greater load on the system to reprocess the excess solvent. *Id.* at 6:17–26. According to Singh, "[w]ater

as an inhibitor is the least preferred solvent of inhibition since large quantities are generally needed to be an effective inhibitor and as such it tends to extract a large amount of acetaldehyde, reducing the purity of stream 52 exiting column 22." *Id.* at 11:37–42. Singh discloses that stream 50, containing water and acetic acid, is preferably employed as an inhibitor since this material does not change the water balance within the reaction system. *Id.* at 11:35–37. Singh clarifies that "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." *Id.* at 11:32–35.

Stream 52, comprising PRC's, 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 11:42–48, 12:7. Part of stream 54, i.e., slip stream 56, is used as reflux for distillation column 22, and the remainder, stream 58, is preferably condensed and sent to extractor 27 as stream 62 to remove and recycle small amounts of methyl iodide. *Id.* at 11:46–47, 57–61.

Singh discloses that "[i]t is important for the extraction mechanism that the top stream of 22 remain cold, generally at a temperature of about 13° C." Ex. 1011, 11:51–53. In extractor 27, PRCs and alkyl iodides are extracted with water, preferably from an internal stream. *Id.* at 11:62–64. PRC-rich aqueous extract stream 64 then leaves extractor 27 from the top and is directed to waste treatment. *Id.* at 12:3–6. Raffinate stream 66, containing methyl iodide, exits the bottom of extractor 27 and is combined with stream 44 for recirculation to the reactor. *Id.* at 11:47–50, 12:1–3.

C. The ’507 Patent (Ex. 1002)

The ’507 patent describes Singh’s multi-stage purification of the light ends column overhead as significantly reducing undesirable impurities in the acetic acid product, and the extraction of the acetaldehyde-rich distillate recycles a raffinate having a significantly lower acetaldehyde concentration to the reactor. *See* Ex. 1002, 3:55–65. According to the ’507 patent, however, further improvements can be made, and the ’507 patent is said to be directed to an “alternative process[] to improve the efficiency of acetaldehyde removal.” *Id.* at 4:6–8. The ’507 patent describes the process as not only useful in processes for carbonylation of methanol to acetic acid, but also in the carbonylation of other materials “such as methyl acetate, methyl formate or dimethyl ether, or mixtures thereof[] to acetic acid in the presence of a Group VIII metal catalyst such as rhodium and an iodide promoter.” *Id.* at 5:39–44. Mr. Jones and Dr. Porcelli agree that the description in the ’507 patent implies that the process is a low water process. Ex. 1039 ¶ 50; Ex. 2029 ¶ 26.

The ’507 patent process is described in connection with Figure 2, reproduced below.

FIG. 2

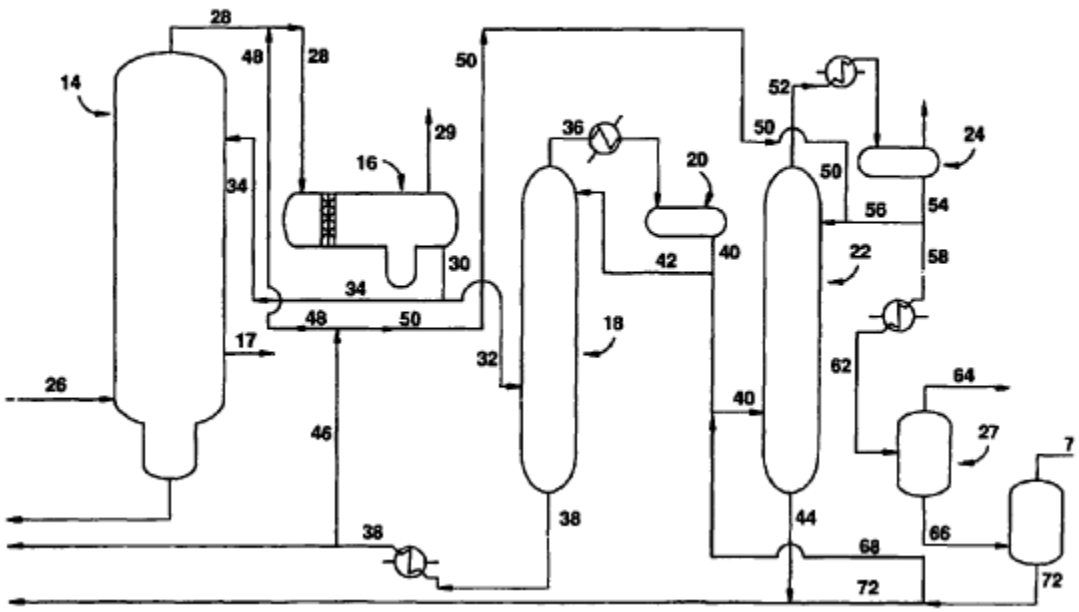


Figure 2 is a process flow diagram of a preferred embodiment. Ex. 1002, 5:14–15. The embodiment depicted and described in Figure 2 is described as using two distillation columns 18, 22, however, the ’507 patent explicitly states that distillation may be performed in a single column as well. *Id.* at 8:39–42. As can be seen by comparing Figure 2 with Singh Figure 1, the ’507 patent contemplates directing raffinate stream 66 to second extractor 25 to extract additional PRCs and alkyl iodides using water, preferably from an internal stream, rather than returning it directly to the reactor as in Singh. *Compare id.* at 10:63–66 *with* Ex. 1011, 12:1–3. Further, unlike Singh which discloses recirculating raffinate stream 66 to the reactor in its entirety, the ’507 patent discloses combining a portion of methyl iodide-rich raffinate stream 72 from second extractor 25 (i.e., stream 68) with stream 40 as feed to second distillation column 22. *See id.*, Fig. 2.

The ’507 patent is subject to a terminal disclaimer based on U.S. Patent 7,223,883 B2, filed March 2, 2004, and issued May 29, 2007 (Ex. 3001, “the ’883 patent”).[7] Ex. 1002, Title page. Figure 2 of the ’883 patent is identical to Singh Figure 1, with the exception that a portion of methyl iodide-rich raffinate stream 66 from extractor 27 (i.e., stream 68) is combined with stream 40 as feed to second distillation column 22 as in the ’507 patent. *See* Ex. 3001, 10:58–63, Fig. 2. Unlike the ’507 patent, however, the ’883 patent illustrates the use of only single extractor 27. *Compare id.*, Fig. 2 *with* Ex. 1002, Fig. 2.

The ’507 patent does not describe a reason for returning a portion of the methyl iodide-rich raffinate stream 72 to second distillation column 22. *See* Ex. 1039 ¶ 31 (“[S]tream 68 is not discussed or disclosed in the specification of the ’507 Patent, other than appearing in Figure 2.”). According to the ’883 patent, however, the inventors “discovered that returning a portion of raffinate stream 66 to distillation column 22 improves the aldehyde removal efficiency of the entire system.” Ex. 3001, 10:61–64. According to the ’883 patent, when the inventors began testing the invention they observed that when raffinate stream 66 was returned to distillation column 22 in its entirety, “the pressure in column 22 rose significantly over time, indicating an accumulation of a volatile component in the system that

[7]It appears that Petitioner intended to file the ’883 patent as Exhibit 1024. Exhibit 1024, U.S. Patent No. 7,223,833 B1, is directed to subject matter unrelated to the subject matter involved in this *inter partes* proceeding. The ’883 patent was filed by Patent Owner as Exhibit 2001 in IPR2014-01514 and in IPR2015-00171. Exhibit 2001 was not filed in the present *inter partes* review, but is referenced by Dr. Porcelli in his declaration. *See, e.g.*, Ex. 2029 ¶ 44.

was not being removed in the extraction." *Id.* at 11:8–12. It was determined "that there were a number of chemical reactions taking place within column 22, including the hydrolysis of methyl acetate and methyl iodide to methanol and the subsequent formation of dimethyl ether (DME). DME was identified as the volatile component that was causing the increase in column pressure." *Id.* at 11:12–18. According to the '883 patent, "[w]hen all of raffinate stream 66 from extractor 27 is recycled to column 22, the DME formed in the column is not removed from the system. . . . [T]he issue can be resolved by recycling a portion of stream 66 directly or indirectly to the reactor system" where "DME can be carbonylated in the reactor to produce acetic acid." *Id.* at 11:18–29.

The '883 patent and the '507 patent, like Singh (*see* Ex. 1011, 11:51–53), disclose that "[i]t is important for the extraction mechanism that the overhead stream of column 22 remain cold, generally at a temperature of about 13° C." Ex. 1002, 10:43–45; Ex. 3001, 10:39–41. According to the '507 patent, it was 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.

Ex. 1002, 11:31–37. The inventors observed, for example, that "the concentration of methyl iodide in stream 64 dropped from about 1.8% when no DME was present to about 0.5% when DME was present." *Id.* at 11:38–41. According to the '507 patent, 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 11:34–37. DME either can be injected into the process upstream of extractor 27, or "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 11:42–47; *see also* Ex. 3001, 11:49–51 ("[I]t is possible to generate additional DME within the process by feeding additional water to column 22 in either feed stream 40 or reflux stream 56.") *Id.* at 11:49–51.

IV. LEVEL OF ORDINARY SKILL IN THE ART

The level of ordinary skill in the art is relevant to claim construction. *In re Suitco Surface, Inc.*, 603 F.3d 1255, 1259–60 (Fed. Cir. 2010) ("[C]laim language should be read in light of the specification as it would be interpreted by one of ordinary skill in the art.").

> Factors that may be considered in determining the ordinary level of skill in the art include: 1) the types of problems encountered in the art; 2) the prior art solutions to those problems; 3) the rapidity with which innovations are made; 4) the sophistication of the technology; and 5) the educational level of active workers in the field.

Ruiz v. A.B. Chance Co., 234 F.3d 654, 666–67 (Fed. Cir. 2000).

Petitioner relies on Mr. Jones's testimony to establish the level of ordinary skill in the art. *See* Pet. 17. Mr. Jones testified that the person of ordinary skill in the art "as of March 2, 2004, the effective filing date of the '507 Patent" (Ex. 1039 ¶ 15) would have been a chemist or chemical engineer with an advanced degree, or a Bachelor of Science degree and equivalent work experience (e.g., five years in an industrial setting producing acetic acid, acetic anhydride, or related chemicals), and

> would have generally understood acetic acid production on an industrial scale including the distillation processes and extraction processes that are used in acetic acid or acetic anhydride production. The person of ordinary skill in the art would have understood chemical reactions in general, and, particularly, the primary reactions and the side-reactions associated with acetic acid production

(*id.* ¶ 16). Mr. Jones further testified that a person of ordinary skill in the art would have had separations experience, an understanding of industrial-scale processes, and experience in the production of acetic acid, acetic anhydride, or related chemicals. *Id.* ¶ 17.

Dr. Porcelli, testified that he "generally agree[s]" with Mr. Jones's opinion as to the level of ordinary skill in the art" (Ex. 2029 ¶ 15; *see also* Ex. 1065, 65:9–66:5). During cross-examination, however, Dr. Porcelli testified that one of ordinary skill in the art would require, more specifically, an understanding of the low water acetic process and its implementation on an industrial scale. *See, e.g.*, Ex. 1065, 126:18–127:1.

Mr. Jones and Dr. Porcelli agree the description in the '507 patent implies that the inventive process is a low water process. Ex. 1039 ¶ 50; Ex. 2029 ¶ 26. As discussed in Section VI.B., below, these experts further agree that there are differences in the reactants and reaction products produced in high and low water processes.

We find both Mr. Jones and Dr. Porcelli are qualified to give an opinion as to the level of ordinary skill in the art. Accordingly, for purposes of this proceeding we adopt Mr. Jones's definition of the level of ordinary skill in the art, and add that the ordinary artisan also would have had a specific understanding of the low water carbonylation process for production of acetic acid and its implementation on an industrial scale.

V. CLAIM CONSTRUCTION

In an *inter partes* review, the Board interprets claim terms in an unexpired patent according to the broadest reasonable construction in light of the specification of the patent in which they appear. 37 C.F.R. § 42.100(b); *PPC Broadband, Inc. v. Corning Optical Commc'ns RF, LLC*, 815 F.3d 747, 755 (Fed. Cir. 2016) ("Above all, the broadest reasonable interpretation must be *reasonable* in light of the claims and specification."); *see also In re Cuozzo Speed Techs., LLC*, 793 F.3d 1268, 1278–80 (Fed. Cir. 2015) ("Congress implicitly approved the broadest reasonable interpretation standard in enacting the AIA," and "the standard was properly adopted by PTO regulation."), *aff'd sub nom. Cuozzo Speed Techs. LLC v. Lee*, No. 15–446, 2016 WL 3369425, at *12 (U.S. June 20, 2016) (upholding the use of the broadest reasonable interpretation standard). Under that standard, and absent any special definitions, we give claim terms their ordinary and customary meaning, as would be understood by one of ordinary skill in the art at the time of the invention. *In re Translogic Tech., Inc.*, 504 F.3d 1249, 1257 (Fed. Cir. 2007); *see also Trivascular, Inc. v. Samuels*, 812 F.3d 1056, 1062 (Fed. Cir. 2016) ("Under a broadest reasonable interpretation, words of the claim must be given their plain meaning, unless such meaning is inconsistent with the specification and prosecution history."). Any special definitions for claim terms must be set forth with reasonable clarity, deliberateness, and precision. *In re Paulsen*, 30 F.3d 1475, 1480 (Fed. Cir. 1994).

"While the Board must give [claim] terms their broadest reasonable construction, the construction cannot be divorced from the specification and the record evidence." *In re Man Mach. Interface Techs. LLC*, No. 2015-

1562, 2016 WL 1567181 at *2 (Fed. Cir. Apr. 19, 2016) (quoting *In re NTP, Inc.*, 654 F.3d 1279, 1288 (Fed. Cir. 2011)). The doctrine of prosecution history disclaimer requires that "the claims of a patent be interpreted in light of the proceedings in the PTO during the application process." *Trivascular*, 812 F.3d at 1063. "Any explanation, elaboration, or qualification presented by the inventor during patent examination is relevant, for the role of claim construction is to capture the scope of the actual invention that is disclosed, described, and patented." *Id*. (quoting *Fenner Invs., Ltd. v. Cellco P'ship*, 778 F.3d 1320, 1323 (Fed. Cir. 2015)).

Expert testimony is useful to explain terms of art, and the state of the art at any given time, but cannot be used to prove "the proper or legal construction of any instrument of writing." *Teva Pharm. USA, Inc. v. Sandoz, Inc.*, 135 S. Ct. 831, 841 (2015) (internal citations omitted). Only terms which are in controversy need to be construed, and only to the extent necessary to resolve the controversy. *Vivid Techs., Inc. v. Am. Sci. & Eng'g, Inc.*, 200 F.3d 795, 803 (Fed. Cir. 1999).

A. The Arguments

Petitioner and Patent Owner offer proposed claim constructions for terms recited in independent claim 58. *See* Pet. 11–17; PO Resp. 7–12; Pet. Reply 2–3. For purposes of this decision, however, we determine the only claim language requiring express construction is the claim 58 phrase: "distilling [a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and at least one PRC] to separate the mixture into a plurality of streams, at least one of said streams being a PRC enriched overhead stream comprising dimethyl ether" (Ex. 1002, claim 58, step (a)).

In their briefs, neither Petitioner nor Patent Owner offers an explicit construction for the claim 58 phrase at issue. *See generally*, Pet. 11–17; PO Resp. 7–12; Pet. Reply 2–3. In its Response, Patent Owner asserts that:

> The invention of the ’507 patent lies in the discovery that dimethyl ether (DME) in the extraction step significantly reduces the amount of methyl iodide that is undesirably extracted into the aqueous extract stream and subsequently lost in wastewater treatment. . . . This discovery led the inventors to conclude that one or more extraction steps could be employed in the process to remove additional acetaldehyde from the raffinate *without the expected loss of methyl iodide in the aqueous extract stream(s)*, so long as DME is present during the extraction step. . . .
>
> Claim 58 of the ’507 patent captures this aspect of the invention by claiming that the PRC enriched overhead stream comprises DME, and that the PRC enriched overhead stream is subjected to at least two extraction steps.

PO Resp. 4–5 (citing Ex. 1002, 11:32–41).

Petitioner argues “the independent claims contain no limitations requiring a specific amount of DME, or any effect on MeI loss” (Pet. Reply 1) and that the presence of a single molecule of DME is sufficient to meet step (a) of claim 58 (Tr. 22:3–7; *see also id.* at 21:11–12 (“[T]he claims don’t require any specific amount of DME. They just require some DME.”)). During oral argument, Petitioner disagreed with interpreting claim 58 as requiring sufficient DME to achieve a reduction in the solubility of methyl iodide in the aqueous extract stream. *See id.* at 23:4–9. According to Petitioner, interpreting claim 58 to include such limitation likely would render claim 58 indefinite because the amount of DME needed to achieve a certain methyl iodide level in the aqueous extract stream varies widely depending on temperature. *See id.* at 23:14–24:1.

At oral argument, Patent Owner, in turn, argued claim 58 requires a detectable amount of DME, i.e., more than a molecule. *See id.* at 41:12–42:4. Patent Owner argued the claims must be construed in light of the understanding of the ordinary artisan, and cited Dr. Porcelli's cross-examination testimony in support of its contention that persons of ordinary skill in the art "speak in terms of effects"—and "do not speak or think in terms of molecules. *Id.* at 41:8–16 (citing Ex. 1065, 178:2–5).

B. Intrinsic Evidence

1. The '507 Patent Claims

Independent claim 1 recites a step of distilling a first overhead comprising methyl iodide, water, and at least one PRC to produce a second overhead stream comprising methyl iodide, dimethyl ether, and the PRC. Independent claims 14, 25, 42, and 58 recite a step of distilling a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and at least one PRC, resulting in a PRC-enriched overhead stream comprising dimethyl ether. All of the independent claims (claims 1, 14, 25, 42, and 58) recite at least one step of extracting the DME-containing overhead stream with water, and recite the presence of methyl iodide in the overhead stream from the distillation or in the raffinate stream produced by the extraction step(s).

The independent claims of the '507 patent do not recite expressly the presence of a specific amount of dimethyl ether in the PRC-enriched overhead stream produced by the distillation step. Claims dependent from claims 25, 42, and 58, however, recite the presence of dimethyl ether in this overhead stream at concentrations that are sufficient, during the extraction step(s), to produce aqueous extract streams having methyl iodide

concentrations of "less than 1.8 wt%" (claims 39, 55, 71), "between about 1.8 wt% and 0.5 wt %" (claims 40, 56, 72) and "less than or equal to about 0.5 wt %" (claims 41, 57, 73). Claim 10, dependent from claim 1, recites the presence of DME in the first overhead.

2. The '507 Patent Specification

The '507 patent discloses that a potential problem with using a multistage extraction for removal of PRC's and alkyl iodides from the overhead distillation stream in a carbonylation process is that each water extraction removes a measurable amount of methyl iodide, "an especially costly component of the reaction system." Ex. 1002, 11:23–28. The '507 patent further discloses that the "applicants have discovered" the loss of methyl iodide in the extraction steps can be limited by adding dimethyl ether (DME) to the feed of the first extractor. *Id.* at 11:31–33. According to the '507 patent, DME can be injected into the process upstream of the extractor, e.g., into stream 62 fed to the extractor. *Id.* at 11:42–45. The '507 patent further discloses that "[t]he 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. The '507 patent discloses that "[t]he 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 11:33–37. According to the '507 patent, the applicants observed that the concentration of methyl iodide in stream 64 dropped from about 1.8% when no DME was present to about 0.5% when DME was present. *Id.* at 11:39–41.

The '507 patent discusses Singh's teaching of adding an inhibitor, such as water, methanol, and/or acetic acid, to distillation column 22 "to

reduce the formation of these . . . metaldehyde and paraldehyde [impurities] and higher molecular weight polymers of acetaldehyde." Ex. 1002, 10:9–15, 24–27. The '507 patent discloses that although "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," use of an internal aqueous stream, e.g., a residue stream containing water and acetic acid from first distillation column 18, is preferable "since this material does not change the reactor water balance." *Id.* at 10:24–29. Similarly, water used in the extractions of the overhead stream from distillation column 22 is described as "preferably . . . from an internal stream so as to maintain water balance within the reaction system." *Id.* at 10:52–53, 65–67; *see also* Ex. 1011, 11:35–37. The '507 patent does not describe a preferred source for any water added to column 22 for the purpose of producing DME. *See generally*, Ex. 1002, 11:23–56.

3. The '507 Patent Prosecution History

The '507 patent was filed on April 20, 2007, as application No. 11/788,455 ("the '455 application"), a continuation of application No. 10/708,420 ("the '420 application), filed March 2, 2004, and issued as the '886 patent. During prosecution of the '420 application,[8] the examiner

[8] *See Advanced Cardiovascular Sys., Inc. v. Medtronic, Inc.*, 265 F.3d 1294, 1305 (Fed. Cir. 2001) ("The prosecution history of a related patent can be relevant if, for example, it addresses a limitation in common with the patent in suit."); *Elkay Mfg. Co. v. Ebco Mfg. Co.*, 192 F.3d 973, 980 (Fed. Cir. 1999) ("When multiple patents derive from the same initial application, the prosecution history regarding a claim limitation in any patent that has issued applies with equal force to subsequently issued patents that contain the same claim limitation.").

rejected claims under 35 U.S.C. § 103 as obvious over the ’930 patent[9] in view of Allison (Organic Chemistry Laboratory II, Chemistry 3712/3612, spring 2003 Edition, department of Chemistry and Biochemistry University of Arkansas, pages 1-35) (Ex. 2007, 3–5). In the Section 103 rejection, the Examiner acknowledged that the ’930 patent “do[es] not teach forming dimethyl ether in the last distillation column” (Ex. 2007, 3), but maintained dimethyl ether would be formed, and present in the second overhead, in the ’930 patent process because “water is already present in the . . . distillation” (*id.* at 4 (citing Ex. 2006, 9:48–50)) and, “[a]ccording to applicant dimethyl ether is formed when water is added to the distillation column” (*id.* (citing the ’420 application, p. 33)). The referenced disclosure in the ’930 patent describes second distillation column 22 as separating acetaldehyde from “the other components,” and identifies the “[c]atalytic components [as] includ[ing] methyl iodide, methyl acetate, methanol, and water.” Ex. 2006, 9:48–50.

In traversing the Section 103 rejection in the ’420 application, Patent Owner argued the ’930 patent’s disclosure that

> water is already present in the distillation column . . . is not the same as adding additional water to the distillation column as disclosed in [the present application]. The present invention discloses advantages obtained by adding DME to the feed to the extractor. . . . [The ’930 patent] does not disclose forming DME, does not recognize benefits of having DME present during the extraction, and does not disclose procedures for causing the formation of DME.

[9] As noted in Section III.B., above, Singh is a continuation-in-part of the ’930 patent.

Ex. 2009, 7.

During prosecution of the ’455 application, the examiner rejected claims under 35 U.S.C. § 102(b) as anticipated by the ’930 patent. *See, e.g.*, Ex. 2019, 4–9. The examiner also rejected claims on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1–3 of the ’930 patent in view of Allison, contending the only difference between claims 1–3 of the ’930 patent and the ’455 application claims was that the latter required multiple extractions. *Id.* at 11–12. The examiner found dimethyl ether is formed inherently in second distillation column 22 of the ’930 patent process because the stream entering second distillation column 22 comprises methanol, and the ’455 application discloses that dimethyl ether is formed in the column by dehydration of methanol. *Id.* at 6.

In its response to the Section 102 rejection, Patent Owner disagreed with the examiner’s finding of inherency, arguing the acetic acid present in second distillation column 22 is not the type of strong mineral acid catalyst required for production of DME via dehydration of methanol. Ex. 1009, 20. Patent Owner argued that unlike the ’930 patent process, DME is formed in the claimed process because water is added to second distillation column 22 to form HI, a sufficiently strong catalyst for production of DME by methanol dehydration. *Id.* Patent Owner acknowledged the ’930 patent discloses adding water to second distillation column 22 to inhibit formation of various aldehyde impurities, but asserted this amount was insufficient to form DME in an amount sufficient to reduce the solubility of MeI in the aqueous phase of the extraction of the overhead stream from second distillation column 22. *Id.* at 21–22. Patent Owner argued, more specifically, that the ’930 patent

"fails to disclose or suggest a process wherein DME is present in [a] process stream . . . []or a process wherein DME is produced in the distillation column." *Id.* at 23. Patent Owner also asserted that "[t]he ability to economically conduct a second extraction is . . . only possible through the unexpected discovery that a sufficient amount of a non-preferred inhibitor[10] in a distillation column results in DME being present in the process stream in complete contradiction to generally accepted principles of Chemistry and Engineering." *Id.* at 24. In response to the double patenting rejection, Patent Owner argued similarly that nothing in the '930 patent or Allison discloses or suggests including dimethyl ether in the second overhead whereby a reduction in solubility of MeI in the aqueous phase extraction is achieved. Ex. 2021, 14. The examiner ultimately withdrew the rejections based on the '930 patent in the '455 application, indicating Patent Owner's arguments were persuasive. *See* Ex. 2020, 3; Ex. 3002.

C. Extrinsic Evidence

Because we give claim terms their ordinary and customary meaning, as would be understood by one of ordinary skill in the art at the time of the invention, *In re Translogic Tech.*, 504 F.3d at 1257, we may consider the testimony of the experts and the prior art to ascertain whether one of ordinary skill in the art would have understood the claim 58 phrase "distilling [a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and at least one PRC] to separate the mixture into a plurality of streams, at least one of said streams being a PRC enriched

[10] *See* Ex. 1002, 10:29–30; Ex. 1011, 11:37–42 (identifying water as a non-preferred inhibitor).

overhead stream comprising dimethyl ether" as requiring the presence of DME in a particular quantity.

Paulik describes DME as an undesirable byproduct, and discloses that DME was formed in "significant quantities" in prior art carbonylation systems that utilized a cobalt catalyst system. Ex. 2037, 12:51-53. Paulik explains that dimethyl ether" attains a very high partial pressure at the reaction conditions employed, and necessitates the use of a high total pressure reaction system to maintain the necessary partial pressure of carbon monoxide for reaction to form carboxylic acid." *Id.* at 12:54–58. Paulik describes various experiments conducted using the Monsanto process, wherein DME, as well as other undesirable byproducts such as aldehydes, higher boiling carboxylic acids, methane, and carbon dioxide, were either not detected, or detected in "insubstantial amounts" using gas chromatography. *See id.* at 12:3–14:49, 15:9–18:75, 20:13–31. In one of these experiments, Paulik used a methanol feedstock containing 10 wt. percent DME, and likewise discloses that no substantial amounts of byproducts, including DME, were formed. *Id.* at 19:2–27. Paulik indicates that conversion of 50 mole percent of a methanol feedstock to dimethyl ether is considered a significant quantity. *See id.* at 12:51–53, 14:50–15:8. Paulik does not state what amount of dimethyl ether, or other undesirable byproduct is considered insubstantial. Nor does Paulik indicate the measurement capabilities of the detection equipment used to analyze byproduct content of the reaction products in the experiments.

Akinori discloses that DME is produced as an intermediate in a side reaction in the Monsanto process, but states that intermediates are ultimately converted to acetic acid. Ex. 1013, 8. Smith describes a low water

carbonylation process, and discloses a substantial reduction in the undesirable reaction byproducts propionic acid, hydrogen and carbon dioxide, as compared to high-water processes. Ex. 2038, 13:1–10. Smith does not discuss the presence or absence of DME, or of any other byproducts. *See generally*, *id.* at 4:10–18:68. Singh and the ’095 patent disclose that acetaldehyde and other carbonyl components are produced in low water processes, but do not disclose the presence of DME at any point in the reaction process. *See* Ex. 1010, 1:44–49; Ex. 1011, 1:18–22.

Mr. Jones testified that DME would be formed in all water carbonylation systems, and indicated that a molecule of DME would be sufficient for him to consider DME as being present in a system. Ex. 2026, 316:18–20; *see id.* at 317:16–18. Mr. Jones testified that the only difference between Figure 1 of the ’507 patent, depicting the prior art method, and Figure 2 of the ’507 patent, depicting the claimed method, “is the incorporation of an additional extractor (25) and streams flowing to and from the additional extractor (25).” Ex. 1039 ¶ 27. Mr. Jones thus opined that “steps (a) and the first part of (b) of claim 58 of the ’507 Patent should be considered to be admitted prior art . . . , because it is merely describing a distillation and first extraction that are done according to Figure 1 of [Singh].” *Id.*

Dr. Porcelli testified that, as a chemical engineer, he “deal[s] with what’s practical, what’s viable.” Ex. 1065, 178:2–4. Dr. Porcelli testified that he would have no reason to believe DME was present in a process stream absent evidence of its presence. *See id.* at 189:10–18. Dr. Porcelli indicated that he would not consider the existence of a molecule of DME as meaning that DME was present in a stream absent some showing that the

existence of a molecule was somehow significant or had an impact on something. *See id.* at 186:3–187:13. Dr. Porcelli testified that one of ordinary skill in the art would understand from the ’507 patent that the invention lies in using DME to enhance the separation for acetaldehyde and methyl iodide. *See id.* at 58:5–8; 126, 9–13.

D. Analysis

Turning first to the claims, we note that claim 58 does not recite explicitly the presence of a specific amount of DME in the second overhead stream. *See* Ex. 1002, claim 58. As noted in Section V.B.1., above, various claims dependent from independent claims 25, 42, and 58 require the presence of DME in the distillation overhead at concentrations sufficient, during the extraction step(s), to produce aqueous extract streams having specific methyl iodide concentrations. The claims alone, however, do not support a narrow interpretation of the claim 58 phrase “distilling [a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and at least one PRC] to separate the mixture into a plurality of streams, at least one of said streams being a PRC enriched overhead stream comprising dimethyl ether” as limited to a specific amount, or concentration, of DME.

According to the ’507 patent specification, the applicants viewed their invention as the discovery that “adding dimethyl ether (DME)” to the feed to the extractor(s) reduces the amount of methyl iodide lost during extraction by reducing the solubility of methyl iodide in water. Ex. 1002, 11:31–35. The ’507 patent specification does not identify the specific amount or concentration of DME needed to effect this desired result, but states that the “required quantity” of DME can be injected or produced by the addition of water to the distillation column. *See id.* at 11:42–47. The ’507 patent

distinguishes between water added to second distillation column 22 for the purpose of inhibiting aldehyde formation (as taught in Singh and the ’930 patent) and for the purpose of producing DME. *Compare id.* at 10:16–29; Ex. 1011, 11:27–37; Ex. 2006, 10:64–11:6 *with* Ex. 1002, 11:45–47. Specifically, the ’507 patent discloses that aldehyde formation in the second distillation column can be inhibited by adding internal process stream 50, comprising both water and acetic acid to second distillation column 22. Ex. 1002, 10:19–22. The ’507 patent identifies water as a non-preferred inhibitor and discloses that use of internal process stream 50 as an inhibitor is preferred, so as not to change the water balance within the reaction system. *Id.* at 10:24–30; *see also* Ex. 1011, 11:37–42; Ex. 2006, 11:4–10. The ’507 patent does not, however, identify an internal process stream as a source for water added to the second distillation column for the purpose of producing DME. *See generally* Ex. 1002, 11:23–56. Rather, the ’507 patent describes adding water to feed 40 or reflux 56[11] without identifying the source thereof or expressing concern for changing the water balance within the reaction system. *Id.* at 11:45–47. One of ordinary skill in the art would understand from the ’507 patent’s distinction in the water sources used for impurity inhibition versus DME production in the second distillation column, that a quantity of water in excess of the amount added via internal process stream 50 is required to produce DME in second distillation column 22 in an amount necessary to obtain the required quantity of DME in extractor feed 62.

[11] The ’507 patent erroneously identifies reflux 56 by reference numeral 50.

During prosecution of the ’507 patent, applicants argued “[t]he ability to economically conduct a second extraction is . . . only possible through the unexpected discovery that a sufficient amount of a non-preferred inhibitor in a distillation column results in DME being present in the process stream.” Ex. 1009, 24. From the issuance of a notice of allowance following applicants’ argument that “[n]othing in [the ’930 patent] or in Allison discloses or suggests inclusion of dimethyl ether in the second overhead, either through addition of dimethyl ether to the process or by in-situ formation of dimethyl ether in the process” (Ex. 2021, 14), we infer the examiner was persuaded by applicants’ arguments, *see Trivascular*, 812 F.3d at 1065, and that the examiner understood (1) applicants’ claims require a sufficient amount of DME in the second distillation overhead to reduce the amount of methyl iodide lost during a water extraction of the overhead to separate acetaldehyde and methyl iodide, and (2) that the amount of water contained in an internal process stream added to the distillation column to inhibit formation of metaldehyde and paraldehyde impurities (e.g., the amount contained in internal process stream 50) would not produce a quantity of DME sufficient to reduce the amount of methyl iodide lost during the extraction step(s).

Based on Paulik’s disclosure, we find one of ordinary skill in the art would understand that, prior to the ’507 patent, DME was viewed as an undesirable byproduct in a carbonylation process. *See* Ex. 2037, 12:51–58. One of ordinary skill in the art would know that amounts of DME considered “insubstantial” in a high water carbonylation process were measurable by gas chromatography as early as 1970. *See id.* at 12:3–14:49, 15:9–18:75, 20:13–31. One of ordinary skill in the art would expect less

DME to be produced in a low water process. *See* Ex. 2029 ¶ 117. Because patents issued subsequent to Paulik, and relating to low water carbonylation processes (such as Smith, Singh, and the ’095 patent), do not describe the presence of DME, one of ordinary skill in the art would understand that DME was either absent, or present in only trace amounts in the processes described in these patents. One of ordinary skill in the art would understand Singh and the ’930 patent as teaching the desirability of adding small amounts of aldehyde impurity inhibitors, but discouraging the use of water alone as an inhibitor because, when used alone, large quantities of water are required, upsetting the water balance within the reaction system. *See* Ex. 1011, 11:32–42; Ex. 2006, 11:1–10. Thus, one of ordinary skill in the art would expect that addition to a distillation column of an internal process stream containing small quantities of acetic acid and water for the purpose of inhibiting production of carbonyl compounds, as in Singh and the ’930 patent, would, at most, result in the production of an amount of DME that would be present in an insubstantial, or trace amount in the distillation overhead. Conversely, one of ordinary skill in the art would understand that where DME is mentioned explicitly as present in a process stream, it is present in more than an insubstantial amount, i.e., an amount that is greater than that expected to be produced during the course of a low water carbonylation process.

Based on our analysis of the intrinsic and extrinsic evidence, we agree with Patent Owner that a person of ordinary skill in the art would understand the ’507 patent to speak in terms of effects, not molecules. *See* PO Resp. 4–5; Tr. 41:8–16. We, therefore, construe the recitation “distilling [a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and

at least one PRC] to separate the mixture into a plurality of streams, at least one of said streams being a PRC enriched overhead stream comprising dimethyl ether" in step (a) of claim 58 as requiring the presence of dimethyl ether in the PRC enriched overhead stream in an amount that one of ordinary skill in the art would recognize as being more than trace or insubstantial. Such amount would be, for example, more than the amount produced in a conventional, prior art, low water carbonylation process that used the same initial reactor contents and conditions, and the same processing equipment. We further interpret this language as requiring the presence of DME in the PRC enriched overhead stream in an amount sufficient to cause (1) a reduction in the solubility of methyl iodide in water during the extraction of the PRC enriched overhead stream and (2) an increase in the amount of methyl iodide recoverable during the subsequent extraction of the first raffinate stream (claim 58, step (b)), which increase in recoverable methyl iodide would be recognized as more than insubstantial by the ordinary artisan.

VI. ALLEGED UNPATENTABILITY OF CLAIMS 58–60, 67, 69, and 70–73

Petitioner contends the '095 patent describes the process as recited in independent claim 58, with the exception that the presence of DME in the overhead streams is not expressly mentioned. Pet. 22 (citing Ex. 1039 ¶ 67). Petitioner contends "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.* Petitioner relies on scientific principles to show that DME is necessarily present in the second overhead in the '095 patent process. Pet. Reply 3–12. Petitioner also relies on the declaration testimony

of Mr. Miura regarding an experiment conducted to show DME was necessarily present in the ’095 patent Example. *Id.* at 12–19. Petitioner concedes the ’095 patent does not describe the aqueous extract streams as having “a methyl iodide concentration of less than or equal to about 0.5 wt %,” as recited in dependent claim 73, but contends this concentration was known in the art based on JP ’712’s disclosure that an MeI concentration of 0.1 wt% was known in the art for the relevant portion of the process. *Id.* at 22.

Patent Owner contends Petitioner has not met its burden to show by a preponderance of the evidence that DME is inherently present in the overhead stream in the ’095 patent process as required by claim 58, step (a). PO Resp. 13.[12]

A. Principles of Law

To prevail in its challenges to the patentability of the claims, a petitioner must establish facts supporting its challenges by a preponderance of the evidence. 35 U.S.C. § 316(e); 37 C.F.R. § 42.1(d).

To establish anticipation, each and every element in a claim, arranged as recited in the claim, must be found in a single prior art reference. *Net MoneyIN, Inc. v. VeriSign, Inc.*, 545 F.3d 1359, 1369 (Fed. Cir. 2008); *Karsten Mfg. Corp. v. Cleveland Golf Co.*, 242 F.3d 1376, 1383 (Fed. Cir. 2001). While the elements must be arranged or combined in the same way

[12] Patent Owner raises additional arguments with respect to dependent claims 59, 67, and 73. *See id.* at 52–59. We need not address these arguments because we are persuaded by Patent Owner’s argument with respect to independent claim 58.

as in the claim, "the reference need not satisfy an *ipsissimis verbis* test." *In re Gleave*, 560 F.3d 1331, 1334 (Fed. Cir. 2009).

> A single prior art reference that discloses, either expressly or inherently, each limitation of a claim invalidates that claim by anticipation. Thus, a prior art reference without express reference to a claim limitation may nonetheless anticipate by inherency. "Under the principles of inherency, if the prior art necessarily functions in accordance with, or includes, the claims limitations, it anticipates."

Perricone v. Medicis Pharm. Corp., 432 F.3d 1368, 1375–76 (Fed. Cir. 2005) (citations omitted). "In general, a limitation or the entire invention is inherent and in the public domain if it is the 'natural result flowing from' the explicit disclosure of the prior art." *Schering Corp. v. Geneva Pharms., Inc.*, 339 F.3d 1373, 1379 (Fed. Cir. 2003).

A claim is unpatentable under 35 U.S.C. § 103(a) if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. *KSR Int'l Co. v. Teleflex Inc.*, 550 U.S. 398, 406 (2007). The question of obviousness is resolved on the basis of underlying factual determinations, including: (1) the scope and content of the prior art; (2) any differences between the claimed subject matter and the prior art; (3) the level of skill in the art; and (4) objective evidence of nonobviousness, i.e., secondary considerations. *See Graham*, 383 U.S. at 17–18. An obviousness analysis "need not seek out precise teachings directed to the specific subject matter of the challenged claim, for a court can take account of the inferences and creative steps that a person of ordinary skill in the art would employ." *KSR*, 550 U.S. at 418.

We analyze the instituted ground of unpatentability in accordance with the above-stated principles.

B. The Expert Testimony

1. Mr. Miura

Mr. Miura testified as follows: The ’095 patent Example was performed under his supervision. *See* Ex. 1046, 40:14–41:3. Analyses of components in the various process streams in the ’095 patent Example were conducted using a gas chromatograph (“GC”) with a detector (FID, ECD, or TCD). *Id.* at 55:15–22, 57:4–5, 60:10–20, 61:10–21. Although the ’095 patent does not identify the presence of DME in the Example, Mr. Miura recalled that “DME was present in the process, and . . . there were DME peaks in the chart[, but it] was . . . not quantified” (*id.* at 65:14–19; *see also id.* at 63:1–6, 64:11–17). DME in the second overhead of the ’095 patent Example was accounted for in the .9 weight percent of components identified as “others.” *Id.* at 241:17–242:9 (explaining that “others” refers to components that were not analyzed, but an amount was determined by subtracting the total of the analyzed components from 100). Mr. Miura believed that DME likely was present on the order of parts per million. *Id.* at 66:1–5 (noting that a normal GC probably would not detect a component present on the order of ppb). Because the concentration of DME in the Example was low, the use of mass spectroscopy for detection would have been preferred. *See id.* at 60:21–61:9.

Mr. Miura further testified that there are many consecutive and simultaneous reactions taking place in a methanol carbonylation system, and the rates of these reactions are controlled by a number of factors, including: temperature; concentrations of the reaction materials, products, water, and

catalyst. *Id.* at 278:7–280:3. These factors could affect the quantity of DME present in the system, but he believed DME would still be present in an amount that was detectable. *Id.* at 280:4–281:1. "It's possible to analyze the individual reactions, but when there are reactions happening at the same time, then . . . it is extremely difficult to make an assumption on what the result may be, especially when it comes to a trace amount." *Id.* at 281:14–19.

Mr. Miura testified that "it is extremely difficult to explain theoretically the relationship between the respective reactions" in a methanol carbonylation system (*id.* at 281:2–4), so he conducted an experiment to prove the presence of DME in the '095 patent Example (*see id.* at 281:6–7; Ex. 1005 ¶ 7 ("[D]ata was gathered, in part, to determine trace level contaminants present in the streams described in the '095 Patent.")). The experiment ("Experiment 1") was conducted in 2011 and "was based upon [the] 'Example' and 'Example 1' as set forth in the '095 Patent." *Id*. There were "slight difference[s] in weight % of methyl iodide, water, acetic acid, lithium iodide, and rhodium in Experiment 1 as compared to . . . the '095 Patent [Example]," but these differences were "inconsequential to the outcome of the experiment relating to the presence of DME in the first and second overheads." *Id.* ¶ 21. More specifically,

> 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 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.

Id.

During cross-examination, Mr. Miura also acknowledged that there were some differences in operating conditions, such as flow rates, between the ’095 patent Example and Experiment 1, but maintained these differences would not have had any impact on whether Experiment 1 provides evidence that DME was present in the first and second overheads in the ’095 patent Example. Ex. 1046, 115:10–18; *see also id.* at 116:4–7 (wherein Mr. Miura testifies that the purpose of his declaration was to show the presence of DME in the first and second overheads); *see generally id.* at 107:4–116:7 (wherein Mr. Miura testifies regarding specific differences in operating conditions).

2. Mr. Jones

Mr. Jones testified as follows: The process described in the ’507 patent is “a variation on the Monsanto process.” Ex. 1039 ¶ 20. The ’507 patent implies that very low water concentrations, i.e., 4 weight percent or less, in the reaction medium are envisaged, and the ’095 patent similarly discloses a water content of 1 to 5 wt% in the reaction liquid (*id.* ¶ 50) and describes a low water carbonlyation process because the reaction liquid contains lithium (Ex. 2026, 264:4–8).

Mr. Jones testified that

> [t]he only apparent aspect of claim 58 of the ’507 patent, that is not expressly disclosed in the ’095 patent, is a recitation of dimethyl ether being present in the PRC enriched overhead stream. . . . [T]he ’507 Patent notes that the water in the system generates the DME, and that 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. . . . [B]ecause the ’095 Patent had already disclosed the addition of water in the

> process, the ’095 Patent must necessarily have disclosed the DME.

Ex. 1039 ¶¶ 70–71.

Mr. Jones testified that the ’095 patent Example uses an intermediate level of water, “not quite low enough to be what [he] would consider to be low water” (Ex. 2026, 264:16–20), and, therefore, the reaction mixture of the ’095 patent Example is more similar to the traditional high water process.” Ex. 1060 ¶ 40. Mr. Jones “read and understood Mr. Miura’s declaration and f[ou]nd that it indeed shows that DME is naturally present in processes run according to the ’095 Patent.” Ex. 1039 ¶ 68. Mr. Jones testified that Miura Experiment 1 used less water than the ’095 patent Example and, therefore, it would be expected that more dimethyl ether was produced in the ’095 patent Example than in Miura Experiment 1. *See* Ex. 2026, 293:18–294:13.

Mr. Jones testified that he made a “ball-park estimate of reactor methanol and DME concentrations in . . . the ‘095 Patent [Example] (neglecting mixture non-ideality) . . . using readily available data.” Ex. 1039 ¶ 97. Specifically, he used equilibrium concentrations provided in Figure 4 of Exhibit 1029, an article entitled “Methyl Acetate Hydrolysis in a Reactive Divided Wall Column,” and extrapolated to determine that at the temperature used in the ’095 patent Example, reactor methanol concentration was 411 ppm by weight. Ex. 1039 ¶ 97. Mr. Jones used equilibrium constants from Exhibit 1030, entitled “Thermodynamic Equilibrium Constants for the Methanol-Dimethyl Ether-Water System,” to approximate DME concentration in the reactor of the ’095 patent Example as 55 ppm. *Id*. Using the same calculation technique, Mr. Jones calculated

the reactor DME content in Miura Experiment 1 as 17 ppm. *Id.* According to Mr. Jones, the reactor DME would have been "concentrated very considerably . . . by distillation in the flasher 12, the splitter column 14, and the subsequent distillation, prior to aqueous extraction" (*id.*) and "would . . . necessarily have been present in the corresponding streams sent to aqueous extraction" (*id.* ¶ 103). Additional DME would have been produced in the distillation column due to the presence of water and methyl iodide. *Id.* ¶ 100; *see also id.* ¶¶ 99–100 (explaining the effects of water on the reaction).

Responsive to Dr. Porcelli's criticisms of his first declaration, Mr. Jones testified in his reply declaration that "ball-park estimates are routinely carried out by chemical engineers when an exact, quantitative number is not needed or not possible." Ex. 1060 ¶ 91. Although a professional modeling program is required to provide precise, quantitative predictions throughout the system, ball-park estimates are a sufficient and reliable method of demonstrating qualitatively the formation and presence of DME. *Id.* Mr. Jones recalculated DME content of the reactor in the '095 patent Example as 32.3 ppm (*id.* ¶ 113) using "a more accurate technique employing actual vapor pressures . . . to show that even more accurate modeling leads to approximately the same result with respect to the presence of DME" (*id.* ¶ 109). Mr. Jones also calculated the concentrations of DME in the reactor and process streams using the reaction components and conditions in Miura Experiment 1. *See id.* ¶¶ 146–153. Mr. Jones calculated the DME content in the splitter overhead in Miura Experiment 1 as 103 ppm, which he describes as "compar[ing] reasonably well with" Mr. Miura's measurement of 48 ppm. *Id.* ¶¶ 152–153. Mr. Jones contends the lower amount measured

by Mr. Miura is likely "due to loss of DME with the vent gas from the splitter condenser, due to the very low boiling point of DME." *Id.* ¶ 153.

In his declarations, Mr. Jones did not provide calculations estimating the quantity of DME that would have been present in the second overhead in the '095 patent, i.e., the overhead of distillation column 40. During his second deposition, however, Mr. Jones testified that he would expect the amount of DME in the second overhead in the '095 patent process to be about three times the amount of DME in the overhead in Miura Experiment 1. Ex. 2040, 289:10—298:7.

3. Dr. Porcelli

Dr. Porcelli testified as follows: "[T]he '507 patent is directed to the production of acetic acid using a low water process with an iodide salt as a catalyst stabilizer." Ex. 2029 ¶ 26. The '095 patent also describes a low water process. Ex. 1065, 169:2–7 ("[T]he reaction composition is 8 weight percent water. That's low water."); *cf. id.* at 80:14–15 ("[A]n improved aldehyde recovery scheme . . . implies a low water process."). Dr. Porcelli disagreed with Mr. Jones's characterization of a low water process as a variation on the Monsanto process, and explained the differences between the Monsanto and the low water carbonylation processes. Ex. 2029 ¶ 27. Dr. Porcelli testified, for example, that "the liquid reaction mixture in a low-water carbonylation system is known to be highly non-ideal." *Id.* ¶ 132. Dr. Porcelli testified that in

> the continuous carbonylation process described in the '095 patent[,] . . . reactants are continuously fed to the reactor as a crude product is continuously removed. The resulting extremely short residence times in such systems would compete against reaching equilibrium, resulting in a system that is

> referred to as a "rate limited" reaction system as opposed to an equilibrium limited" reaction system. There is no indication that the DME is formed at a rate equal to or faster than the rate at which it is consumed. This understanding is due to 1) little if any unreacted methanol in the reactor, 2) the fact that DME in the reactor would be above its critical temperature, and 3) because not all losses of any DME from the reactor vent are replaced through methanol dehydration, and 4) because DME would be immediately consumed by the carbonylation reaction."

Id. ¶ 121. Dr. Porcelli testified that "DME would be consumed by carbonylation faster than it would be formed by methanol dehydration, especially under low water conditions . . . due to the low concentration of methanol in the reactor and due to the low HI content," and that the conversion of DME to acetic acid is irreversible. *Id.* ¶¶ 130–131.

Dr. Porcelli testified that, like Mr. Miura, he believes "it is 'extremely difficult' to anticipate the presence of DME based on a theoretical relationship between the carbonylation reactions." *Id.* ¶ 112 (citing Ex. 1046, 280:13–281:7). Dr. Porcelli further testified as follows: There are numerous interactions between the solvent and other species present in a complex carbonylation liquid phase which influence activity of the components. *Id.* ¶ 136. Because Mr. Jones failed to consider activity coefficients, his calculated DME concentration is meaningless and unsupported. *Id.* ¶ 137. Ex. 1029 is directed to methyl acetate hydrolysis in a reactive divided wall column, which is a completely different reaction and system than in the '095 patent. *Id.* ¶ 138. In relying on Exhibit 1029 to calculate reactor methanol concentration, Mr. Jones failed to consider the competing reactions present in the '095 patent Example. *Id.* ¶ 139. Mr. Jones's extrapolation using Ex. 1029 was "well beyond the accepted practice

used by a chemical engineer and renders the values calculated by Mr. Jones meaningless. *Id.* ¶ 140. Mr. Jones's calculation of reactor DME content was based, in turn, on the flawed calculation of reactor methanol concentration, as well as Exhibit 1030, that uses a different catalyst and is based on a vapor phase, rather than liquid phase equilibrium constants. *Id.* ¶¶ 142–143. It was "improper and incorrect" for Mr. Jones "to fail to account for DME lost with the vent stream in the reactor, especially because of the high volatility of DME." *Id.* ¶ 148.

Dr. Porcelli testified that Mr. Jones's opinion that DME would be formed by the addition of water to the distillation column in the '095 patent is overly simplistic and does not support a conclusion that DME is necessarily formed in the column. *Id.* ¶ 151. More specifically, according to Dr. Porcelli, Mr. Jones failed to provide evidence that HI, required to form via methanol dehydration, was formed in the column, and failed to consider the number of trays in the column, point of introduction of water and the feed, the reflux components and ratio, and the residence time in the column. *Id.* ¶ 151.

Dr. Porcelli also testified that it is impossible to determine from Miura Experiment 1 whether DME was necessarily present in the '095 patent Example for several reasons, including significant differences in the reaction liquid compositions (*id.* ¶ 89), and the omission of numerous operational variables from both the '095 patent Example and Miura Experiment 1 (*id.* ¶¶ 108–109).

C. The Arguments

Patent Owner argues Mr. Jones's estimations of DME contents in the reactor and resulting overhead streams in the '095 patent Example and

Miura Experiment 1 are based on "numerous erroneous assumptions and critical omissions—falling far short of the standard for proving inherency." PO Resp. 15; *see also id.* at 16 ("[T]he Miura experiments *do not replicate* the conditions and compositions of . . . the '095 patent [Example] . . . and should be disregarded as irrelevant to the inherency issue.").

Petitioner contends Patent Owner "attempts to identify imperfections related to the quantitative accuracy of Mr. Jones' calculations," but "does not identify a single flaw in Mr. Jones' analysis that would (or even could) alter his conclusion that DME is necessarily present in the process of the '095 patent." Pet. Reply 3–4. Petitioner concedes the conditions used by Mr. Miura in his Experiment were not identical to those used in the '095 patent Example, but contends "Mr. Miura's experiment confirmed the presence of DME even under conditions less favorable to DME formation, supporting a conclusion that DME would necessarily exist in the process described in the '095 patent, thereby proving inherent anticipation." *Id.* at 15.

D. Analysis

In Section V.D., above, we construed the claim 58 recitation "distilling [a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and at least one PRC] to separate the mixture into a plurality of streams, at least one of said streams being a PRC enriched overhead stream comprising dimethyl ether" as requiring the presence of dimethyl ether in an amount sufficient to reduce solubility of methyl iodide in water during the subsequent water extraction of the overhead to separate the PRC(s) and methyl iodide. We stated that this amount would be more than what one of ordinary skill in the art would consider an insubstantial

amount, e.g., the amount produced, if any, in the high water Monsanto process or the prior art low water carbonylation process for production of an acetic acid product from methanol, methyl acetate, methyl formate and/or dimethyl ether.

Neither Mr. Miura nor Mr. Jones testified that dimethyl ether would have been present in the distillation overhead in the ’095 patent Example in an amount sufficient to reduce solubility of methyl iodide in water during subsequent water extraction steps. Mr. Miura testified that 1.2 wt% DME was present in the second overhead in Experiment 1. Ex. 1005 ¶ 13, Table 1. Mr. Jones testified that he would expect the amount of DME in the second overhead in the ’095 patent process to be about three times the amount of DME in the overhead in Miura Experiment 1 (Ex. 2040, 289:10—298:7), because Mr. Miura’s experiment was carried out under conditions less favorable to DME formation as compared to the conditions in the ’095 patent Example (*see* Ex. 1060 ¶¶ 130, 139). A preponderance of the evidence, however, does not support a finding that DME was present, if at all, in more than a trace quantity/insubstantial amount in the second overhead of the ’095 patent Example, for the following reasons:

Although Mr. Miura reported that the second overhead in his Experiment 1 contained 1.2% DME (Ex. 1005 ¶ 13, Table 1), he did not testify that this quantity would be present in the ’095 patent Example despite acknowledged differences in quantities of reaction components and operating conditions (*see id.* ¶ 21). Rather, Mr. Miura testified that Experiment 1 provided evidence that DME simply was present in some amount in the second overhead in the ’095 patent Example. *Id.* (“[T]he deviations would not affect the outcome with respect to the presence of

DME in the first and second overheads.”). Mr. Miura testified that he supervised the ’095 patent Example and believed DME was present and accounted for in the 0.9 wt% reported as “others.” Ex. 1046, 241:17–242:9. Therefore, we are not persuaded by Mr. Jones’s testimony that the amount of DME in the second overhead in the ’095 patent process would be about three times the amount of DME in the overhead in Miura Experiment 1, i.e. 3.6 wt% (Ex. 2040, 289:10—298:7). *See* Tr. 70:3–8 (wherein Patent Owner’s counsel raises this inconsistency). We are not persuaded by Mr. Jones’s testimony that an amount as high as 3.6 wt% DME could have been present in the ’095 patent Example, but not reported (*see* Ex. 2040, 301:18–303:2; Paper 73, 5–6 (Observation #C.4)), since the prior art, e.g., Paulik, clearly indicates DME was considered an undesirable reaction byproduct in a carbonylation process. Mr. Miura did not testify as to what portion of the 0.9 wt% reported as “others” in the ’095 patent Example constituted DME, or whether the amount of DME would be considered more than a trace quantity/insubstantial amount. Nor is there any testimony or evidence that 0.9 wt% or less DME in the overhead/feed to the extractor would have been an amount sufficient to cause a reduction in the solubility of methyl iodide in water during extraction of the PRC enriched overhead stream and an increase in the amount of methyl iodide recoverable during the subsequent extraction of the first raffinate stream as required under the broadest reasonable interpretation of claim 58 of the ’507 patent.

We are not persuaded that Mr. Jones’s calculations provide a reliable method of determining the amount of DME present in a methanol carbonylation process. Mr. Miura and Dr. Porcelli agreed that it is extremely difficult to anticipate the presence or amount of a component such

as DME in a methanol carbonylation system such as the one described in the ’095 patent. *See* Ex. 1046, 281:14–22; Ex. 2029 ¶ 112. Dr. Porcelli testified that Mr. Jones’s estimates of DME content in the ’095 patent Example were based on numerous assumptions that rendered his estimations of reactor and distillation overhead DME concentrations meaningless. *See* Ex. 2029 ¶¶ 137, 140, 151. Dr. Porcelli testified, for example, that it was “improper and incorrect” for Mr. Jones “to fail to account for DME lost with the vent stream in the reactor, especially because of the high volatility of DME.” *Id.* ¶ 148. Mr. Miura’s measured amount of DME present in the first overhead in Experiment 1 was less than half the amount Mr. Jones calculated would be present, supporting Dr. Porcelli’s testimony that the loss of DME via the vents would have had a marked impact on the actual amount of DME sent to distillation column 40 (from which the second overhead stream is produced) in the ’095 patent Example. *See* Ex. 1060 ¶ 153 (“(103 ppm (calc.) vs. 48 ppm (actual)) is likely to due to loss of DME with the vent gas from the splitter condenser, due to the very low boiling point of DME”); *see also* Ex. 1039 ¶ 97; Ex. 1060 ¶ 121 (wherein Mr. Jones agreed that the loss of DME from the vent stream in the separator (or decanter) after splitter column 14 in the ’095 patent process would be substantial).

Dr. Porcelli testified that he believed removal of DME by rapid carbonylation would have had a significant effect on the presence of DME in the ’095 patent Example and that this irreversible reaction should have been considered by Mr. Jones in his calculations. Ex. 2029 ¶¶ 130–131. Mr. Jones conceded “some DME will be carbonylated in the reactor” (Ex. 1060 ¶ 45), and that the carbonylation of DME to make acetic acid under low water conditions is an irreversible reaction (Ex. 2026, 227:19–228:1). *See*

also Ex. 1060 ¶ 27 (arguing Exhibits 2032, 2034 evidence DME formation in a batch reactor during methanol carbonylation, but admitting that as the water and methyl acetate start to diminish, DME concentration begins to drop and DME is eventually carbonylated into either acetic acid or acetic anhydride). Mr. Jones disagreed with Dr. Porcelli's testimony that "carbonylation of DME is . . . known to have a fast reaction rate" and "would be consumed by carbonylation faster than it would be formed by methanol dehydration, especially under low water conditions" (Ex. 2029 ¶ 130). Ex. 1060 ¶ 44. Mr. Jones provided testimony explaining why he believes "the carbonylation of DME is not particularly fast." *Id.*; *see id.* ¶¶ 45–48. Mr. Jones relied on a textbook by Gauthier-Lafaye (Ex. 1062), published in 1987, as evidence that the carbonylation of DME proceeds more slowly than the carbonylation of methyl acetate. Ex. 1060 ¶ 47. Mr. Jones testified that because the reaction liquid in the '095 patent Example is reported to contain 1.6 wt% methyl acetate, and the carbonylation rate of DME is even slower than that of methyl acetate, then "it stands to reason that the carbonylation rate of DME is not going to be so fast as to 'immediately' react away any DME that is formed via the methanol dehydration equilibrium reaction." *Id.* ¶ 48. Mr. Jones further testified that consideration of this competing side reaction was "irrelevant to calculating equilibrium concentration" of DME in the reactor in the '095 patent Example. *Id.* ¶ 51.

The disclosure in Gauthier-Lafaye relied upon by Mr. Jones relates to the carbonylation of methyl acetate into acetic anhydride (Ex. 1062, 9), i.e., a different reaction system than the '095 and '507 patents (*see* Paper 73, 8 (Observation #F.1)). This evidence, therefore, does not support Mr. Jones's

testimony that the carbonylation of DME in the ’095 patent reaction system was irrelevant to his calculation of equilibrium DME concentration. Rather we credit Dr. Porcelli’s testimony that removal of DME by rapid carbonylation would have had an effect on the presence, or at least the quantity, of DME in the ’095 patent Example, and that this factor should have been taken into consideration by Mr. Jones in his calculations. *See* Ex. 2029 ¶ 130.

Mr. Jones testified that

> in view of the low boiling point of DME (-23.7 ºC), most of the DME will immediately flash from the reactor product . . . and be concentrated in the overhead streams. In view of the relatively low amount of DME in the reactor and overhead, DME’s very low boiling point, and the elevated temperatures in the reactor, special handling techniques would be needed to sample for such a compound in the reaction mixture. It is therefore not surprising that the presence of DME was not measured in the ’095 patent, despite the fact that it was undoubtedly present.

Ex. 1060 ¶ 118. Given Mr. Miura’s testimony that he believed DME, if present, would have been detectable (Ex. 1046, 280:9–281:1), and Paulik’s disclosure that insubstantial amounts of DME were measurable as early as 1970 (*see, e.g.*, Ex. 2037, 13:47–49, 15:32–34), Mr. Jones’s testimony (*see, e.g.*, Ex. 1060 ¶ 101 (“It should be clear to someone skilled in the art that very dilute and trace components are frequently not reported in chemical analytical results or in listed process stream compositions.”)) does not show convincingly that DME was present in the second overhead in more than a trace quantity, if at all. *See id.* ¶ 90 (“[T]he only issue is whether some amount of DME exists (which clearly it does).”).

In sum, we determine Petitioner has failed to meet its burden to show,

by a preponderance of the evidence, that the ’095 patent describes a process that inherently results in dimethyl ether in a PRC enriched overhead stream (produced by distilling a mixture comprising water, acetic acid, methyl iodide, methyl acetate, methanol, and at least one PRC) in an amount that is (1) more than trace or insubstantial, and (2) sufficient to cause (a) a reduction in the solubility of methyl iodide in water during extraction of the PRC enriched overhead stream and (b) an increase in the amount of methyl iodide recoverable during the subsequent extraction of the first raffinate stream, as required by the broadest reasonable interpretation of claim 58. Absent from the record is any evidence that DME is present in the second overhead in the ’095 patent Example in the amount required by independent claim 58 and its dependent claims 59, 60, 67, 69, and 70–73. Petitioner’s challenge with respect to claim 73 is based on obviousness over the combination of the ’095 patent and JP ’712. Petitioner relies on JP ’712 for a teaching that “methyl iodide concentration can be controlled by extraction temperature, . . . and that the concentration[] recited in [claim 73 is] well known in the art.” Pet. 49. Petitioner has not explained how JP ’712 cures the deficiency in the ’095 patent’s failure to describe step (a) of claim 58. Accordingly, we likewise determine Petitioner has failed to meet its burden to show unpatentability of claim 73 by a preponderance of the evidence. We conclude Petitioner has failed to meet its burden to show unpatentability of claims 58–60, 67, 69, and 70–73 of the ’507 patent.

VII. MOTIONS

Patent Owner filed a Motion to Exclude the declaration of Mr. Hiroyuki Miura (Ex. 1005). Paper 74. The party moving to exclude

evidence bears the burden of proving that it is entitled to the relief requested—namely, that the material sought to be excluded is inadmissible under the Federal Rules of Evidence. *See* 37 C.F.R. §§ 42.20(c), 42.62(a). Patent Owner also filed a Motion for Observation on the cross-examination testimony of Mr. Jones. Paper 73.

A. Motion to Exclude the Miura Declaration

Patent Owner requests that we "exclude the declaration of Mr. Hiroyuki Miura (Ex. 1005) pursuant to 37 C.F.R. § 42.64(c) or, alternatively, . . . give no weight and an adverse inference to this evidence pursuant to 37 C.F.R. § 42.65(a)." Paper 74, 1. Patent Owner argues Mr. Miura's opinions regarding an Experiment described in his declaration and the Example in the '095 patent cannot be deemed reliable because neither Petitioner nor Mr. Miura supplied the underlying data in its entirety, and the data that was provided is misleading. *See id.* at 1–4.

Patent Owner's arguments are directed to the weight we should accord Mr. Miura's testimony, rather than its admissibility. It is within our discretion to assign the appropriate weight to the testimony offered by Mr. Miura. *See, e.g.*, *Yorkey v. Diab*, 601 F.3d 1279, 1284 (Fed. Cir. 2010) (holding the Board has discretion to give more weight to one item of evidence over another "unless no reasonable trier of fact could have done so"); *In re Am. Acad. of Sci. Tech Ctr.*, 367 F.3d 1359, 1368 (Fed. Cir. 2004) ("[T]he Board is entitled to weigh the declarations and conclude that the lack of factual corroboration warrants discounting the opinions expressed in the declarations."). We considered Mr. Miura's declaration in our analysis of Petitioner's patentability challenges and concluded it was not persuasive evidence that the second distillation overhead in the '095 patent Example

contained DME in the amount required by claim 58 of the ’507 patent. *See* Section VI.D.

Accordingly, Patent Owner’s motion is denied.

B. Motion for Observation

Patent Owner filed a Motion for Observation regarding the cross-examination of Mr. Jones (Paper 73) and Petitioner filed a response (Paper 78). We have taken into account the respective positions of Patent Owner and Petitioner in this Final Written Decision, as indicated, e.g., in Section VI.D.

VIII. CONCLUSION

Petitioner has not shown by a preponderance of the evidence that:

(1) claims 58–60, 67, 69, 71, and 72 of the ’507 patent are unpatentable under 35 U.S.C. § 102(b) based on anticipation by the ’095 patent; and

(2) claim 73 of the ’507 patent is unpatentable under 35 U.S.C. § 103(a) based on obviousness over the ’095 patent and JP ’712.

IX. ORDER

In consideration of the foregoing, it is hereby:

ORDERED that claims 58–60, 67, 69, 71, and 72 of U.S. Patent No. 8,076,507 B2 are not held unpatentable under 35 U.S.C. § 102(b) based on anticipation by the ’095 patent;

FURTHER ORDERED that claim 73 of U.S. Patent No. 8,076,507 B2 is not held unpatentable under 35 U.S.C. § 103(a) based on obviousness over the ’095 patent and JP ’712;

FURTHER ORDERED that Patent Owner’s Motion to Exclude the Declaration of Mr. Hiroyuki Miura pursuant to 37 C.F.R. § 42.64(c) or,

alternatively, to give no weight and an adverse inference to this evidence pursuant to 37 C.F.R. § 42.65(a) is *denied*;

FURTHER ORDERED that, because this is a final decision, parties to the proceeding seeking judicial review of the decision must comply with the notice and service requirements of 37 C.F.R. § 90.2.

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