UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

ARKEMA FRANCE,

Petitioner,

v.

HONEYWELL INTERNATIONAL, INC.,

Patent Owner.

IPR No. IPR2015-00916

U.S. Patent No. 8,710,282

PETITION FOR *INTER PARTES* REVIEW UNDER 35 U.S.C. § 311 & 37 C.F.R. 42.101

TABLE OF CONTENTS

I.	INTRODUCTION			
II.	CERTIFICATION OF GROUNDS FOR STANDING1			
III.	MANDATORY NOTICES			
IV.	OVERVIEW OF THE '282 PATENT			
	A.	Overview of the Prosecution History and Effective Priority Date of the Claims of the '282 Patent	5	
V.	OVERVIEW OF CHALLENGE PURSUANT TO 37 C.F.R. 42.104(B)			
	A.	Prior Art & Evidence Relied Upon	9	
	B.	Grounds for Challenge	10	
VI.	CLAIM CONSTRUCTION			
	A.	"Spent DHA"	12	
	B.	"Withdrawing"	14	
	C.	"Recovering Spent DHA"	15	
	D.	"Recycling"	17	
	E.	"Dehydrofluorinated Product Stream" or "Product Stream"	17	
	F.	"Product Reaction Stream", "Second Product Stream", or "Reaction Stream"	17	
	G.	"Dissolved Organics"	18	
VII.			19	
VIII.	SUMMARY OF PRIOR ART19			
	A.	State of the Art	19	
	B.	Summary of Primary Prior Art References	20	
IX.	THERE IS A REASONABLE LIKELIHOOD THAT THE CHALLENGED CLAIMS OF THE '282 PATENT ARE UNPATENTABLE			
	A.	Claims 1-20 are Obvious over <i>Smith</i> in View of <i>Masayuki</i> and <i>Harrison</i>		
		1. Claim 1	25	

IPR2015-00916

Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282

2.	Claim 2-6	38
3.	Claim 7	39
4.	Claim 8	40
5.	Claim 9	41
6.	Claim 10	41
7.	Claims 11 and 12	46
8.	Claims 13 and 14	48
9.	Claim 15	50
10.	Claim 16	52
11.	Claim 17	53
12.	Claim 18	53
13.	Claim 19	54
14.	Claim 20	56
CONCLUS	ION	57

Х.

IPR2015-00916 Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282

LIST OF EXHIBITS

Exhibit No.	Document	Abbreviation	
Ex. 1001	U.S. Patent No. 8,710,282	'282 patent	
Ex. 1002 Declaration of Leo E. Manzer, Ph.D.		Manzer Dec.	
Ex. 1003	International Publication No. WO 2009/138764 A1	Smith	
Ex. 1004	Japanese Publication No. JP S59-70626	Masayuki	
Ex. 1005	English language translation of Japanese Publication No. JP S59-70626, and the Declaration of Jonathan Kent attesting to the accuracy of the translation	Masayuki	
Ex. 1006	U.S. Patent No. 4,414,185	Harrison	
Ex. 1007	Manual of Patent Examining Procedure (9th ed. 2014)	MPEP	
Ex. 1008	L. Knunyants et al., <i>Reactions of Fluoro</i> <i>Olefins</i> , 9 Bulletin of the Academy of Sciences of the USSR 1312 (1960)	Knunyants	
Ex. 1009	U.S. Publication No. 2009/0278075 A1	Mahler	
Ex. 1010	International Publication No. WO 2007/056194 A1	Mukhopadhyay	
Ex. 1011	U.S. Provisional Application No. 61/392,242	'242 provisional	
Ex. 1012	U.S. Provisional Application No. 61/036,526	'526 provisional	
Ex. 1013	U.S. Patent Application No. 12/402,372	'372 non-provisional	
Ex. 1014	U.S. Patent Application No. 13/195,429	'429 non-provisional	
Ex. 1015	Prosecution History for U.S. Patent No. 8,710,282	'282 file wrapper	
Ex. 1016	J. M. Douglas, Conceptual Design of Chemical Processes (1988)	Douglas	
Ex. 1017	Declaration of Brian Durrance		
Ex. 1018	Declaration of Joshua E. Ney		

I. INTRODUCTION

Arkema France ("Petitioner") requests *inter partes* review ("IPR") under 35 U.S.C. §§ 311-319 and 37 C.F.R. § 42.100 *et seq.* of claims 1-20 of U.S. Patent No. 8,710,282 ("the '282 patent") to Bektesevic et al., titled "Integrated Process for the Manufacture of Fluorinated Olefins". *See* Ex. 1001.

Two (2) additional petitions for IPR of the '282 patent are being filed contemporaneously with the filing of the instant petition. The numbers for these concurrent petitions are as follows: IPR2015-00915 and IPR2015-00917. In the event that Patent Owner raises 35 U.S.C. § 325(d), the filing of multiple petitions is justified in this instance because of uncertainty as to whether the Patent Owner will attempt to antedate one of the prior art references relied upon in IPR2015-00915. The effective dates of the prior art references cited in support of the instant petition and the IPR2015-00917 petition are earlier than the effective date of at least one of the prior art references cited in IPR2015-00915. The petitions in the instant proceeding and IPR2015-00917 are not duplicative or cumulative of each other because they address different claims (1-20 in the former and 21-46 in the latter).

II. CERTIFICATION OF GROUNDS FOR STANDING

Petitioner certifies under 37 C.F.R. § 42.104(a) that the '282 patent for which review is sought is available for IPR, and that the real parties-in-interest are

not barred or estopped from requesting IPR of any claim of the '282 patent on the grounds set forth herein.

Petition for Inter Partes Review of U.S. Pat. No. 8.710,282

III. MANDATORY NOTICES

Arkema France, Arkema, Inc., and Arkema S.A. are the real parties-in-IPR2015-00915 and IPR2015-00917 are the only other judicial or interest. administrative matters that would affect, or be affected by, a decision in this proceeding. Pursuant to 37 C.F.R. §§ 42.8(b)(3) and 42.10(a), Arkema France designates the following counsel: Lead Counsel is Jon Beaupré (Reg. No. 54,729); Back-up Counsel are Allen R. Baum (Reg. No. 36,086), Allyn B. Elliott (Reg. No. 56,745), Joshua E. Ney (Reg. No. 66,652), and Nicholas A. Restauri (Reg. No. 71,783). Service information is as follows: Brinks Gilson & Lione, 524 South Main St., Suite 200, Ann Arbor, MI 48104, 734.302.6000, 734.994.6331 (fax). Arkema France service electronic consents to by mail at jbeaupre@brinksgilson.com and Arkema_HoneywellIPRs@brinksgilson.com. Α Power of Attorney is filed concurrently herewith under 37 C.F.R. § 42.10(b).

The Petition Fee of \$25,000 is paid concurrently with the filing of this Petition by Deposit Account 23-1925. The undersigned representative of Petitioner hereby authorizes the Patent Office to charge any additional fees or credit any overpayment to deposit account 23-1925.

IV. OVERVIEW OF THE '282 PATENT

The '282 patent was filed on September 9, 2011 and issued on April 29, 2014. According to USPTO records, Honeywell International Inc. (Morristown, New Jersey) is its assignee.

In general, the process described in the '282 patent includes the following steps: (a) hydrogenating a first haloolefin to produce a haloalkane; (b) optionally separating said haloalkane into a plurality of intermediate product streams comprising two or more streams selected from the group consisting of a first stream rich in at least a first alkane, a second stream rich in a second alkane and an alkane recycle stream; (c) dehydrohalogenating the haloalkane from (a) or (b) in the presence of a dehydrohalogenating agent [hereinafter "DHA"] to produce a second haloolefin; (d) withdrawing a reaction stream comprising spent DHA and, optionally, metal fluoride salt by products; and (e) recovering, purifying and/or regenerating spent DHA. Ex. 1001 at 3:4-17. It is noted that step (b) is optional and not required by any of the claims of the '282 patent.

As admitted in the Background section of the '282 patent (and demonstrated by other prior art not cited during prosecution), the hydrogenation and dehydrofluorination steps claimed in the '282 patent were known. *Id. at* 1:48-2:33. Against this background prior art, the "invention" is summarized as directed "... to

a method of increasing the cost efficiency for dehydrohalogenation production of a

fluorinated olefin by recovering and recycling spent reagent." Ex. 1001 at 2:49-52.

Representative claim 1 provides as follows:

- 1. A method for producing at least one fluorinated olefin comprising:
- a. hydrogenating a starting material stream comprising at least one alkene according to Formula (I):

 $(CX_nY_{3-n})(CR^1_aR^2_b)_zCX=CH_mX_{2-m}$ (I)

by contacting said starting material with a reducing agent to produce an intermediate product stream comprising at least one alkane according to Formula (II):

$$(CX_nY_{3-n})(CR^1_aR^2_b)_zCHXCH_{m+1}X_{2-m}$$
(II)

where:

- each X is independently Cl, F, I or Br, provided that at least two Xs are F;
- each Y is independently H, Cl, F, I or Br;
- each R¹ is independently H, Cl, F, I, Br or unsubstituted or halogen substituted methyl or ethyl radical;
- each R² is independently H, Cl, F, I, Br or unsubstituted or halogen substituted methyl or ethyl radical;

n is 1, 2 or 3;

- a and b are each 0, 1 or 2, provided that a+b=2;
- m is 0, 1 or 2; and
- z is 0,1,2 or 3;
- b. optionally, separating said intermediate product stream into a plurality of intermediate product streams, said plurality of intermediate product streams comprising two or more streams

selected from the group consisting of a first stream rich in at least a first alkane according to Formula II, a seconds stream rich in at least a second alkane according to Formula II, and an alkane recycle stream;

- c. dehydrofluorinating, in the presence of a dehydrohalogenating agent, at least a portion of said intermediate process stream from step (a) or said plurality of intermediate process streams of step (b) to produce an alkene product stream comprising 1,1,1,2,3-pentafluoropropene and at least one additional alkene having a lower degree of fluorine substitution compared to the degree of fluorination of the compound of formula (I); and
- d. withdrawing from a dehydrofluorinated product stream a product reaction stream comprising spent dehydrohalogenating agent; and

e. recovering spent dehydrohalogenating agent.

As explained below, the absence of the "withdrawing" and "recovering" steps in the art cited by the Examiner led to allowance of the '282 patent. However, as also explained below, prior art references not cited during prosecution disclose these "withdrawing" and "recovering" steps in combination with the "dehydrohalogenating" step, and it would have been obvious to practice the claimed methods.

A. Overview of the Prosecution History and Effective Priority Date of the Claims of the '282 Patent

The '282 patent was filed on September 9, 2011, and prosecuted as application no. 13/229,016 ("the '016 application"). The '282 patent claims

priority to two provisional applications and two nonprovisional applications. However, as discussed in more detail below, the earliest possible priority date for the claims of the '282 patent is October 12, 2010, the filing date of provisional application no. 61/392,242 (hereinafter "the '242 provisional," Ex. 1011). This earliest possible priority date is consistent with the Examiner's untraversed findings set forth below.

Petition for Inter Partes Review of U.S. Pat. No. 8.710.282

The '282 patent claims, but is not entitled to, priority to the following applications: provisional application no. 61/036,526 (hereinafter "the '526 provisional," Ex. 1012), filed on March 14, 2008; non-provisional application no. 12/402,372 (hereinafter "the '372 non-provisional," Ex. 1013), filed March 11, 2009; and non-provisional application no. 13/195,429 (hereinafter "the '429 non-provisional," Ex. 1014), filed on August 1, 2011. A diagram of the claims of priority for the '282 patent is provided below for reference.¹

¹ The Related U.S. Application Data on the face of the '282 patent characterizes the '429 non-provisional as a continuation-in-part of the '372 non-provisional. This characterization contradicts the specifications of the '282 patent (Ex. 1001 at 1:14-16) and the '429 non-provisional (Ex. 1014 at [0001]), both of which characterize the '429 non-provisional as a continuation of the '372 non-



For a claim in a later-filed application to be entitled to an earlier filing date, the earlier application must comply with the requirements of 35 U.S.C. § 112, ¶1. *See* 35 U.S.C. §§ 119(e), 120. That is, the specification must "contain a written description of the invention, and of the manner and process of making and using it." 35 U.S.C. § 112, ¶1.

The claims of the '282 patent are not entitled to the filing dates of any of the '526 provisional, the '372 non-provisional, or the '429 non-provisional applications (collectively, "the Catalysis Applications") because these applications

provisional. Petitioner herein treats the '429 non-provisional as a continuation of the '372 non-provisional.

do not explicitly or implicitly disclose, support, or enable "withdrawing" spent DHA or "recovering" spent DHA, as required by independent claims 1 and 10. *See* Ex. 1007 at Chapter 21, Section 2163(II)(A)(3)(b); Ex. 1002 at ¶ 43. The dependent claims incorporate the limitations of the claims from which they depend, and therefore the dependent claims also lack support in the Catalysis Applications.

Petition for *Inter Partes* Review of U.S. Pat. No. 8.710,282

The Catalysis Applications are directed to gas phase dehydrohalogenation in the presence of a solid catalyst rather than the DHA-mediated dehydrohalogenation process claimed in the '282 patent, and thus they do not teach anything with respect to the use of a DHA (e.g., KOH) for dehydrohalogenation or with respect to withdrawing or recovering spent DHA or KOH. Ex. 1002 at ¶ 43. Furthermore, a person of ordinary skill would not understand the descriptions of the Catalysis Applications to require withdrawing or recovering spent DHA. *Id.* On the contrary, since the Catalysis Applications do not describe a DHA-mediated process, the concepts of withdrawing and recovering spent DHA are completely absent therefrom. *Id.* Therefore, the '282 patent should not be awarded a priority date prior to the October 12, 2010, filing date of the '242 provisional.

This conclusion is consistent with the Examiner's unrebutted statements during prosecution of the '282 patent. During prosecution, when noting that claims 1-10 and 21-46 were allowed, the Examiner stated that U.S. Publication No.

2009/0234165 ("D1"), the U.S. publication of the '372 non-provisional, "differs from instant claim 1 in d) withdrawal of the product stream comprising the dehydrohalogenating agent and e) recovery of spent dehydrohalogenating agent. Withdrawal and recovery is not depicted, taught or suggested in D1." Ex. 1015 at June 6, 2013, Office Action, page 3 (Production Page 0000337).

Petition for Inter Partes Review of U.S. Pat. No. 8.710.282

Honeywell did not traverse or otherwise dispute this finding. *See* Ex. 1015, Aug. 29, 2013, Reply (Production Pages 0000394-403). Honeywell should not be permitted to argue now that "withdrawal" and "recovery" are somehow taught in the '372 non-provisional application or its progeny.

V. OVERVIEW OF CHALLENGE PURSUANT TO 37 C.F.R. 42.104(B)

Pursuant to 37 C.F.R. §§ 42.22(a)(1) and 42.104(b), Petitioner challenges claims 1-20 of the '282 patent.

A. Prior Art & Evidence Relied Upon

Petitioner relies on the following prior art to the '282 patent:² (1) PCT No. PCT/GB2009/001263 ("*Smith*"; Ex. 1003), filed May 15, 2009, published as

² During prosecution of the '282 patent, the Examiner considered Knunyants,

Mahler, and a U.S. counterpart to *Smith*. However, as discussed below, Petitioner does not rely on these references to teach the purported point of novelty identified by the Examiner during prosecution.

WO2009/138764 (English) on November 19, 2009, and prior art under at least 35 U.S.C. § 102(e); (2) Japanese Pub. No. JP S59-70626 (Ex. 1004), published April 21, 1984, and prior art under 35 U.S.C. § 102(b), an English language translation of which is provided herewith as Ex. 1005, pursuant to 37 C.F.R. § 42.63 (*"Masayuki"*; Ex. 1005); and (3) U.S. Patent No. 4,414,185 (*"Harrison"*; Ex. 1006), published Nov. 8, 1983, and prior art under 35 U.S.C. § 102(b).

Petitioner also relies on the following additional evidence: (1) Declaration of Leo E. Manzer, Ph.D (Ex. 1002); (2) L. Knunyants et al., *Reactions of Fluoro Olefins*, 9 Bulletin of the Academy of Sciences of the USSR 1312 (1960) (*"Knunyants"*; Ex. 1008), prior art under 35 U.S.C. § 102(b) (*see* Ex. 1017 at ¶ 11(available as of at least 1961)); (3) U.S. Publication No. 2009/0278075 A1 (*"Mahler"*; Ex. 1009), published Nov. 12, 2009, and prior art under at least 35 U.S.C. § 102(a); (4) Int'l Pub. No. WO2007/056194 (*"Mukhopadhyay"*; Ex. 1010), published May 18, 2007, and prior art under 35 U.S.C. § 102(b); and (5) J. M. Douglas, *Conceptual Design of Chemical Processes* (1988) (*"Douglas"*; Ex. 1016), prior art under 35 U.S.C. § 102(b).

B. Grounds for Challenge

Petitioner requests cancellation of claims 1-20 of the '282 patent on the ground that such claims are obvious under 35 U.S.C. § 103(a) over *Smith* in view of *Masayuki* and *Harrison*.

As demonstrated below, there is a reasonable likelihood that Petitioner will prevail with respect to at least one of the challenged claims. *See* 35 U.S.C. § 314(a).

VI. CLAIM CONSTRUCTION

During IPR, a claim is to be given its "broadest reasonable construction in light of the specification." *See* 37 C.F.R. § 42.100(b). Claim terms "are generally given their ordinary and customary meaning," which is "the meaning that the term would have to a person of ordinary skill in the art in question at the time of the invention." *Phillips v. AWH Corp.*, 415 F.3d 1303, 1312-13 (Fed. Cir. 2005). The discussion below provides construction of certain terms of the '282 patent claims. Petitioner's construction of claim terms is not binding upon Petitioner in any litigation related to the '282 patent. Petitioner submits, for the purposes of this *inter partes* review only, that the claim terms not specifically addressed in this section take on the ordinary and customary meaning that they would have to one of ordinary skill in the art in view of the specification of the '282 patent.

The terms "spent", "withdrawing", "recovering", "recycling", "product stream" (and certain variants thereof), "reaction stream" (and certain variants thereof), and "dissolved organics" require construction. None of these terms are explicitly defined in the '282 patent. The terms should be construed as follows:

IPR2015-00916 Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282

Term	Construction
"spent DHA"	"unreacted DHA remaining after the
	dehydrohalogenation reaction"
"withdrawing"	"taking out or removing"
"recovering spent DHA"	"collecting spent DHA for further use (may include
	separating, purifying, concentrating, and/or
	regenerating)"
"recycling"	"returning a component of the process to the
	process"
"dehydrofluorinated	"a volume of material resulting from
product stream" or	dehydrofluorination including dehydrofluorinated
"product stream"	organic product(s)"
"product reaction stream",	"a volume of material including spent DHA that
"second product stream",	may also contain by-product salts and dissolved
or "reaction stream"	organics"
"dissolved organics"	"organic that is dissolved in a stream"

A. "Spent DHA"

The term "spent DHA", which is used in independent claims 1 and 10, should be construed to mean "unreacted DHA" remaining after the dehydrohalogenation reaction. Ex. 1002 at $\P\P$ 45-46.

Examples of the dehydrohalogenation reactions discussed in the '282 patent are described by the following reaction equations (1) and (2):

$$CF_3$$
— CHF — CHF_2 + KOH \rightarrow CF_3CF = CHF + KF + H_2O (1)

$$CF_3$$
— CHF — CH_2F+KOH $\rightarrow CF_3CF=CH_2+KF+H_2O$ (2)

Ex. 1001 at 12:26-29. In view of these equations, a person of ordinary skill in the art ("POSITA") could understand the term "spent DHA" to mean either (1) unreacted DHA remaining after the dehydrohalogenation reaction or (2) a byproduct salt of the DHA (e.g., KF). Ex. 1002 at ¶ 45. However, after reviewing the specification of the '282 patent, a POSITA would understand that "spent DHA" does not refer to DHA that has been converted to a by-product salt (e.g., KF) because the patent distinguishes between "spent DHA" and "by-product salts". For example, the '282 patent states "... (d) withdrawing a reaction stream comprising spent [DHA] and, optionally, metal fluoride salt by products ...". Ex.1001 at 3:13-15. Further, in the "Dehydrohalogenation" section of the '282 patent starting at 12:11, spent DHA and by-product salts are referred to as two separate components: "[s]pent [DHA] and by-product salts (e.g., metal fluoride salts) may be withdrawn from the reactor by a product stream either continuously or intermittently using one or more separation techniques". See, e.g., id. at 13:16-Thus, a POSITA would understand that the term spent DHA relates to DHA 19.

Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282 in its unconverted form and would understand spent DHA to mean unreacted DHA remaining after the dehydrohalogenation reaction. Ex. 1002 at ¶ 46.

B. "Withdrawing"

The term "withdrawing" as used in all of the independent claims should be construed as "taking out or removing". Ex. 1002 at ¶¶ 47-49. This construction is supported by the description of the process in the specification of the '282 patent. The process described includes the "[r]emoval of spent [DHA] from the reactor..." Ex. 1001 at 2:61-63. The examples in the specification further support this construction. In Examples 1 and 2, spent KOH is removed from the reactor: "Organic out (the overhead portion) was constantly taken out [of the reactor]" and "valves for the spent KOH to go out [of the reactor] were open". *Id.* at 15:30-31, 36, 58-59; Ex. 1002 at ¶ 48. Example 3 further supports this construction. Ex. 1002 at ¶ 48. Accordingly, the term "withdrawing" as used in all of the independent claims should be construed as "taking out or removing".

Claim 1 specifies "withdrawing *from a dehydrofluorinated product stream* a product reaction stream comprising spent [DHA]." Similarly, claim 10 recites "withdrawing *from the product stream* a second product stream comprising spent [DHA]." A POSITA would not understand these claims to require spent DHA to be *part of* the gaseous (dehydrofluorinated) product stream from which it is withdrawn because such an understanding would be inconsistent with how a

POSITA would expect materials of different phases (solid, liquid, gas) to interact with one another under the operating conditions described in the '282 patent and would also be inconsistent with the examples in the specification. Ex. 1002 at ¶ 49. As explained above, Example 1 describes organic product (i.e., the dehydrofluorinated product stream) being taken out of the reactor separately from spent KOH (i.e., the product reaction stream/second product stream comprising spent KOH). Ex. 1001 at 15:30-36. Consistent with the description in the specification, a POSITA would understand the "withdrawing" limitations of claims 1 and 10 to encompass "taking out or removing" a stream comprising spent DHA from a reactor separately from a product stream that includes dehydrofluorinated product.

C. "Recovering Spent DHA"

The term "recovering spent DHA" as used in claims 1 and 10 should be construed as "collecting spent DHA for further use (may include separating, purifying, concentrating and/or regenerating)". Ex. 1002 at ¶ 50. The '282 patent describes "the invention" as relating "...to a method of increasing the cost efficiency for dehydrohalogenation production of a fluorinated olefin by recovering and recycling spent reagent" as well as "(d) recovering, purifying and/or regenerating spent [DHA]." Ex. 1001 at 2:49-52, 3:15-17. The

"Dehydrohalogenation" section of the '282 patent includes the following paragraph describing withdrawing and recovering:

The product stream containing spent dehydrohalogenating agent typically carries with it some dissolved organic (e.g. HFC-236ea and/or HFC-245eb). By stopping the stirrer and then removing [i.e., "withdrawing"] spent dehydrohalogenating [from agent the dehydrohalogenation reactor] ..., separation of dehydrohalogenating agent and such organic can be facilitated. Spent dehydrohalogenating agent and dissolved organic would be taken into a container where additional separation of dehydrohalogenating agent and organic can be accomplished using one or more of the foregoing separation techniques [i.e., "recovering"]. ... The organic free KOH isolate can be immediately recycled to the reactor or can be concentrated and the concentrated solution can be returned to reactor.

Id. at 13:29-45 (parentheticals added).

The Examples of the '282 patent also describe using a Scrubber Product Collection Cylinder (SPCC) "...to collect spent KOH and the portion of organic that was carried out with spent KOH." *Id.* at 15:44-47. The term "recovered" is also used in connection with the recovery of HFO-1234yf: "This HFO-1234yf is then separated from the final product stream and recovered as a purified product." *Id.* at 4:52-53.

D. "Recycling"

The '282 patent uses the term "recycling" in claims 15 and 19. The term "recycling" should be construed as "returning a component of the process to the process". Ex. 1002 at ¶ 51. The '282 patent uses the term recycling in a manner consistent with its generally understood meaning: "The organic free KOH isolate can be immediately recycled to the reactor or can be concentrated and the concentrated solution can be returned to reactor." Ex. 1001 at 13:43-45.

E. "Dehydrofluorinated Product Stream" or "Product Stream"

The terms "dehydrofluorinated product stream" (claim 1) and "product stream" (claim 10) mean "a volume of material resulting from dehydrofluorination including dehydrofluorinated organic product(s)." Ex. 1002 at ¶ 52. In Example 1, an exemplary dehydrofluorinated product stream is the organic product (overhead) that was constantly taken out. The specification of the '282 patent explains that "the resulting product stream formed [in the dehydrohalogenation reaction] would be fed into a condenser or distillation column for separation of 1225ye and/or 1234yf from ... un-reacted 236ea and/or 245eb, as well as other by-products of the reaction." Ex. 1001 at 12:66-13:3.

F. "Product Reaction Stream", "Second Product Stream", or "Reaction Stream"

The terms "product reaction stream" (claim 1), "second product stream" (claim 10), and "reaction stream" (claim 16) mean "a volume of material present

Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282 after dehydrofluorination including spent DHA that may also contain by-product salts and/or dissolved organics." Ex. 1002 at ¶¶ 53-54.

Claim 1 refers to the "product reaction stream" as "comprising spent [DHA]" in paragraph d. Claim 10 also refers to the "second product stream" as "comprising spent [DHA]" in paragraph c. Claims 21 and 36 refer to the "reaction stream" as "comprising spent KOH."

The "product reaction stream", "second product stream", and "reaction stream" may also include dissolved organics and by-product salts. Claim 11 recites that the "second product stream" further comprises one or more dissolved organics. Claim 16 states that the reaction stream further comprises by-product salts of the DHA. Claims 33 and 44 recite that the "reaction stream further comprises KF". Moreover, the Examples include separating dissolved organics from spent KOH. Accordingly, the terms "product reaction stream", "second product stream", and "reaction stream" should be construed as "a volume of material including spent DHA that may also contain by-product salts and/or dissolved organics."

G. "Dissolved Organics"

The term "dissolved organics" as used in claims 11 and 12 should be construed to mean "organic that is dissolved in a stream". Ex. 1002 at \P 55. The specification states that "[t]he product stream containing spent [DHA] typically

carries with it some dissolved organic (e.g. HFC-236ea and/or HFC-245eb)". Ex. 1001 at 13:29-31. Claims 11, 28, and 39 specify that the second product stream or reaction stream further comprises dissolved organics. Claims 12, 29, and 40 indicate that the dissolved organics may be 236ea or 245eb. Using the ordinary meaning of the term "dissolved," the term "dissolved organics" means that the dissolved organic is in solution with at least a portion of the stream. Ex. 1002 at ¶ 55.

VII. LEVEL OF ORDINARY SKILL IN THE ART

The level of ordinary skill in the art is evidenced by the references. *See In re GPAC Inc.*, 57 F.3d 1573, 1579 (Fed. Cir. 1995) (the Board did not err in finding the level of skill in the art was best determined by references of record). The level of ordinary skill in the art of the '282 patent is a person having either: (1) a Ph.D. in chemistry, chemical engineering, or a closely related discipline with at least 2 years of practical experience in hydrofluorocarbon synthesis or (2) a B.S. in chemistry, chemical engineering, or a closely related discipline with at least 5 years of practical experience in hydrofluorocarbon synthesis. *See* Ex. 1002 at ¶ 37.

VIII. SUMMARY OF PRIOR ART

A. State of the Art

October 12, 2010 is the relevant date for assessing the state of the art. At the relevant date, it was known in the art of chemical process design that:

There is a rule of thumb in process design that it is desirable to recover more than 99% of all valuable materials. For initial design calculations we use the order of magnitude argument to say that *this rule of thumb is equivalent to requiring that we completely recover and then recycle all valuable reactants*.

Ex. 1016 at p. 116-117 (emphasis added, internal cross-citation omitted).

B. Summary of Primary Prior Art References

1. Smith

Smith was filed on May 15, 2009, designated the United States, and was published under Article 21(2) of the Patent Cooperation Treaty in the English language on November 19, 2009. *Smith* is available as prior art at least under 35 U.S.C. § 102(e) as of May 15, 2009, nearly 17 months prior to the earliest effective filing date of the '282 patent. *Smith* is also available as prior art under 35 U.S.C. § 102(a) as of its publication date on November 19, 2009, more than 10 months prior to the earliest effective filing date of the '282 patent.

Smith discloses a process for preparing fluorinated olefins including hydrogenating HFP to yield 236ea (step (a) of *Smith*) and hydrogenating 1225ye to yield 245eb (step (c) of *Smith*). Ex. 1003 at 1:23-2:2. Hydrogenation of HFP and 1225ye may be conducted simultaneously in the same reactor to produce both 236ea and 245eb. *Id.* at 2:29-3:5. *Smith* teaches that 236ea and 245eb may be separated from each other (e.g., by distillation) before being fed to separate

Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282 dehydrofluorination reactors for carrying out dehydrofluorination steps (b) and (d). *Id. at* 3:5-7.

Smith also discloses dehydrofluorinating 236ea and 245eb using KOH to produce 1225ye and 1234yf, respectively (steps (b) and (d) of *Smith*). *Id.* at 1:23-2:2 and 11:9-12. *Smith* teaches that the dehydrofluorination of 236ea and 245eb may be carried out in separate reactors (as mentioned above) and simultaneously in the same reactor. According to *Smith*, a preferred solvent for the dehydrofluorinating reaction is water. *Id.* at 12:26-27

Smith also discloses that the products from any of steps (a), (b), (c), and/or (d) may be subjected to a purification step, which may be achieved by separation of products or reagents by one or more known separation techniques. *Id.* at 2:11-14. Exemplary separation techniques include distillation, condensation, and phase separation. *Id.*

2. Masayuki

Masayuki was published on April 21, 1984, and is available as prior art under at least 35 U.S.C. § 102(b). *Masayuki* discloses a process for producing chloroprene (2-chloro-butadiene-1,3) by dehydrochlorinating 3,4-dichloro-butene-1 using an alkali metal hydroxide. Ex. 1005 at 4:2-4, 6:14-19. According to *Masayuki*, "the alkali metal hydroxide used for the present invention is typically sodium hydroxide ..., but hydroxides of metals such as potassium, lithium, etc. can

IPR2015-00916

Petition for Inter Partes Review of U.S. Pat. No. 8,710,282

also be used." Id. at 6:37-7:2. In the dehydrochlorination process of Masayuki, 3.4-dichloro-butene-1 is dehydrochlorinated in an aqueous solution containing a relatively high concentration of an alkali metal hydroxide (e.g., NaOH) in the presence of an organic catalyst to produce a gas phase mixture containing chloroprene, unreacted 3,4-dichloro-butene-1, and water vapor and a water phase comprising an aqueous solution of alkali metal hydroxide and alkali metal chloride and solid alkali metal chloride. Id. at 6:14-33. The gas phase mixture and the water phase are removed from the reactor separately. *Id.* at 10:33-34 (water phase) and 11:24-25 (gas phase mixture). Some water is evaporated from the water phase, increasing the concentration of alkali metal hydroxide in the water phase and resulting in additional precipitation of alkali metal chloride. Id. at 6:28-33. The solid alkali metal chloride is separated and removed from the aqueous phase, *id.* at 11:9-14, and the concentrated aqueous alkali metal hydroxide is recycled to the dehydrochlorination reaction. Id. at 11:14-16.

Example 1 and the accompanying Figure (annotated copy provided below) illustrate the process taught in *Masayuki*. In Example 1, a mixture of fresh 3,4-dichloro-butene-1 (conduit 1), recovered 3,4-dichlorobutene-1 (conduit 18), 50% by weight sodium hydroxide aqueous solution (conduit 2), and the catalyst lauryl trimethyl ammonium chloride (conduit 3) are fed into a stirred reactor 19. *Id*. at 10:23-30.



The aqueous phase resulting from the dehydrochlorination reaction is extracted via conduit 4 such that the level of the reactor 19 remains constant. *Id.* at 10:33-35. The aqueous phase leaving the reactor 19 has a lipid phase present therein, which is separated from the aqueous phase using the decanter 20 and is returned to the reactor 19 via conduit 5. *Id.* at 10:35-11:1. The aqueous phase,

which contains unreacted NaOH and NaCl in solution and also precipitated sodium chloride, is fed to a loop comprising a circulation-type enricher 21 via conduit 6, where concentration of the aqueous phase is carried out through evaporation. Id. at 11:1-5. In the loop, the aqueous phase passes through conduit 8 and 9, is heated in heat exchanger 22 and is circulated back to the enricher 21 via conduit 10. Id. at 11:6-9. As water evaporates, dissolved sodium chloride precipitates out of the aqueous phase. Id. at 11:9-10. The aqueous phase containing concentrated NaOH solution and precipitated NaCl is extracted from the loop via conduit 8, directed into a slurry tank 24 via conduit 11, and fed into a centrifuge 25 where the precipitated NaCl is separated out and discarded via conduit 12. Id. at 11:10-14. The aqueous phase containing concentrated, recovered sodium hydroxide is returned to conduit 2 via conduit 13 and recycled to the dehydrochlorination reaction (reactor 19). Id. at 11:14-16.

Petition for Inter Partes Review of U.S. Pat. No. 8.710.282

3. Harrison

Harrison was published on November 8, 1983, and is available as prior art under at least 35 U.S.C. § 102(b). *Harrison* describes a process for converting alkali metal salt to alkali metal hydroxide using lime (Ca(OH)₂). Initially, industrial waste waters containing fluoride are contacted with aqueous potassium hydroxide to obtain an aqueous solution of potassium fluoride and potassium hydroxide (HF + excess KOH \rightarrow KOH + KF + H₂O). Then, the solution of the

initial step is contacted with finely divided high purity lime $(Ca(OH)_2)$ in a conversion step to obtain a calcium fluoride precipitate in aqueous potassium hydroxide $(KF + KOH + Ca(OH)_2 \rightarrow CaF_2 + KOH)$. Next, the reaction product of the conversion step is settled into two phases comprising an aqueous potassium hydroxide supernatant and an aqueous slurry of calcium fluoride. The aqueous potassium hydroxide supernatant is recovered. Ex.1006 2:10-21.

IX. THERE IS A REASONABLE LIKELIHOOD THAT THE CHALLENGED CLAIMS OF THE '282 PATENT ARE UNPATENTABLE

Pursuant to 37 C.F.R. § 42.104(b)(4)-(5), all of the challenged claims are unpatentable for the reasons set forth in detail below.

A. Claims 1-20 are Obvious over *Smith* in View of *Masayuki* and *Harrison*

1. Claim 1

Claim 1 of the '282 patent is directed to: (a) hydrogenating a starting material including alkene(s) with a reducing agent to produce an intermediate product stream, (b) optionally separating the intermediate product stream into a plurality of intermediate product streams, (c) dehydrofluorinating, in the presence of a DHA, at least a portion of the intermediate process stream(s) to produce 1225ye and at least one additional alkene having a lower degree of fluorination than the alkene (e.g., 1234yf), (d) withdrawing from a dehydrofluorinated product stream a product reaction stream comprising spent DHA, and (e) recovering spent

DHA. As explained below, *Smith* discloses steps (a)-(c), *Masayuki* teaches step (d), and the combination of *Masayuki* and *Harrison* teaches step (e).

a. Overview of Combination

Smith discloses the chemistry recited in claim 1 (i.e., hydrogenating a haloalkene by contacting it with a reducing agent to produce a haloalkane and dehydrohalogenating the haloalkane in the presence of KOH to produce a haloalkene). To achieve a more efficient and cost effective process than the one disclosed in Smith, a POSITA would look to analogous, base-mediated dehydrohalogenation processes for suggestions of how to recover and/or recycle unreacted KOH and by-product salt of the KOH (e.g., KF) formed in the reaction. Ex. 1002 at ¶ 102. Masayuki teaches recovering and recycling unreacted alkali metal hydroxide in one such analogous, base-mediated dehydrohalogenation Masayuki also provides a flow diagram of a base-mediated process. dehydrohalogenation process, which can be used as a guide in implementing the recovery and recycling steps in Masayuki. A POSITA would be motivated to apply the steps of *Masayuki* to the dehydrohalogenation process described in *Smith* to recover and recycle the unreacted alkali metal hydroxide (KOH) that remains after the dehydrohalogenation reaction and would have a reasonable expectation of success in doing so. *Id.* at \P 102.

To make the dehydrohalogenation process of *Smith* even more efficient and cost effective, a POSITA would be motivated to increase the amount of DHA recovered and recycled to the dehydrohalogenation reaction and to make productive use of the by-product salt. *Id.* at ¶ 102. *Harrison* teaches a method for converting by-product salt (KF) back to DHA (KOH), which would enable such increase in recovery and recycle of DHA. *Id.* at ¶ 102.

b. Detailed Analysis of Claim 1

Turning to the elements of claim 1, as described in more detail below, *Smith* discloses steps (a)-(c), *Masayuki* teaches step (d), and the combination of *Masayuki* and *Harrison* teaches step (e).

Smith discloses hydrogenating a starting material including alkene(s) with a reducing agent to produce an intermediate product stream (step (a) of claim 1). Specifically, *Smith* discloses a process for preparing fluorinated olefins that includes hydrogenating HFP with hydrogen (a reducing agent) to provide 236ea (step (a) of *Smith*). Ex. 1003 at 1:23-2:2. *Smith* further describes hydrogenating 1225ye with hydrogen to yield 245eb (step (c) of *Smith*). *Id*. Both HFP and 1225ye meet the requirements of Formula I of claim 1 of the '282 patent (For HFP, X is F, n is 3, m is 0, and z is 0, and the values of Y, R^1 , R^2 , a, and b need not be defined due to the values of n and z; for 1225ye, X is F, n is 3, m is 1, z is 0, and the values of Y, R^1 , R^2 , a, and b need not be defined due to the values of n and z).

Ex. 1002 at ¶ 103. Likewise, both 236ea and 245eb meet the requirements of Formula II of claim 1 of the '282 patent (For 236ea, X is F, n is 3, m is 0, and z is 0; for 245eb, X is F, n is 3, m is 1, and z is 0.). Ex. 1002 at ¶ 103. Therefore, *Smith* discloses step (a) of claim 1 of the '282 patent.

Petition for Inter Partes Review of U.S. Pat. No. 8.710,282

Although step (b) is not required by claim 1 of the '282 patent because it is an optional step, *Smith* further discloses optionally separating the intermediate stream into a plurality of intermediate product streams (step (b) of claim 1). *Smith* discloses that hydrogenation of HFP and 1225ye may be conducted simultaneously in the same reactor to produce both 236ea and 245eb. Ex.1003 at 2:29-3:5. *Smith* further discloses optionally separating 236ea and 245eb after hydrogenation and before dehydrofluorination. For example, 236ea and 245eb may be separated from each other (e.g., by distillation) before being fed into separate dehydrofluorination reactors for carrying out steps (b) and (d) of *Smith*. *Id*. at 3:5-7. *Smith* further discloses an alkane recycle stream wherein a diluent gas stream "... can be a gas such as ... one or both of the products from steps (a) and (c), 245eb or 236ea." Ex. 1003 at 3:15-20. Therefore, *Smith* also discloses step (b) of the '282 patent.

Smith further discloses dehydrofluorinating, in the presence of a DHA, at least a portion of the intermediate process stream(s) to produce 1225ye and at least one additional alkene having a lower degree of fluorination than the compound of Formula I (e.g., 1234yf) (step (c) of claim 1). *Smith* discloses dehydrofluorinating

236ea and 245eb with a DHA (e.g., a base) to provide 1225ye and 1234yf, respectively (steps (b) and (d) of *Smith*). Ex.1003 at 1:23-2:2, 11:9-10. Dehydrofluorination of 236ea and 245eb may be carried out in separate reactors or simultaneously in the same reactor. For example, separate feeds may be fed to a single reactor in which steps (b) and (d) of *Smith* are carried out simultaneously. *Id.* at 3:4-14. Accordingly, *Smith* discloses steps (a)-(c) of claim 1.

Petition for Inter Partes Review of U.S. Pat. No. 8.710.282

Masayuki teaches withdrawing from a dehydrohalogenated product stream a product reaction stream comprising spent DHA. As explained above, the withdrawing limitation of claim 1 should be construed to encompass "taking out or removing" from a reactor a product stream including dehydrofluorinated product and a separate stream including spent DHA. Masayuki discloses a base-mediated dehydrohalogenation process, in particular, dehydrochlorinating 3,4-dichlorobutene-l using an aqueous solution of alkali metal hydroxide (e.g., NaOH or KOH) to produce a gas phase mixture comprising chloroprene and a water phase comprising an aqueous solution of alkali metal hydroxide and alkali metal halide (e.g., NaCl or KCl) and solid alkali metal halide. Ex. 1005 at 6:14-33, 6:37-7:2. In an example of the process described in *Masayuki*, the gas phase mixture and the water phase are removed from the reactor separately. *Id.* at 10:33-34 (water phase) and 6:19-23, 11:24-25 (gas phase mixture). The aqueous phase containing unreacted alkali metal hydroxide (NaOH) and by-product salt (NaCl) is withdrawn from the reactor via conduit 4 for additional processing. Ex.1005 at 10:33-35. Thus, *Masayuki* teaches withdrawing an aqueous phase comprising spent DHA and alkali metal halide salt and a separate stream comprising dehydrohalogenated product (i.e., chloroprene) from the dehydrohalogenation reactor. Applying the withdrawing step of *Masayuki* to the dehydrohalogenation process of *Smith* results in withdrawing an aqueous phase (i.e., a "product reaction stream") comprising spent DHA (and by-product KF) from the dehydrofluorination reactor, as recited in step (d) of claim 1. Ex. 1002 at ¶ 106.

Petition for Inter Partes Review of U.S. Pat. No. 8.710.282

Masayuki in combination with *Harrison* teaches step (e) of claim 1, namely, recovering spent DHA. *Masayuki* teaches that the aqueous phase that is withdrawn from the reactor 19 via conduit 4, as described above, is introduced to a separator (specifically a decanter 20) wherein an organic lipid phase is separated and removed via conduit 5 from the aqueous phase. Ex.1005 at 10:33-11:1. The aqueous phase is introduced to a loop including a circulation-type enricher 21 and a heat exchanger 22. *See* Ex. 1005 at 11:1-9, Figure. The aqueous phase is concentrated in the loop, leading to precipitation of alkali metal chloride and concentration of alkali metal hydroxide in the aqueous phase. Ex. 1005 at 11:9-14; Ex. 1002 at ¶ 107. The aqueous phase is then transported via conduit 11 to the slurry tank 24 and then to the centrifuge 25, where alkali metal chloride precipitate is separated from the aqueous phase, leading to the recovery of an aqueous phase

comprising a concentrated solution of alkali metal hydroxide. *Id.* at 11:9-14. As explained above, when the withdrawing step of *Masayuki* is applied to the dehydrofluorination process of *Smith*, an aqueous reaction stream comprising spent KOH (and by-product KF) is withdrawn from the dehydrofluorination reactor of *Smith*. Applying the decanting step of *Masayuki*, as described above, to this withdrawn reaction stream results in the recovery of spent DHA as an aqueous mixture of DHA (KOH) and by-product salt (KF) (step (e) of claim 1). Ex. 1002 at ¶ 107.

Petition for Inter Partes Review of U.S. Pat. No. 8.710.282

Harrison teaches that KF in an aqueous mixture of KOH and KF can be converted to KOH by treating the mixture with Ca(OH)₂, slaked lime. Ex. 1006 at 2:14-16, 3:6-10. Applying the conversion step of *Harrison* to the decanted aqueous mixture of DHA (KOH) and by-product salt (KF) from the process of *Smith* in combination with *Masayuki* results in recovery of additional aqueous DHA (KOH) and makes productive use of the by-product salt (KF) (i.e., by converting the KF to KOH and CaF₂). Ex. 1002 at ¶ 107. A POSITA would recognize that the conversion step of *Harrison* could be implemented in the slurry tank 24 of *Masayuki* simply by introducing the Ca(OH)₂ of *Harrison* to the slurry tank 24. Ex. 1002 at ¶ 107. The other processing steps in *Masayuki* would remain the same.

The conversion step of *Harrison* improves the recovering and recycling process of *Masayuki* because it enables recovery and recycling of more DHA (KOH) and makes productive use of the by-product salt (KF). Ex. 1002 at ¶ 107. In addition, the aqueous phase comprising the recovered DHA (KOH) has a higher purity due to the conversion of the by-product salt (KF) back to DHA (KOH). A POSITA would have been motivated to apply the method of *Harrison* to the dehydrofluorination process of *Smith*, as modified by *Masayuki*, in order to increase the recovery of spent DHA (KOH) and to utilize the by-product salt (KF). Ex. 1002 at ¶ 107.

A POSITA would be motivated to combine the teachings of *Smith*, *Masayuki*, and *Harrison* to arrive at the invention of claim 1. *See* Ex. 1002 at ¶ 102. *Smith* discloses hydrogenating a haloalkene by contacting it with a reducing agent to produce a haloalkane and dehydrohalogenating the haloalkane in the presence of KOH to produce a haloalkene, which is the chemistry recited in claim 1. As mentioned above, it is a "rule of thumb" in chemical process design to try to recover and re-use as much valuable material as possible in a process. Further, operating as efficiently and economically as possible is always a goal in chemical processing. Thus, as with any process, a POSITA would be motivated to try to recover and recycle as much valuable material (e.g., dehydrohalogenating agent) as possible from the reactions described in *Smith*. To do so, the skilled artisan would
IPR2015-00916

Petition for Inter Partes Review of U.S. Pat. No. 8,710,282 look to analogous, base-mediated dehydrohalogenation processes for suggestions of how to recover and/or recycle unreacted KOH and by-product salt of the KOH (e.g., KF) formed in the reaction. The search for analogous processes would lead to Masayuki, which teaches a method for recovering and recycling unreacted alkali metal hydroxide in an analogous, base-mediated dehydrohalogenation process. Masayuki also provides a flow diagram of a base-mediated dehydrohalogenation process, which a POSITA could use as a guide in implementing the recovery and recycling steps in Masayuki. A POSITA would thus be motivated to apply the steps of Masayuki to the dehydrohalogenation process described in Smith to recover and recycle the unreacted, spent alkali metal hydroxide (KOH) that remains after the dehydrohalogenation reaction and would have a reasonable expectation of success in doing so. To make the dehydrohalogenation process of Smith even more efficient and cost effective, a POSITA would be motivated to increase the amount of KOH recovered and recycled to the dehydrohalogenation reaction and to make productive use of the KF by-product salt. To that end, Harrison teaches a method for converting by-product salt KF back to KOH using Ca(OH)₂, which would enable an increase in recovery and recycle of KOH to the dehydrohalogenation process in *Smith*, and sale of CaF₂ to increase cost efficiency. *See* Ex. 1002 at ¶ 102.

Petition for Inter Partes Review of U.S. Pat. No. 8,710,282

A POSITA would have a reasonable expectation of success in combining the teachings of *Smith* and *Masayuki* because these references describe very similar processes, namely the aqueous base-mediated dehydrohalogenation of a halogenated organic compound, which produce organic products and an aqueous phase comprising unreacted (spent) DHA and a by-product salt of the DHA. Ex. 1002 at ¶¶ 102, 107. A POSITA would further have a reasonable expectation of success in combining *Harrison* with *Smith* and *Masayuki* because all of the references describe processes involving an aqueous phase comprising a DHA and a by-product salt of the DHA. Ex. 1002 at ¶¶ 102, 107. A POSITA would further have a reasonable expectation of success in combining *Harrison* with *Smith* and *Masayuki* because all of the references describe processes involving an aqueous phase comprising a DHA and a by-product salt of the DHA. Ex. 1002 at ¶¶ 102, 107. Additionally, a POSITA would consider applying the converting step of *Harrison* to *Masayuki* to be straightforward, causing little to no disruption to the process of *Masayuki*. Ex. 1002 at ¶¶ 102, 107.

Because the combined disclosure of *Smith*, *Masayuki*, and *Harrison* teaches each limitation of claim 1, the claim is invalid as obvious under 35 U.S.C. § 103, as shown in the following chart. *See* Ex. 1002 at ¶¶ 102-108.

'282 patent claim	Disclosure from Smith (Ex. 1003)
1. A method for producing at	"The subject invention addresses the
least one fluorinated olefin	deficiencies of the known methods for
comprising:	preparing 1234yf by providing a process for the
a. hydrogenating a starting	preparation of 1234yf comprising (a)
material stream comprising at	contacting 1,1,2,3,3,3-hexafluoropropene
least one alkene according to	(referred to hereinafter as 1216 or HFP) with
Formula (I):	hydrogen in the presence of a hydrogenation
(CX_nY_{3-})	catalyst to produce 1,1,2,3,3,3-
$(CX_{n}^{1})_{3}^{3}$ n) $(CR_{a}^{1}R_{b}^{2})_{z}CX = CH_{m}X_{2-m}(I)$	hexafluoropropane (referred to hereinafter as
	236ea); (c) contacting 1225ye with

by contacting said starting material with a reducing agent to produce an intermediate product stream comprising at least one alkane according to Formula (II): $(CX_nY_{3-})_n (CR_a^1R_b^2)_z CHXCH_{m+1}X_{2-m}$ (II) where:	hydrogen in the presence of a hydrogenation catalyst to produce 1,2,3,3,3- pentafluoropropane (referred to hereinafter as 245eb)." (Ex. 1003 at 1:23-2:2 .)
each X is independently Cl, F, I or Br, provided that at least two Xs are F;	
each Y is independently H, Cl, F, I or Br;	
each R ¹ is independently H, Cl, F, I, Br or unsubstituted or halogen substituted methyl or ethyl radical;	
each R ₂ is independently H, Cl, F, I, Br or unsubstituted or halogen substituted methyl or ethyl radical;	
n is 1, 2 or 3;	
a and b are each 0, 1 or 2, provided that a+b=2;	
m is 0, 1 or 2; and	
z is 0, 1, 2 or 3;	
b. optionally, separating said intermediate product stream into a plurality of intermediate product streams, said plurality of intermediate product streams comprising two or more streams selected from the group consisting of a first stream rich in at least a first alkane according to Formula II, a second stream rich in at least a second alkane according to Formula II, and an alkane recycle stream;	"In a further reaction topology, steps (a) and (c) of the process of the invention may be carried out simultaneously in the same reactor The product of the reaction wherein steps (a) and (c) are conducted simultaneously in the same reactor comprises 236ea and 245eb." (Ex. 1003 at 2:29-3:5.) "These may be separated from each other (e.g. by distillation) before being fed into separate dehydrofluorination reactors for carrying out steps (b) and (d)." (Ex. 1003 at 3:5-7.) "[T]he exothermic hydrogenation reactions of step (a) and/or (c) may be controlled by the use of a diluent gas stream The diluent gas stream can be a gas such as one or both of

	the products from steps (a) and (c), 245eb or 236ea." (Ex. 1003 at 3:15-20.)
c. dehydrofluorinating, in the presence of a dehydrohalogenating agent, at least a portion of said intermediate process stream from step (a) or said plurality of intermediate process streams of step (b) to produce an alkene product stream comprising 1,1,1,2,3-pentafluoropropene [1,2,3,3,3-pentafluoropropene (1225ye)] and at least one additional alkene having a lower degree of fluorine substitution compared to the degree of fluorination of the compound of formula (I); and	"The subject invention addresses the deficiencies of the known methods for preparing 1234yf by providing a process for the preparation of 1234yf comprising(b) dehydrofluorinating 236ea to produce 1,2,3,3,3-pentafluoropropene (referred to hereinafter as 1225ye); and (d) dehydrofluorinating 245eb to produce 1234yf." (Ex. 1003 at 1:23-2:2 .)
	"Another preferred method of effecting the dehydrofluorination is by contacting 236ea and/or 245eb with a base." (Ex. 1003 at 11:9-10.)
	"The product of the reaction wherein steps (a) and (c) are conducted simultaneously in the same reactor comprises both 236ea and 245eb. These may be separated from each other (e.g. by distillation) before being fed into separate dehydrofluorination reactors for carrying out steps (b) and (d). Alternatively, a combined stream of 236ea and 245eb may be fed to a single reactor. Thus, dehydrofluorination steps (b) and (d) may be carried out simultaneously in the same reactor Of course, separate feeds of 236ea and 245eb originating from separate hydrogenation reactors may be fed to a single dehydrofluorination reactor in which steps (b) and (d) are carried out simultaneously." (Ex. 1003 at 3:4-14.)
Claim 1 (cont.)	Disclosure from <i>Masayuki</i> (Ex. 1005)
d. withdrawing from a dehydrofluorinated product stream a product reaction stream comprising spent dehydrohalogenating agent; and	 (Ex. 1005 Figure, stream 4.) "The aqueous phase was extracted via Conduit 4 such that the level of Reactor 19 remained constant" (Ex. 1005 at 10:33-35.) "[A] mixture containing chloroprene, unreacted 3,4-dichloro-butene-1 and water produced by performing a reaction at a temperature of 20 – 100°C is removed in a gas phase via evaporation without reflux of said mixture into the reaction system." (Ex. 1005 at 6:19-23.)

IPR2015-00916

Petition for Inter Partes Review of U.S. Pat. No. 8,710,282

Claim 1 (cont.)	Disclosure from <i>Masayuki</i> (Ex. 1005)	Disclosure from <i>Harrison</i> (Ex. 1006)
e. recovering spent dehydrohalogenatin g agent.	(Ex. 1005 Figure, decanter 20, enricher 21, centrifuge 25.) "The aqueous phase was extracted via Conduit 4 such that the level of Reactor 19 remained constant and the lightly mixed lipid and aqueous phases (including precipitated sodium chloride) were separated using Decanter 20, after which the lipid phase was returned to the reactor via Conduit 5." (Ex. 1005 at 10:33-11:1.) "The aqueous phase was fed into a forced circulation- type Enricher 21 via Conduit 6 and concentration was carried out After passing through Conduit 9 from Conduit 8, the aqueous phase was heated in Heat Exchanger 22 after which it was circulated in Enricher 21 via Conduit 10." (Ex. 1005 at 11:1-9.) "As concentration was performed, dissolved sodium chloride precipitated out, but said aqueous phase was extracted via Conduit 8, directed into Slurry Tank 24 via Conduit 11 and fed into Centrifuge 25 where precipitated sodium chloride was separated out and also discarded via Conduit 12." (Ex.1005 at 11:9-14.)	"(b) contacting [an aqueous solution of potassium fluoride and potassium hydroxide] with finely divided high purity lime to thereby obtain a calcium fluoride precipitate in aqueous potassium hydroxide (c) settling the reaction product of step (b) into two phases comprising an aqueous potassium hydroxide supernatant and an aqueous slurry of calcium fluoride; (d) recovering the aqueous potassium hydroxide supernatant" (Ex.1006 at 2:14-21.) "The basic potassium fluoride solution is then mixed with solid hydrated (slaked) lime in a stirred vessel where this calcium hydroxide [Ca(OH) ₂] reacts to form the calcium fluoride precipitate and regenerate the potassium hydroxide." (Ex. 1006 at 3:6-10.)

2. Claim 2-6

Claim 2 depends from claim 1 and further defines "the additional alkene" produced in (c) of Formula step as a compound (III), (CX_nY_{3-}) $_{n}$)(CR¹_aR²_b)_zCX=CH_{m+1}X_{1-m}, where each of X, Y, R¹, R², n, a, and b is the same value as in formula (I) and m is 0 or 1. Claims 3-6 further define Formula (III), specifying that Z is 0 (claim 3), m is 0 or 1 (claim 4), n is 3 (claim 5), and X is F (claim 6).

As discussed above in connection with claim 1, *Smith* teaches the hydrogenation of HFP and 1225ye to produce 236ea and 245eb, respectively (steps (a) and (c) of *Smith*), and the dehydrofluorination of 236ea and 245eb to produce 1225ye and 1234yf, respectively (steps (b) and (d) of *Smith*). Ex.1003 at 1:23-2:2. Formula (I) reads on HFP and 1225ye when X is F, n is 3, and z is 0. The values of Y, R¹, R², a, and b need not be defined due to the values of n and z. Ex. 1002 at ¶ 109. Thus, when the alkene according to formula (I) is understood to be HFP or 1225ye, as disclosed in *Smith*, the values of the variables of Formula (III), $(CX_nY_{3-n})(CR^{1}{}_{a}R^{2}{}_{b})_{z}CX=CH_{m+1}X_{1-m}$, are as follows: X is F, n is 3, m is 0 or 1, and Y, R¹, R², a, b, and z can take any value permitted by formula (I) (because these variables are undefined in formula (I) when the alkene is HFP or 1225ye). Ex. 1002 at ¶ 109.

IPR2015-00916

Petition for Inter Partes Review of U.S. Pat. No. 8,710,282

Formula (III) reads on 1234yf (CF₃-CF=CH₂), when X is F, n is 3, z is 0,

and m is 1. Ex. 1002 at ¶ 109. As such, 1234yf meets the requirements of formula

(III), as defined in claims 2-6. Because the combined disclosure of Smith,

Masayuki, and Harrison teaches each limitation in claims 2-6, claims 2-6 are

invalid under 35 U.S.C. § 103, as shown in the following chart. See Ex. 1002 at ¶

109.

²⁸² patent claim	Disclosure from <i>Smith</i> (Ex. 1003)
 2. The method of claim 1 wherein said additional alkene is a compound according to formula (III): (CX_nY_{3-n}) (CR¹_aR²_b)_zCX=CH_{m+1}X_{1-m} (III) where each of X, Y, R¹, R², n, a, and b is the same value as in formula (I) and m is 0 or 1. 3. The method of claim 2 wherein Z is 0. 4. The method of claim 3 wherein m is 0 or 1 5. The method of claim 4 wherein said n is 3. 6. The method of claim 5 wherein X is F. 	"The subject invention addresses the deficiencies of the known methods for preparing 1234yf by providing a process for the preparation of 1234yf comprising (d) dehydrofluorinating 245eb to produce 1234yf." (Ex. 1003 at 1:23-2:2.)

3. Claim 7

Claim 7 depends from claim 1 and further requires that the reducing agent is H_2 . *Smith* discloses using hydrogen as the reducing agent in the hydrogenation of HFP to produce 236ea and the hydrogenation of 1225ye to produce 245eb. Ex. 1003 at 1:23-2:2. Because the combined disclosure of *Smith*, *Masayuki*, and *Harrison* teaches each limitation in claim 7, the claim is invalid under 35 U.S.C. § 103, as shown in the following chart. *See* Ex. 1002 at ¶ 110.

²⁸² patent claim	Disclosure from <i>Smith</i> (Ex. 1003)
7. The method of claim 1 wherein said reducing agent is H ₂ .	"The subject invention addresses the deficiencies of the known methods for preparing 1234yf by providing a process for the preparation of 1234yf comprising (a) contacting 1,1,2,3,3,3- hexafluoropropene (referred to hereinafter as 1216 or HFP) with hydrogen in the presence of a hydrogenation catalyst to produce 1,1,2,3,3,3- hexafluoropropane (referred to hereinafter as 236ea) (c) contacting 1225ye with hydrogen in the presence of a hydrogenation catalyst to produce 1,2,3,3,3- pentafluoropropane (referred to hereinafter as 245eb)." (Ex. 1003 at 1:23-2:2 .)

Claim 8 depends from claim 1 and further requires that the starting material stream comprises hexafluoropropylene [HFP] and that said additional alkene is 2,3,3,3-tetrafluoropropene [1234yf]. *Smith* discloses a process for producing 1234yf that begins with hydrogenation of a 1,1,2,3,3,3-hexafluoropropene [HFP] starting material. Ex. 1003 at 1:23-2:2. A POSITA would understand that 1,1,2,3,3,3-hexafluoropropene (HFP) is a synonym for hexafluoropropylene. Ex. 1002 at ¶ 111. Because the combined disclosure of *Smith*, *Masayuki*, and *Harrison* teaches each limitation in claim 8, the claim is invalid under 35 U.S.C. § 103, as shown in the following chart. *See* Ex. 1002 at ¶ 111.

'282 patent claim	Disclosure from <i>Smith</i> (Ex. 1003)
8. The method of claim 1 wherein said starting material stream comprises hexafluoropropylene and said additional alkene is	"The subject invention addresses the deficiencies of the known methods for preparing 1234yf by providing a process for the preparation of 1234yf comprising (a) contacting 1,1,2,3,3,3- hexafluoropropene (HFP) with hydrogen in the presence of a hydrogenation catalyst to produce
2,3,3,3-	1,1,2,3,3,3-hexafluoropropane (236ea); and (d)

tetrafluoropropene	dehydrofluorinating 245eb to produce 1234yf." (Ex.
[1234yf].	1003 at 1:23-2:2.)

Claim 9 depends from claim 1 and further requires that the DHA is selected from the group consisting of KOH, NaOH, Ca(OH)₂, LiOH, Mg(OH)₂, CaO, and combinations thereof. *Smith* discloses using KOH as a DHA. Ex. 1003 at 11: 9-15. Because the combined disclosure of *Smith*, *Masayuki*, and *Harrison* teaches each limitation in claim 9, the claim is invalid under 35 U.S.C. § 103, as shown in the following chart. *See* Ex. 1002 at ¶ 112.

'282 patent claim	Disclosure from Smith (Ex. 1003)
9. The method of	"Another preferred method of effecting the
claim 1 wherein the	dehydrofluorination in steps (b) and (d) is by contacting
dehydrohalogenating	236ea and/or 245eb with a base. Preferably, the base is a
agent is selected from	metal hydroxide, e.g. an alkali or alkaline earth metal
the group consisting	hydroxide[. T]he term "alkali metal hydroxide" [refers]
of KOH, NaOH,	to a compound or mixture of compounds selected from
Ca(OH) ₂ , LiOH,	lithium hydroxide, sodium hydroxide, potassium
Mg(OH) ₂ , CaO, and	hydroxide, rubidium hydroxide and caesium
combinations thereof.	hydroxide." (Ex. 1003 at 11: 9-15 .)

6. Claim 10

Independent claim 10 is directed to a method of producing 1234yf comprising (a) hydrogenating HFP by contacting it with a reducing agent in a hydrogenation reactor to produce 245eb or 236ea, (b) dehydrofluorinating, in the presence of a DHA, 245eb or 236ea in a dehydrofluorination reactor to produce a product stream comprising 1234yf or 1225ye, (c) withdrawing a second product stream including spent DHA from the product stream, and (d) recovering spent

DHA. As explained below, *Smith* discloses steps (a) and (b), *Masayuki* teaches step (c), and the combination of *Masayuki* and *Harrison* teaches step (d).

Petition for Inter Partes Review of U.S. Pat. No. 8.710,282

As explained above for claim 1, it would have been obvious to modify the dehydrohalogenation process of *Smith* to recover and recycle DHA using the "withdrawing" step and the "recovering" step as taught in the combination of *Masayuki* and *Harrison*. A POSITA would have been motivated to apply the combined teaching of *Masayuki* and *Harrison* to the dehydrohalogenation process of *Smith* to maximize the amount of DHA recovered and recycled to the dehydrohalogenation process, and to make productive use of the by-product(s) formed in the reaction, thereby reducing operating costs and increasing process efficiency and would have had a reasonable expectation of success in making such a combination. *See* Ex. 1002 at ¶ 114.

Smith discloses steps (a) and (b) of claim 10 of the '282 patent. *Smith* discloses hydrogenating a starting material stream comprising HFP by contacting the starting material stream with a reducing agent in a hydrogenation reactor to produce an intermediate stream comprising 245eb or 236ea (step (a) of claim 10). Specifically, *Smith* discloses a process for preparing fluorinated olefins including hydrogenating HFP with hydrogen (a reducing agent) to provide 236ea. Ex. 1003 at 1:23-2:2. *Smith* further describes hydrogenating 1225ye with hydrogen to yield 245eb. *Id.* Hydrogenation of HFP and 1225ye may be carried out separately or

Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282 simultaneously in the same reactor to produce both 236ea and 245eb simultaneously. *Id.* at 2:16-17, 2:29-3:5.

Smith further discloses dehydrofluorinating, in the presence of a DHA, 245eb or 236ea in a dehydrofluorination reactor to produce a product stream comprising 1234yf or 1225ye (step (b) of claim 10). *Smith* discloses dehydrofluorinating 236ea and 245eb with a DHA (e.g., a base) to produce 1225ye and 1234yf (steps b and d of *Smith*). Ex. 1003 at 1:23-2:2, 11:9-10. Dehydrofluorination of 236ea and 245eb may be carried out in separate reactors or simultaneously, in the same reactor. For example, separate feeds may be fed to a single reactor in which steps (b) and (d) are carried out simultaneously. *Id.* at 3:4-14. Accordingly, *Smith* discloses steps (a)-(b) of claim 10 of the '282 patent.

Masayuki teaches the withdrawing step of claim 10 (step (c)). For all of the same reasons discussed above with regard to claim 1, applying the withdrawing step of *Masayuki* to the dehydrofluorination process of *Smith* results in withdrawing from a dehydrofluorinated product stream a product reaction stream comprising spent DHA, as recited in step (c) of claim 10. Ex. 1002 at ¶ 117.

The combination of *Masayuki* and *Harrison* teaches the recovering step of claim 10. The recovering step of claim 10 is identical to the recovering step of claim 1. Thus, the combination of *Masayuki* and *Harrison* teaches the recovering

43

Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282

step of claim 10 for all of the same reasons discussed with regard to claim 1 above.

Ex. 1002 at ¶ 118.

It would have been obvious to a POSITA to combine the withdrawing and recovering steps taught in *Masayuki* and *Harrison* with the hydrogenating and dehydrohalogenating steps of *Smith*, set forth in claim 10 for all of the same reasons as set forth above with regard to claim 1. Because the combined disclosure of *Smith*, *Masayuki*, and *Harrison* teaches each limitation in claim 10, the claim is invalid under 35 U.S.C. § 103, as shown in the following chart. *See* Ex. 1002 at ¶¶

113-119.

'282 patent claim	Disclosure from <i>Smith</i> (Ex. 1003)
10. A method for producing 2,3,3,3-tetrafluoropropene [1234yf] comprising: a. hydrogenating a starting material stream comprising hexafluoropropylene [1,1,2,3,3,3- hexafluoropropene (HFP)] by contacting said starting material stream with a reducing agent in a hydrogenation reactor to produce an intermediate	"The subject invention addresses the deficiencies of the known methods for preparing 1234yf by providing a process for the preparation of 1234yf comprising (a) contacting 1,1,2,3,3,3- hexafluoropropene (referred to hereinafter as 1216 or HFP) with hydrogen in the presence of a hydrogenation catalyst to produce 1,1,2,3,3,3- hexafluoropropane (referred to hereinafter as 236ea); (c) contacting 1225ye with hydrogen in the presence of a hydrogenation catalyst to produce 1,2,3,3,3-pentafluoropropane (referred to hereinafter as 245eb)." (Ex. 1003 at 1:23-2:2.)
stream comprising	"[T]he process may be carried out continuously
1,1,1,2,3-	with steps (a), (b), (c), and (d) being conducted
pentafluoropropane	sequentially in that order using a separate reactor
[1,2,3,3,3-	for each step." (Ex. 1003 at 2:16-17.)
pentafluoropropane	"In a further reaction topology, steps (a) and (c) of
(245eb)] or $1,1,1,2,3,3$ -	the process of the invention may be carried out
hexafluoropropane	simultaneously in the same reactor The
[$1,1,2,3,3,3$ -	product of the reaction wherein steps (a) and (c)
hexafluoropropane	are conducted simultaneously in the same reactor
(236ea)];	comprises both 236ea and 245eb." (Ex. 1003 at

		2:29-3:5.)	
b. dehydrofluorinating, the presence of a dehydrohalogenating a said 1,1,1,2,3- pentafluoropropane [1,2,3,3,3- pentafluoropropane (245eb)] or said 1,1,1,2 hexafluoropropane [23 in a dehydrofluorinatio reactor to produce a pro- stream comprising 2,3, tetrafluoropropene [123 or 1,2,3,3,3- pentafluoropropene [1225ye];	gent, 2,3,3- 6ea] on oduct 3,3-	"The subject invention addresses the deficiencies of the known methods for preparing 1234yf by providing a process for the preparation of 1234yf comprising (b) dehydrofluorinating 236ea to produce 1,2,3,3,3-pentafluoropropene (referred to hereinafter as 1225ye); and (d) dehydrofluorinating 245eb to produce 1234yf. (Ex.1003 at 1:23-2:2 .) "The product of the reaction wherein steps (a) and (c) are conducted simultaneously in the same reactor comprise both 236ea and 245eb. These may be separated from each other (e.g. by distillation) before being fed into separate dehydrofluorination reactors for carrying out steps (b) and (d). Alternatively, a combined stream of 236ea and 245eb may be fed to a single reactor. Thus, dehydrofluorination steps (b) and (d) may be carried out simultaneously in the same reactor Of course, separate feeds of 236ea and 245eb originating from separate hydrogenation reactors may be fed to a single dehydrofluorination reactor in which steps (b) and (d) are carried out simultaneously." (Ex. 1003 at 3:4-14 .) "Another preferred method of effecting the	
		dehydrofluorination in steps (b) and (d) is by contacting 236ea and/or 245eb with a base." (Ex. 1003 at 11:9-10.)	
Claim 10 (cont.)		Disclosure from Masay	uki (Ex. 1005)
c. withdrawing from th	e	(Ex. 1005 Figure, stream 4.)	
product stream a second product stream comprising spent dehydrohalogenating agent; and		"The aqueous phase was extracted via Conduit 4 such that the level of Reactor 19 remained constant." (Ex.1005 at 10:33-35)	
		"[A] mixture containing chloroprene, unreacted 3,4-dichloro-butene-1 and water produced by performing a reaction at a temperature of $20 - 100^{\circ}$ C is removed in a gas phase via evaporation without reflux of said mixture into the reaction system." (Ex. 1005 at 6:19-23 .)	
Claim 10 (cont.)	(Ex. 1	,	Disclosure from <i>Harrison</i> (Ex. 1006)
d. recovering spent	(Ex. 1	005 Figure, decanter	"(b) contacting [an

dehydrohalogenating agent.	20, enricher 21, centrifuge 25.) "The aqueous phase was extracted via Conduit 4 such that the level of Reactor 19 remained constant and the lightly mixed lipid and aqueous phases (including precipitated sodium chloride) were separated using Decanter 20, after which the lipid phase was returned to the reactor via Conduit 5." (Ex. 1005 at 10:33-11:1.) "The aqueous phase was fed into a forced circulation- type Enricher 21 via Conduit 6 and concentration was carried out After passing through Conduit 9 from Conduit 8, the aqueous phase was heated in Heat Exchanger 22 after which it was circulated in Enricher 21 via Conduit 10." (Ex. 1005 at 11:1-9.) "As concentration was performed, dissolved sodium chloride precipitated out, but said aqueous phase was extracted via Conduit 8, directed into Slurry Tank 24 via Conduit 11 and fed into Centrifuge where precipitated sodium chloride was separated out and also discarded via Conduit 12." (Ex.1005 at 11:9-14.)	aqueous solution of potassium fluoride and potassium hydroxide] with finely divided high purity lime to thereby obtain a calcium fluoride precipitate in aqueous potassium hydroxide (c) settling the reaction product of step (b) into two phases comprising an aqueous potassium hydroxide supernatant and an aqueous slurry of calcium fluoride; (d) recovering the aqueous potassium hydroxide supernatant" (Ex.1006 2:14-21 .) "The basic potassium fluoride solution is then mixed with solid hydrated (slaked) lime in a stirred vessel where this calcium hydroxide [Ca(OH) ₂] reacts to form the calcium fluoride precipitate and regenerate the potassium hydroxide." (Ex. 1006 at 3:6-10 .)

7. Claims 11 and 12

Claim 11 depends from claim 10 and recites "... wherein the second product stream further comprises one or more dissolved organics." Claim 12 depends from

claim 11 and recites "...wherein the dissolved organics are selected from the group consisting of 1,1,1,2,3,3-hexafluoropropane (HFC-236ea), 1,1,1,2,3-pentafluoropropane (HFC-245eb), and combinations thereof."

Petition for Inter Partes Review of U.S. Pat. No. 8.710.282

Smith describes the use of organic co-solvents or diluents in combination with water and a base (e.g., KOH) in the aqueous phase. Exemplary solvents or organics diluents include fluorinated such as hexafluoroisopropanol, perfluorotetrahydrofuran, and perfluorodecalin in the dehydrofluorination reaction mixture. Ex. 1003 at 12:27-13:4. Other prior art references also describe basemediated dehydrofluorination reactions (e.g., $245eb + KOH \rightarrow 1234yf + KF$) conducted with organic solvents or co-solvents in which hydrofluoroalkanes (e.g., 245eb) are at least partially miscible. Ex. 1002 at ¶ 120; Ex. 1008 at 1316; Ex. 1009 at \P [0052] and [0086]. Given the solubility of 236ea and 245eb (the fluoroalkane starting materials of Smith) in organic liquids, a POSITA would expect some portion of the organic starting materials (236ea and 245eb) to be dissolved in an organic co-solvent or diluent component of the reaction stream if one was present. Ex. 1002 at ¶ 120. Accordingly, Smith, in combination with Masayuki and Harrison, teaches a second product stream further comprising one or more dissolved organics (claim 11) and that said dissolved organics are selected from the group consisting of 236ea, 245eb, and combinations thereof (claim 12). Because the combination of *Smith*, *Masayuki*, and *Harrison* teaches each limitation

in claims 11 and 12, the claims are invalid under 35 U.S.C. § 103, as shown in the

following chart. See Ex. 1002 at ¶ 120.

'282 patent claim	Disclosure from Smith (Ex. 1003)
11. The method of claim 10 wherein the second product stream further comprises one or more dissolved organics.	"Thus, the dehydrofluorination reaction may preferably use an aqueous solution of at least one base without the need for a co-solvent or diluent. However, a co-solvent or diluent can be used to act as a preferred phase for reaction by-products Useful co-solvents or diluents include those that are not reactive with or negatively impact the equilibrium or kinetics of the process and include alcohols such as methanol and ethanol; diols such as ethylene glycol; ethers such as diethyl ether, dibutyl ether; esters such as methyl acetate, ethyl acetate and the like; linear, branched and cyclic alkanes such as cyclohexane, methylcyclohexane; fluorinated diluents such as hexafluoroisopropanol, perfluorotetrahydrofuran and perfluorodecalin." (Ex.1003 at 12:27-13:4.)
12. The method of claim 11 wherein the dissolved organics are selected from the group consisting of 1,1,1,2,3,3- hexafluoropropane (HFC-236ea), 1,1,1,2,3- pentafluoropropane (HFC-245eb), and combinations thereof.	"Thus, the dehydrofluorination reaction may preferably use an aqueous solution of at least one base without the need for a co-solvent or diluent. However, a co-solvent or diluent can be used to act as a preferred phase for reaction by-products Useful co-solvents or diluents include those that are not reactive with or negatively impact the equilibrium or kinetics of the process and include alcohols such as methanol and ethanol; diols such as ethylene glycol; ethers such as diethyl ether, dibutyl ether; esters such as methyl acetate, ethyl acetate and the like; linear, branched and cyclic alkanes such as cyclohexane, methylcyclohexane; fluorinated diluents such as hexafluoroisopropanol, perfluorotetrahydrofuran and perfluorodecalin." (Ex.1003 at 12:27-13:4 .)

8. Claims 13 and 14

Claim 13 depends from claim 10 and recites "...wherein the spent dehydrohalogenating agent is purified using at least one separation method." Claim 14 depends from claim 13 and recites "...wherein the separation method is

Petition for Inter Partes Review of U.S. Pat. No. 8,710,282 selected from the group consisting of distillation or phase separation." Masayuki discloses separating aqueous spent alkali metal hydroxide from a lipid phase using a decanter, thereby purifying the spent alkali metal hydroxide. Ex. 1005 at 10:33-37 and Figure; Ex. 1002 at ¶ 121. Additionally, Harrison teaches separating KOH from KF by converting the KF to CaF₂ and separating the CaF₂ from the KOH by settling, thereby purifying the KOH. Ex. 1006 at 2:14-21; Ex. 1002 at ¶ 121. The decanter 20 in Masayuki and the settling in Harrison are both phase separation techniques. Ex. 1002 at ¶ 121. It would be obvious to a POSITA to use any of these well-known separation techniques to separate components for purification. Ex. 1002 at ¶ 121. Because the combination of Smith, Masayuki, and Harrison teaches each element of claims 13 and 14, the claims are invalid under 35 U.S.C. § 103 over the combination of Smith, Masayuki, and Harrison, as shown in the following chart. See Ex. 1002 at ¶ 121.

²⁸² patent claim	Disclosure from <i>Masayuki</i> (Ex. 1005)	Disclosure from <i>Harrison</i> (Ex. 1006)
13. The method of claim 10 wherein the spent	(Ex. 1005 Figure, decanter 20 and centrifuge 25.)	"(b) contacting [an aqueous solution of potassium fluoride and
dehydrohalogenatin g agent is purified using at least one separation method.	"The aqueous phase was extracted via Conduit 4 such that the level of Reactor 19 remained constant and the slightly mixed lipid and aqueous phases (including precipitated sodium chloride) were separated using Decanter 20." (Ex.	potassium hydroxide] with finely divided high purity lime to thereby obtain a calcium fluoride precipitate in aqueous potassium hydroxide; (c) settling the reaction product of step (b) into two phases comprising an aqueous potassium hydroxide supernatant and an

	1005 at 10:33-37.)	aqueous slurry of calcium fluoride; (d) recovering the aqueous potassium hydroxide supernatant" (Ex.1006 2:14-21.)
14. The method of claim 13 wherein the separation method is selected from the group consisting of distillation or phase separation.	(Ex. 1005 Figure, decanter 20 and centrifuge 25.) "The aqueous phase was extracted via Conduit 4 such that the level of Reactor 19 remained constant and the slightly mixed lipid and aqueous phases (including precipitated sodium chloride) were separated using Decanter 20." (Ex. 1005 at 10:33-37.)	" (b) contacting [an aqueous solution of potassium fluoride and potassium hydroxide] with finely divided high purity lime to thereby obtain a calcium fluoride precipitate in aqueous potassium hydroxide; (c) settling the reaction product of step (b) into two phases comprising an aqueous potassium hydroxide supernatant and an aqueous slurry of calcium fluoride; (d) recovering the aqueous potassium hydroxide supernatant" (Ex.1006 2:14-21.)

Claim 15 depends from claim 10 and further provides "optionally, concentrating the purified dehydrohalogenating agent and recycling it back to the dehydrohalogenation reaction." *Masayuki* discloses that "[t]he aqueous phase ... was fed into [a] forced circulation-type Enricher 21 via Conduit 6 and concentration [of the aqueous phase] was carried out ... Recovered sodium hydroxide aqueous solution was returned to Conduit 2 via Conduit 13 and fed back into the reaction." Ex. 1005 at 11:1-16. As explained above, the decanter 20 is a phase separator that separates the lipid phase (e.g., organic catalyst) from the

Petition for Inter Partes Review of U.S. Pat. No. 8,710,282

aqueous phase. Ex. 1002 at ¶ 122. Thus, the aqueous phase that is withdrawn from the decanter 20 is purified relative to the aqueous phase that was removed from the reactor and introduced to the decanter 20. *Id.* The circulation-type enricher 21 concentrates the aqueous phase by evaporating water. *Id.* The concentrated, purified aqueous phase remaining after the centrifuge, which comprises an alkali metal hydroxide solution, is returned to the reactor for re-use. *Id.* Thus, the combination of *Smith, Masayuki,* and *Harrison* teaches concentrating the purified DHA and recycling it back to the dehydrohalogenation reaction. Because the combination of *Smith, Masayuki,* and *Harrison* teaches each element of claim 15, the claim is invalid under 35 U.S.C. § 103 over the combination of *Smith, Masayuki,* and *Harrison* teaches each element of 20 claim 15, the claim is invalid under 35 U.S.C. § 103 over the combination of *Smith, Masayuki,* and *Harrison* teaches each element of 20 claim 15, the claim is invalid under 35 U.S.C. § 103 over the combination of *Smith, Masayuki,* and *Harrison* teaches each element of 20 claim 15, the claim is invalid under 35 U.S.C. § 103 over the combination of *Smith, Masayuki,* and *Harrison* teaches each element of claim 15, the claim is invalid under 35 U.S.C. § 103 over the combination of *Smith, Masayuki,* and *Harrison,* as shown in the following chart. *See* Ex. 1002 at ¶ 122.

'282 patent claim	Disclosure from Masayuki (Ex. 1005)
15. The method of claim 10 further comprising, optionally, concentrating the purified dehydrohalogenating agent and recycling it back to the dehydrohalogenation reaction.	"The aqueous phase containing solid sodium chloride from which the lipid phase was removed using Decanter 20 was fed into forced circulation-type Enricher 21 via Conduit 6 and concentration was carried out As concentration was performed, dissolved sodium chloride precipitated out, but said aqueous phase was extracted via Conduit 8, directed into Slurry Tank 24 via Conduit 11 and fed into Centrifuge 25 where precipitated sodium chloride was separated out Recovered sodium hydroxide aqueous solution was returned to Conduit 2 via Conduit 13 and fed back into the reaction." (Ex. 1005 at 11:1-16 .)

Claim 16 depends from claim 10 and requires that the reaction stream further comprises by-product salts of the dehydrohalogenating agent. *Smith* teaches the dehydrofluorination of 245eb using KOH to produce 1234yf. Ex. 1003 at 1:23-2:2, 11:9-15; Ex. 1002 at ¶ 123. A POSITA would understand that a by-product of the dehydrofluorination of 245eb with KOH is KF and that the reaction stream therefore comprises KF, which is a by-product salt of the DHA. *See* Ex. 1002 at ¶ 123; Ex. 1010 at 13:16-22. Because the combination of *Smith*, *Masayuki*, and *Harrison* teaches each element of claim 16, the claim is invalid under 35 U.S.C. § 103 over the combination of *Smith*, *Masayuki*, and *Harrison*, as shown in the following chart. *See* Ex. 1002 at ¶ 123.

'282 patent claim	Disclosure from Smith (Ex. 1003)
16. The method of claim 10 wherein the reaction stream further comprises by-product salts of the dehydrohalogenatin g agent.	"The subject invention addresses the deficiencies of the known methods for preparing 1234yf by providing a process for the preparation of 1234yf comprising(b) dehydrofluorinating 236ea to produce 1,2,3,3,3- pentafluoropropene (referred to hereinafter as 1225ye); and (d) dehydrofluorinating 245eb to produce 1234yf ." (Ex. 1003 at 1:23-2:2 .)
	"Another preferred method of effecting the dehydrofluorination in steps (b) and (d) is by contacting 236ea and/or 245eb with a base. Preferably, the base is a metal hydroxide, e.g. an alkali or alkaline earth metal hydroxide or amide) the term "alkali metal hydroxide" [refers] to a compound or mixture of compounds selected from lithium hydroxide, sodium hydroxide, potassium hydroxide, rubidium hydroxide and caesium hydroxide." (Ex. 1003 at 11: 9-15 .)

Claim 17 depends from claim 16, which depends from claim 10. Claim 17 recites converting the by-product salts to the DHA. As discussed above, claims 10 and 16 are obvious in view of the combination of *Smith*, *Masayuki*, and *Harrison*. *Harrison* discloses conversion of a by-product salt (i.e., KF) to the DHA (i.e., KOH). Ex. 1006 at 2:14-16, 3:6-10; Ex. 1002 at ¶ 124. Because the combined disclosure of *Smith*, *Masayuki*, and *Harrison* teaches each limitation in claim 17, the claim is invalid under 35 U.S.C. § 103, as shown in the following chart. *See* Ex. 1002 at ¶ 124.

²⁸² patent claim	Disclosure from Harrison (Ex. 1006)
17. The method of claim 16 further comprising converting the by-product salts to the DHA.	"(b) contacting [an aqueous solution of potassium fluoride and potassium hydroxide] with finely divided high purity lime to thereby obtain a calcium fluoride precipitate in aqueous potassium hydroxide." (Ex. 1006 at 2:14-16.)
	"The basic potassium fluoride solution is then mixed with solid hydrated (slaked) lime in a stirred vessel where this calcium hydroxide $[Ca(OH)_2]$ reacts to form the calcium fluoride precipitate and regenerate the potassium hydroxide." (Ex. 1006 at 3:6-10 .)

12. Claim 18

Claim 18 depends from claim 16, which depends from claim 10. Claim 18 states that "...the dehydrohalogenating agent is KOH and the by-product salt is KF." *Smith* teaches a dehydrofluorination reaction in which a fluoroalkane (e.g., 245eb) is reacted with a DHA (e.g., KOH) to produce a fluoroalkene. Ex. 1003 at 1:23-2:2, 11:9-15. As stated above, a POSITA would understand that KF is a by-

product salt of the dehydrofluorination of 245eb with KOH. Ex. 1002 at ¶ 125;

Ex.1010 at 13:16-22. Because the combined disclosure of Smith, Masayuki, and

Harrison teaches each limitation in claim 18, the claim is invalid under 35 U.S.C.

§ 103, as shown in the following chart. See Ex. 1002 at ¶ 125.

'282 patent claim	Disclosure from Smith (Ex. 1003)
18. The method of claim 16 wherein the dehydrohalogenating agent is KOH and the by-product salt is KF.	"The subject invention addresses the deficiencies of the known methods for preparing 1234yf by providing a process for the preparation of 1234yf comprising(b) dehydrofluorinating 236ea to produce 1,2,3,3,3- pentafluoropropene (referred to hereinafter as 1225ye); and (d) dehydrofluorinating 245eb to produce 1234yf ." (Ex. 1003 1:23-2:2 .)
	"Another preferred method of effecting the dehydrofluorination in steps (b) and (d) is by contacting 236ea and/or 245eb with a base. Preferably, the base is a metal hydroxide, e.g. an alkali or alkaline earth metal hydroxide or amide) the term "alkali metal hydroxide" [refers] to a compound or mixture of compounds selected from lithium hydroxide, sodium hydroxide, potassium hydroxide, rubidium hydroxide and caesium hydroxide." (Ex. 1003 at 11: 9-15 .)

13. Claim 19

Claim 19 depends from claim 18 and recites "optionally, concentrating the converted KOH and recycling it back to the dehydrohalogenation reaction." The step of concentrating the converted KOH is not required by claim 19 because it is an "optional" step. As discussed above in connection with claim 1, *Harrison* teaches that KF in the aqueous mixture of KOH and KF is converted to KOH by treating the mixture with Ca(OH)₂. Ex. 1006 at 2:14-16, 3:6-10. Further, a POSITA would understand that the conversion step of *Harrison* can be

implemented in the slurry tank 24 of *Masayuki* without disruption to any of the other processing steps in *Masayuki*. Ex. 1002 at ¶ 126. In *Masayuki*, the centrifuge 25 separates out the precipitated solid (which would be CaF_2 when *Masayuki* is combined with *Smith* and *Harrison*) from the aqueous phase containing concentrated alkali metal hydroxide (KOH). Ex. 1002 at ¶ 126. The aqueous phase containing converted KOH is returned to conduit 2 via conduit 13 and recycled to the dehydrofluorination reaction. Ex. 1005 at 11:1-16; Ex. 1002 at ¶ 126.

Petition for Inter Partes Review of U.S. Pat. No. 8,710,282

Because the combined disclosure of *Smith*, *Masayuki* and *Harrison* teaches each limitation in claim 19, the claim is invalid under 35 U.S.C. § 103 over the combination of *Smith*, *Masayuki*, and *Harrison*, as shown in the following chart. *See* Ex. 1002 at ¶ 126.

'282 patent claim	Disclosure from <i>Masayuki</i> (Ex. 1005)	Disclosure from <i>Harrison</i> (Ex. 1006)
19. The method of claim 18 further comprising, optionally, concentrating the converted KOH and recycling it back to the dehydrohalogenation reaction.	"The aqueous phase containing solid sodium chloride from which the lipid phase was removed using Decanter 20 was fed into forced circulation-type Enricher 21 via Conduit 6 and concentration was carried out As concentration was performed, dissolved sodium chloride precipitated out, but said aqueous phase was	"(b) contacting [an aqueous solution of potassium fluoride and potassium hydroxide] with finely divided high purity lime to thereby obtain a calcium fluoride precipitate in aqueous potassium hydroxide." (Ex. 1006 at 2:14-16 .)
	extracted via Conduit 8, directed into Slurry Tank 24 via Conduit 11 and fed into Centrifuge 25 where	"The basic potassium fluoride solution is then mixed with solid hydrated (slaked) lime in

Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282 precipitated sodium chloride a stirred vessel where was separated out this calcium hydroxide Recovered sodium hydroxide $[Ca(OH)_2]$ reacts to form the calcium fluoride aqueous solution was returned to Conduit 2 via Conduit 13 precipitate and and fed back into the regenerate the potassium reaction." (Ex. 1005 at 11:1hydroxide." (Ex. 1006 at **16**.) 3:6-10.)

IPR2015-00916

14. Claim 20

Claim 20 depends from claim 18 and recites converting KF to KOH in the presence of Ca(OH)₂. As discussed above in connection with claims 1 and 17, *Harrison* teaches that KF in the aqueous mixture of KOH and KF is converted to KOH by treating the mixture with Ca(OH)₂. Ex. 1006 at 2:14-16, 3:6-10. Because the combination of *Smith*, *Masayuki*, and *Harrison* teaches each element of claim 20, the claim is invalid as obvious under 35 U.S.C. § 103(a), as shown in the following chart. *See* Ex. 1002 at ¶ 127.

'282 patent claim	Disclosure from Harrison (Ex. 1006)
20. The method of claim 18 further comprising converting KF to KOH in the presence of Ca(OH) ₂ .	 "(b) contacting [an aqueous solution of potassium fluoride and potassium hydroxide] with finely divided high purity lime to thereby obtain a calcium fluoride precipitate in aqueous potassium hydroxide." (Ex. 1006 at 2:14-16.) "The basic potassium fluoride solution is then mixed with solid hydrated (slaked) lime in a stirred vessel where this calcium hydroxide [Ca(OH)₂] reacts to form the calcium fluoride precipitate and regenerate the potassium hydroxide." (Ex. 1006 at 3:6-10.)

X. CONCLUSION

For the foregoing reasons, Petitioner respectfully requests that *inter partes* review of the '282 patent be instituted and that claims 1-20 be cancelled as unpatentable under 35 U.S.C. § 318(b).

Respectfully submitted, BRINKS GILSON & LIONE

April 20, 2015 Date / Jon Beaupré/ Jon Beaupré (Reg. No. 54,729) Allen R. Baum (Reg. No. 36,086) Allyn B. Elliott (Reg. No. 56,745) Joshua E. Ney (Reg. No. 66,652) Nicholas A. Restauri (Reg. No. 71,783) 524 South Main St., Suite 200 Ann Arbor, MI 48104 Attorneys for Petitioner Arkema France

CERTIFICATE OF SERVICE

I certify that, on April 20, 2015, copies of the following documents have

been served in their entirety on the Patent Owner, Honeywell International, Inc.

("Honeywell"):

- Petition for *Inter Partes* Review Under 35 U.S.C. § 311 & 37 C.F.R. 42.101
- 2. Exhibit List
- 3. Exhibits 1001-1018
- 4. Power of Attorney Under 37 C.F.R. § 42.10(b).

The copies have been served on Honeywell by causing them to be sent by

Express Mail® (Label No. EV 950035917 US and EV 950035894 US,

respectively) to the following addresses:

Honeywell International, Inc. Patent Services 101 Columbia Road Morristown, NJ 07962 Fox Rothschild LLP Attn: J. Eric Sumner 747 Constitution Drive Suite 100 Exton, PA 19341-0673

Respectfully submitted,

BRINKS GILSON & LIONE

April 20, 2015

/Nicholas Restauri/ Jon Beaupré (Reg. No. 54,729) Allen R. Baum (Reg. No. 36,086) Allyn B. Elliott (Reg. No. 56,745) Joshua E. Ney (Reg. No. 66,652) Nicholas A. Restauri (Reg. No. 71,783)

IPR2015-00916 Petition for *Inter Partes* Review of U.S. Pat. No. 8,710,282

BRINKS GILSON & LIONE 524 South Main Street, Suite 200 Ann Arbor, Michigan 48104 734.302.6000 734.994.6331 (fax) Attorneys for Petitioner