ARKEMA FRANCE,
Petitioner
v.
HONEYWELL INTERNATIONAL INC.,
Patent Owner

Case IPR2015-00915, 916, 917
Re: U.S. Patent 8,710,282

Patent Owner's Demonstratives
Under §42.70(b) for July 18, 2016 Oral Hearing

INTRODUCTION

- Arkema's characterization of Lousenberg's testimony
- Arkema's arguments in its Reply Briefs
- Certain references are not prior art
- 2008 Reduction to Practice performed in laboratory

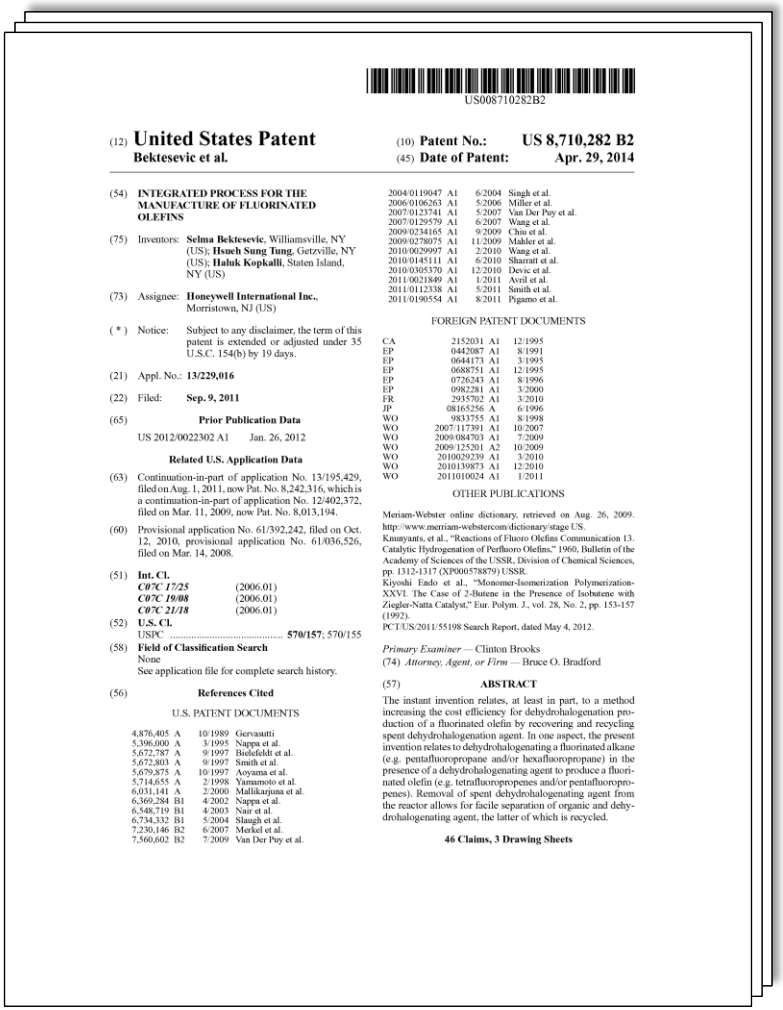
**Unless noted otherwise, all references to Papers are from IPR2015-00915

ROADMAP

- Honeywell's Invention in 2008
- Proper Claim Construction
- Honeywell's Diligence and Actual Reduction to Practice
- Honeywell's Diligence to Constructive Reduction to Practice
- Sedat, Smith, Masayuki and Harrison are Distinguished
- The Asserted References Cannot be Combined

The History of the '282 Patent

BACKGROUND OF THE '282 PATENT



US008710282B2

(12) **United States Patent**
Bektesevic et al.

(10) **Patent No.: US 8,710,282 B2**
(45) **Date of Patent: Apr. 29, 2014**

(54) **INTEGRATED PROCESS FOR THE MANUFACTURE OF FLUORINATED OLEFINS**

(75) Inventors: **Selma Bektesevic**, Williamsville, NY (US); **Hsueh Sung Tung**, Getzville, NY (US); **Haluk Kopkalli**, Staten Island, NY (US)

(73) Assignee: **Honeywell International Inc.**, Morristown, NJ (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 19 days.

(21) Appl. No.: **13/229,016**

(22) Filed: **Sep. 9, 2011**

(65) **Prior Publication Data**

US 2012/0022302 A1 Jan. 26, 2012

Related U.S. Application Data

(63) Continuation-in-part of application No. 13/195,429, filed on Aug. 1, 2011, now Pat. No. 8,242,316, which is a continuation-in-part of application No. 12/402,372, filed on Mar. 11, 2009, now Pat. No. 8,013,194.

(60) Provisional application No. 61/392,242, filed on Oct. 12, 2010, provisional application No. 61/036,526, filed on Mar. 14, 2008.

(51) **Int. Cl.**
C07C 17/25 (2006.01)
C07C 19/08 (2006.01)
C07C 21/18 (2006.01)

(52) **U.S. Cl.**
USPC **570/157**; 570/155

(58) **Field of Classification Search**
None
See application file for complete search history.

(56) **References Cited**

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Primary Examiner — Clinton Brooks
(74) *Attorney, Agent, or Firm* — Bruce O. Bradford

(57) **ABSTRACT**

The instant invention relates, at least in part, to a method increasing the cost efficiency for dehydrohalogenation production of a fluorinated olefin by recovering and recycling spent dehydrohalogenation agent. In one aspect, the present invention relates to dehydrohalogenating a fluorinated alkane (e.g. pentafluoropropane and/or hexafluoropropane) in the presence of a dehydrohalogenating agent to produce a fluorinated olefin (e.g. tetrafluoropropenes and/or pentafluoropropenes). Removal of spent dehydrohalogenating agent from the reactor allows for facile separation of organic and dehydrohalogenating agent, the latter of which is recycled.

46 Claims, 3 Drawing Sheets

U.S. 8,710,282 to Bektesevic et al.

Claim Construction

SPENT DHA/KOH

- "An aqueous solution containing [DHA or KOH] and by-product salts"
  - "POSITA would understand that the aqueous solution would necessarily comprise a mix of the [DHA or KOH] and by-product salts." PO Response, Paper 19, at 5.

Spent dehydrohalogenating agent 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 known separation techniques. To this end, spent dehydrohalogenating agent separation may occur using any known compound separation techniques, such, but not limited to, distillation, phase separation, etc. In certain

EX1001 at 13:16-22

91. While the above referenced section may be read to indicate that the [DHA or KOH] can be withdrawn separately from the by-product salts, the person of ordinary skill in the art would understand that the aqueous solution would necessarily comprise a mix of the [DHA or KOH] and by-product salts. For example, in the aqueous solution, there are alkali metal cations, fluoride anions, and hydroxide anions. In such a case, alkali metal cations are not associated with any one species of anion.

EX2002, ¶ 91

SPENT DHA/KOH

EXAMPLES

The following examples are provided for the purpose of illustrating the present invention but without limiting the scope thereof.

Example 1

A CSTR (Continuously Stirred Tank Reactor) was used to convert 245eb to 1234yf. The reactor was designed and operated in such a way that the end of the dip tube was in the middle of KOH phase. After the startup, organic and fresh KOH were constantly fed in. Organic out (the overhead portion) was constantly taken out. In a typical cycle organic and KOH flow in and overhead is taken out for 30 min. Flowrates were 5 and 12 ml/min, respectively. The mixture is stirred during this period of time. Then stirrer is stopped for 10 min, while feeds still go in. While stirrer is off, after 10 min, applicable valves for the spent KOH to go out were open. Between 500 and 550 g was taken out every 40 min.

Experiment was carried out for 7 hours. Temperature was 48° C. Pressure was 45 psig. GC area percent of the organic feed was ~97% of 245eb, the rest being most probably 254. The flowrates translate to mol ratio of KOH to G-245eb of 1, or slightly above 1. Theoretical residence time was 100 min.

Scrubber Product Collection Cylinder (SPCC) was used to collect spent KOH and the portion of organic that was carried out with spent KOH. Organic was trapped in methylene chloride which was previously added to SPCC. The volume of methylene chloride inside the SPCC was 15% of the total volume of liquid in SPCC. Product and un-reacted organic was collected in a Product Collection Cylinder (PCC) that was held in dry ice. Vapor samples were taken once per hour while liquid samples were taken at much longer intervals.

Overall mass balance was 94% for organic. Average conversion was 48%. Selectivity to 1234yf was 73%.

EX2004 at 85:20-86:8

20 Q. So in that example, the KF and the KOH
21 would both be collected together, correct?
22 MR. BEAUPRE: Objection, form and
23 foundation.
24 A. They would be part of the aqueous stream
25 that was collected.
1 Q. So when they collect the spent KOH, that
2 includes unreacted KOH, the KF, which is the result of
3 the reaction, and water, correct?
4 MR. BEAUPRE: Objection, form and
5 foundation.
6 A. That's an interpretation, yes.
7 Q. Is that a reasonable interpretation?
8 A. That's a reasonable interpretation.

EX2004 at 85:20-86:8

RECOVERING SPENT DHA/KOH

- Substantially separating spent [DHA or KOH] from any other materials in the product reaction stream (Claim 1)/second product stream (Claim 10)/reaction stream (Claims 21 and 36)]

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 spent dehydrohalogenating agent during the time agitation is stopped, 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. In one non-limiting embodiment, for example, KOH is separation by distillation, i.e. by heating organic just above boiling point of 236ea and/or 245eb, thus fractionating the organic from the spent KOH. Alternatively, one can use phase separator to separate between the two phases. 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.

EX1001 at 13:29-45

Stream

- “A continuous flow of a fluid consisting of at least spent [DHA or KOH] resulting from a dehydrofluorination reaction”

Q. Okay. And it's not intended to go anywhere. I'm just trying to understand how the term stream equates with volume, because to me, they mean two different things, and I want to get an understanding of how volume is the definition of stream?

A. I was simply meaning that the stream, which you're right, implies that it's flowing, and the volume of material is a part of that stream that you're collecting. It could include the entire stream you are collecting for further use. So that stream is flowing, and it's being collected somewhere for further use, otherwise it's being discarded.

So it could include, you know, if you ran this in a batch process, it could include everything that flowed out of the reactor and was collected in a bucket, which would then be the volume of material, that's what I meant.

EX2004 at 101:2-19

Actual Reduction to Practice

HONEYWELL'S DEVELOPMENT OF THE INVENTION

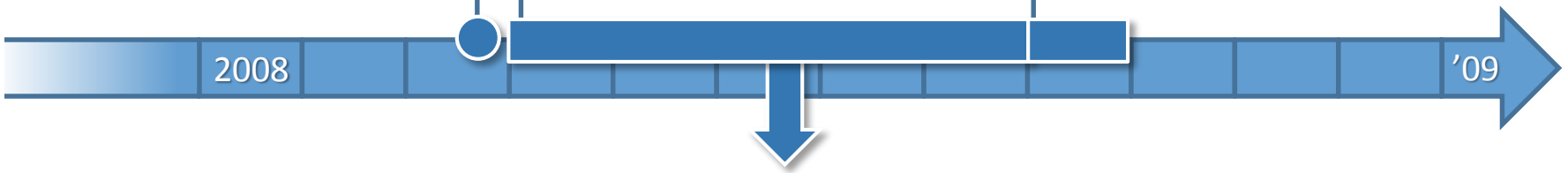
- Conception by Haluk Kopkalli
- Coordination by Dr. Tung with Dr. Bektesevic and Mr. Kopkalli
- Experiments Conducted by Dr. Bektesevic
- Actual Reduction to Practice by September 2008

TIMELINE

***Conception*:**
March 26, 2008
Kopkalli E-mail
(Paper 19, at 38; EX2007)

April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf. (Paper 19, at 38)

September 2008
Further Evaluating the Dehydrofluorination Reaction Pathway, Specifically With Respect to the Use of 236ea. (Paper 19, at 39)



"The inventors . . . actually reduced to practice . . . on or before September 2008." (Paper 19, at 27)

CORROBORATION – EX2006

- Kopkalli E-mail – March 26, 2008

> 11. On March 26, 2008, I received an email from Mr. Haluk Kopkalli, including an attachment titled "Apollo HHP + KOH BFD-080318.pdf" which is a document illustrating a potential commercial process including the steps of hydrogenation of HFP, dehydrofluorination with KOH, and regeneration and recycling of spent KOH. Submitted herewith as Exhibit 2007 is a true and accurate copy of the Kopkalli Email dated March 26, 2008 and associated attachments which I received from Mr. Kopkalli in the ordinary course of business. The email demonstrates that we conceived of the idea to use the hydrogenation of HFP and dehydrofluorination with KOH as a possible continuous process for commercial scale-up at least as early as March 26, 2008.

Tung Dec., EX2006

CORROBORATION – EX2002

- Kopkalli E-mail – March 26, 2008

141. It is my opinion that the Kopkalli Email dated March 26, 2008 and associated attachments reflect the formation in the mind of the inventor of a definite and permanent idea of the complete and operative invention, as it is thereafter to be applied in practice, as reflected in the drawings from the Kopkalli Email. (EX2007.)

142. Indeed, the Kopkalli Email dated March 26, 2008 and associated attachments reveals that the idea was definite and permanent enough that one skilled in the art could understand the invention without extensive research or experimentation. The Kopkalli Email dated March 26, 2008 and associated attachments encompasses all limitations of the claimed invention.

143. Accordingly, it is my opinion that the inventors conceived of the invention of Claims 1-46 of the '282 patent at least as early as March 26, 2008.

Lousenberg Dec., EX2002

CORROBORATION – EX2007

- Kopkalli E-mail – March 26, 2008

To establish conception, Honeywell submits that the Lousenberg and Tung Declarations establish an invention date that precedes the effective date of each of Smith and Sedat. The evidence, specifically, the March 26, 2008 email and attachments prepared by inventor Haluk Kopkalli ("Kopkalli Email dated March 26, 2008 and associated attachments") (EX2007), overwhelmingly demonstrate conception of the inventions claimed in the '282 Patent at least as early as March 26, 2008, more than one year before the alleged prior art dates of each of Smith and Sedat. (EX2002, ¶¶ 141-143; EX2006, ¶ 11.) The Kopkalli Email dated March 26, 2008 and associated attachments demonstrate that the ideas for the complete invention of the '282 Patent were definite and provided a particular solution to the problem at hand at least as early as March 26, 2008. (*Id.*) Accordingly, the conception date of the claims of the '282 Patent is at least as early as March 26, 2008.

PO Response, Paper 19, at 31

CORROBORATION – EX2010

- Bektesevic's Monthly Technology Report – July 2008

> In July 2008, Dr. Bektesevic prepared experiments to begin investigating the "[a]pplication of [Continuously Stirred Tank Reactor] CSTR concept on reaction between G-245eb and KOH" to yield 1234yf, as reflected in her monthly technology report for July 2008. (EX2010 at 1; *see also* EX2006, ¶ 21.) In doing so, Dr. Bektesevic provided instructions to lab technicians in order to carry out experiments related to caustic dehydrofluorination utilizing KOH on at least July 16, July 29, and July 30, 2008. (EX2006, ¶¶ 22, 27; *see also* EX2037, ¶¶ 4, 5.)

PO Response, Paper 19, at 39

CORROBORATION – EX2037

- Declaration of Angela Goodie

4. As is customary, Dr. Selma Bektesevic prepared and provided me with detailed instructions on or near July 16, 2008, in the regular course of business, so that I could perform caustic dehydrofluorination experiments utilizing KOH ("July 16 Instructions"). As instructed, I performed experiments in line with the July 16, 2008 Instructions using the lab setup described in the Project Permit Change Form (EX2009). I recall that pursuant to the July 16 Instructions, I filled

5. Dr. Bektesevic subsequently prepared and provided instructions for myself and my colleague Steven Phillips on or near July 29, 2008, in the regular course of business, instructing us to perform additional caustic dehydrofluorination experiments utilizing KOH on July 29-30, 2008 ("July 29 Instructions"). As instructed, we performed experiments in line with the July 29 Instructions. I recall

Exhibit 2037, ¶¶ 4, 5

CORROBORATION – EX2038

- Declaration of Daniel Merkel

> 4. Subsequent to the approval of the project permit and as noted on the invention disclosure form, I witnessed the performance of the caustic dehydrofluorination experiments utilizing KOH experiments by lab technicians Steven Phillips and Angela Mazurkiewicz (now Angela Goodie) on July 29-30, 2008 ("July 29 Instructions"). The July 29 Instructions that guided the experiments that I witnessed I understand have been submitted here as Exhibit 2012.

Exhibit 2038, ¶ 4

CORROBORATION – EX2013

- Bektesevic's Monthly Technology Report – August 2008

> As evidenced by Dr. Bektesevic's August 2008 Monthly Technology Report ("Bektesevic August 2008 Report"), several 1234yf experiments were conducted as well as experiments which reflected the CSTR concept being further applied to the reaction of 236ea and KOH to form 1225ye. (EX2006, at ¶¶ 29-32.)

PO Response, Paper 19, at 39

> data). A true and accurate copy of Dr. Bektesevic's August 2008 Report which I received in the ordinary course of business at or near the time is submitted herewith as Exhibit 2013. Dr. Bektesevic's August 2008 Report indicates that the major compound formed during this experiment was 1234yf, as expected, and only unreacted 245eb was observed in the scrubber PCC.

Tung Dec., EX2006, ¶ 29

> 32. Dr. Bektesevic's August 2008 Report also summarizes experiments conducted which reflect the CSTR concept being further applied to the reaction of 236ea and KOH to form 1225ye. The GC data from this experiment is found in Appendix 7 thereto.

Tung Dec., EX2006

CORROBORATION – EX2016

- Bektesevic's Monthly Technology Report – September 2008

> The inventors continued in September 2008, as they further evaluated the dehydrofluorination reaction pathway, specifically with respect to the use of 236ea. (*Id.*, ¶ 34.) Indeed, Dr. Bektesevic had an experiment run on September 15, 2008 (which continued through September 19, 2008) wherein spent KOH was taken out of the reactor intermittently using a 5-inch dip tube and she provided instructions to lab technicians to perform additional caustic dehydrofluorination experiments utilizing KOH, including instructions for HCl titration. (*Id.*, at ¶¶ 36-37; *see also* EX2037, ¶ 6.) It was in September 2008, as a result of the experiments, that the "[p]reliminary results demonstrated that CSTR is a feasible option for commercial operation." (EX2006, at ¶¶ 39-40; *see also* EX2017.)

PO Response, Paper 19, at 39-40

CORROBORATION – EX2037

- Declaration of Angela Goodie

> 6. Dr. Bektesevic subsequently prepared and provided instructions to me on or near September 15, 2008, in the regular course of business, to perform additional caustic dehydrofluorination experiments utilizing KOH ("September 15 Instructions"). As instructed, we performed experiments in line with the September 15 Instructions. I recall that pursuant to the September 15 Instructions,

Exhibit 2037, ¶ 6

CORROBORATION – EX2017

- September Disclosure ("feasible option")

> The inventors continued in September 2008, as they further evaluated the dehydrofluorination reaction pathway, specifically with respect to the use of 236ea. (*Id.*, ¶ 34.) Indeed, Dr. Bektesevic had an experiment run on September 15, 2008 (which continued through September 19, 2008) wherein spent KOH was taken out of the reactor intermittently using a 5-inch dip tube and she provided instructions to lab technicians to perform additional caustic dehydrofluorination experiments utilizing KOH, including instructions for HCl titration. (*Id.*, at ¶¶ 36-37; *see also* EX2037, ¶ 6.) It was in September 2008, as a result of the experiments, that the "[p]reliminary results demonstrated that CSTR is a feasible option for commercial operation." (EX2006, at ¶¶ 39-40; *see also* EX2017.)

PO Response, Paper 19, at 39-40

REDUCTION TO PRACTICE OF CLAIMED INVENTION

21. A process for the manufacture of a fluoroolefin comprising:

(a) dehydrohalogenating a haloalkane in the presence of KOH to produce a haloalkene;

(b) withdrawing a reaction stream comprising spent KOH; and

(c) recovering spent KOH.

"Application of CSTR concept on reaction between G-245eb and KOH has been started. Formation of G-1234yf was observed . . ." (EX2010 at 1; *see also* EX2006, ¶¶ 21-26; EX2037, ¶ 4.)

"Reaction between G-245eb and KOH to yield G-1234yf was studied using Continuously Stirred Tank Reactor (CSTR). At ~50 C, ~45 psig, and residence time of approximately 100 min, mass balance and conversions were over 90, and ~50%, respectively. Major compound formed was…G-1234yf." (EX2013 at 1; *see also* EX2006, ¶¶ 29-32 and EX2037, ¶ 5.)

"Reaction between 236ea and 25% KOH was carried out in Continuous Stirred Tank Reactor (CSTR). The experiment lasted for 97 hours. The theoretical residence time was 200 min; flowrates of KOH and Organic were 4, and 2ml/min. Reaction was carried out at ~43 C, while pressure varied between 35 and 45 psig. Mass balance was 88.67% and conversion of 236ea was ~50%." (EX2016 at 1; *see also* EX2006, ¶¶ 34-38 and EX2037, ¶ 16.)

PO Response, Paper 19, at 31

REDUCTION TO PRACTICE OF CLAIMED INVENTION

21. A process for the manufacture of a fluoroolefin comprising:

(a) dehydrohalogenating a haloalkane in the presence of KOH to produce a haloalkene;

(b) withdrawing a reaction stream comprising spent KOH; and

(c) recovering spent KOH.

"Vapor samples were taken once in 1.5 hour, liquid samples once in 4 hours." (EX2010 at 2; *see also id.* at Appendices 3 and 4; EX2006, ¶ 25 and EX2037, ¶ 4.)

"The first experiment was run in 'true' continuous mode . . . Both organic and spent KOH were taken constantly." (EX2013 at 1; EX2006, ¶ 29.)

"The next experiment we tried was to run in semi-continuous mode . . . Spent KOH is taken out through the dip tube. We decided that we will try to just remove spent KOH so the dip tube is cut in such a manner that the end of the dip tube is in the middle of KOH phase in reactor. Reactor length is 81/2" and KOH dip tube is 5" . . . While stirrer is off, after 10 min, applicable valves for the spent KOH to go out were open. We would take between 500 and 550 g . . . every 40 min." (EX2013 at 2; *see also* EX2006, ¶ 28, EX2037, ¶ 5.)

"Spent KOH was taken out of reactor periodically (it was fed continuously as was organic; overhead product was taken out continuously → semi-continuous operation)." (EX2016 at 2; EX2006, ¶ 36; EX2037, ¶ 6.)

PO Response, Paper 19, at 31-32

REDUCTION TO PRACTICE OF CLAIMED INVENTION

21. A process for the manufacture of a fluoroolefin comprising:

(a) dehydrohalogenating a haloalkane in the presence of KOH to produce a haloalkene;

(b) withdrawing a reaction stream comprising spent KOH; and

(c) recovering spent KOH.

Appendix 3 illustrates the recovery of spent KOH in a scrubber PCC. (EX2010 at Appendix 3; *see also* EX2006, ¶¶ 23-26; EX2037, ¶ 4.)

Appendices 1, 3 and 5 illustrate the recovery of spent KOH in a scrubber PCC. (EX2013 at Appendices 1, 3, and 5; *see also* EX2006, ¶ 30 and EX2037, ¶ 5.)

"Spent KOH was collected in scrubber PCC (SPCC). SPCC was pre-filled with methylene chloride (the target was to have 15-20 wt% of MeCl2 in the final spent KOH-MeCl2 solution). Analysis of methylene chloride layer revealed that ~70 GC area % was 236ea. There was small amount of 1225ye (~2%) while the rest were some unknown heavies. In order to confirm that all organic was extracted with MeCl2, part KOH was added to part MeCl2 (i.e. 1:1). GC analysis revealed that amount of organic was negligible." (EX2016 at 2; *id.* at Appendix 1; *see also* EX2006, ¶¶ 34-38; EX2037, ¶ 6.)

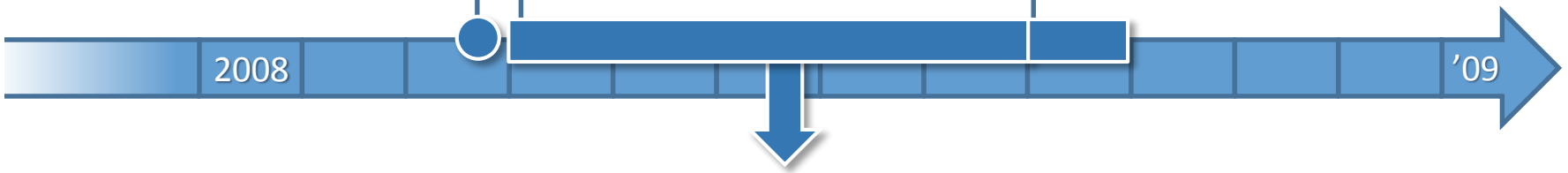
PO Response, Paper 19, at 32

SMITH AND SEDAT ARE NOT PRIOR ART

***Conception*:**
March 26, 2008
Kopkalli E-mail
(EX2007)

April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf

September 2008
Further Evaluating the Dehydrofluorination Reaction Pathway, Specifically With Respect to the Use of 236ea



"The inventors . . . actually reduced to practice . . . on or before September 2008."
(Paper 19, at 27)

SMITH AND SEDAT ARE NOT PRIOR ART

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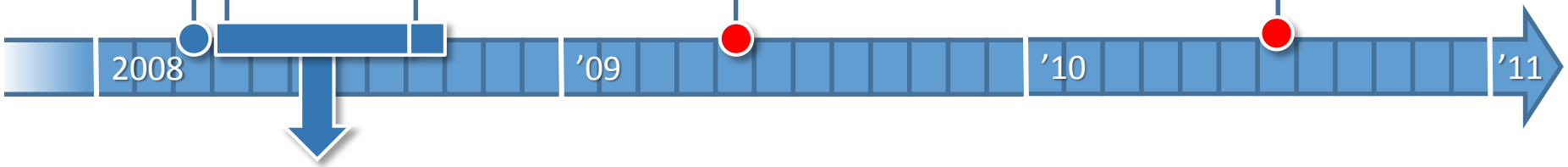
September 2008
Further Evaluating the Dehydrofluorination Reaction Pathway, Specifically With Respect to the Use of 236ea (Paper 19, at 39)

May 15, 2009
PCT/GB2009/001263
("Smith")

July 16, 2010
FR 2940968 ("Sedat")



"The inventors . . . actually reduced to practice . . . on or before September 2008."
(Paper 19, at 27)

Constructive Reduction to Practice

CONSTRUCTIVE REDUCTION TO PRACTICE

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March 26, 2008
Kopkalli E-mail
(Paper 19, at 38; EX2007)

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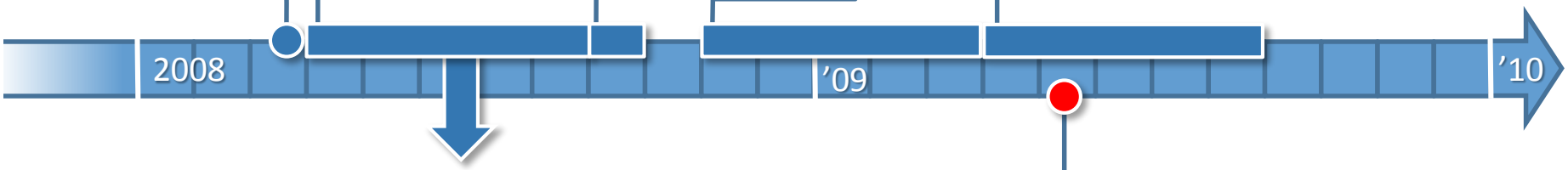
October 2008 – March 2009
Further Experiments and Analysis Were Performed in Order to Improve the Quality of the Experimental Data and to Complete Mass Balance (Paper 19, at 40)

April – August 2009
Honeywell Pursuing Commercialization and Development of 1234yf Globally (Paper 19, at 40)



"The inventors . . . actually reduced to practice . . . on or before September 2008." (Paper 19, at 27)

May 15, 2009
PCT/GB2009/001263 ("Smith")

CONSTRUCTIVE REDUCTION TO PRACTICE

***Conception*:**
March 26, 2008
Kopkalli E-mail
(Paper 19, at 38; EX2007)

April – August 2008
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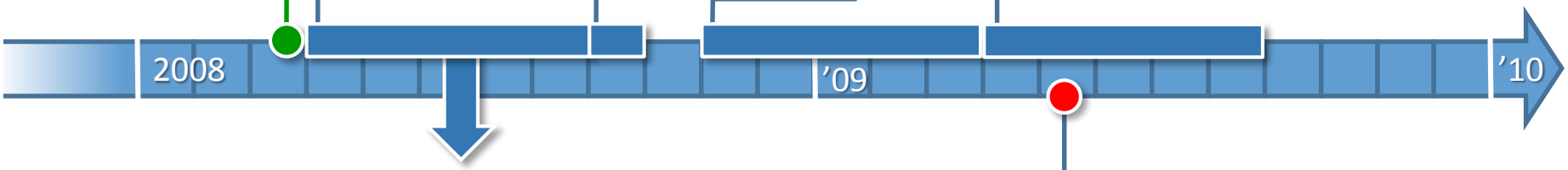
April – August 2009
Honeywell Pursuing Commercialization and Development of 1234yf Globally (Paper 19, at 40)

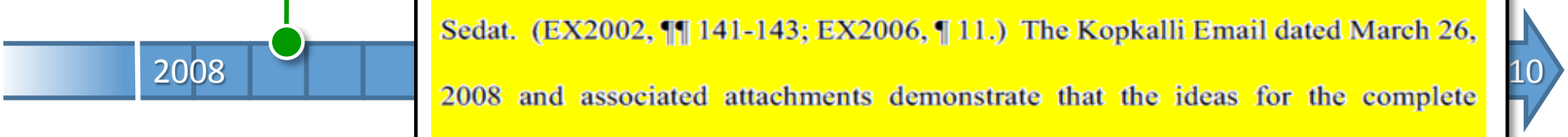


"The inventors . . . actually reduced to practice . . . on or before September 2008." (Paper 19, at 27)

May 15, 2009
PCT/GB2009/001263 ("Smith")

CONSTRUCTIVE REDUCTION TO PRACTICE

***Conception*:**
March 26, 2008
Kopkalli E-mail
(Paper 19, at 38; EX2007)



To establish conception, Honeywell submits that the Lousenberg and Tung Declarations establish an invention date that precedes the effective date of each of Smith and Sedat. The evidence, specifically, the March 26, 2008 email and attachments prepared by inventor Haluk Kopkalli ("Kopkalli Email dated March 26, 2008 and associated attachments") (EX2007), overwhelmingly demonstrate conception of the inventions claimed in the '282 Patent at least as early as March 26, 2008, more than one year before the alleged prior art dates of each of Smith and Sedat. (EX2002, ¶¶ 141-143; EX2006, ¶ 11.) The Kopkalli Email dated March 26, 2008 and associated attachments demonstrate that the ideas for the complete invention of the '282 Patent were definite and provided a particular solution to the problem at hand at least as early as March 26, 2008. (*Id.*) Accordingly, the conception date of the claims of the '282 Patent is at least as early as March 26, 2008.

PO Response, Paper 19, at 26-27

CONSTRUCTIVE REDUCTION TO PRACTICE

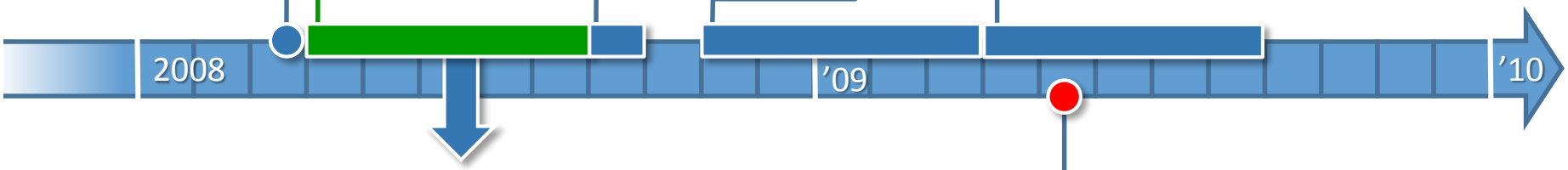
***Conception*: March 26, 2008**
Kopkalli E-mail (Paper 19, at 38; EX2007)

April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf (Paper 19, at 38)

September 2008
Further Evaluating the Dehydrofluorination Reaction Pathway, Specifically With Respect to the Use of 236ea (Paper 19, at 39)

October 2008 – March 2009
Further Experiments and Analysis Were Performed in Order to Improve the Quality of the Experimental Data and to Complete Mass Balance (Paper 19, at 40)

April – August 2009
Honeywell Pursuing Commercialization and Development of 1234yf Globally (Paper 19, at 40)



"The inventors . . . actually reduced to practice . . . on or before September 2008." (Paper 19, at 27)

May 15, 2009
PCT/GB2009/001263 ("Smith")

CONSTRUCTIVE REDUCTION TO PRACTICE

April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf (Paper 19, at 38)

As discussed above, the inventors conceived of the invention of claims 1-46 of the '282 Patent at least as early as March 26, 2008. Dr. Tung, as the Director of Technology of Fluorine Products, leads the development of the manufacturing technology of 1234yf. The Tung Declaration demonstrates that after conception of the invention at least as early as March 26, 2008, Dr. Tung worked with Dr. Selma Bektesevic to further evaluate utilizing a caustic reaction for the dehydrofluorination steps in producing 1234yf. (EX2006, ¶ 14.) On July 10, 2008, in preparation of such evaluation, Dr. Bektesevic submitted a Change Form (Exhibit 2008) to Permit #626, indicating that new chemicals 236ea, 245eb, 1234yf, 1225yeZ, and 1225yeE would be introduced into the existing apparatus. (*Id.*, at ¶¶ 16-17.) "The reaction proceeds by reacting organic chemicals with KOH to form salts in addition to new chemicals." The two reactions proposed to be carried out include: 236ea + KOH -> 1225ye + KF and 245eb + KOH -> 1234yf + KF. (*Id.*, at ¶18.)

PO Response, Paper 19, at 38

CONSTRUCTIVE REDUCTION TO PRACTICE

April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf (Paper 19, at 38)

In July 2008, Dr. Bektesevic prepared experiments to begin investigating the "[a]pplication of [Continuously Stirred Tank Reactor] CSTR concept on reaction between G-245eb and KOH" to yield 1234yf, as reflected in her monthly technology report for July 2008. (EX2010 at 1; *see also* EX2006, ¶ 21.) In doing so, Dr. Bektesevic provided instructions to lab technicians in order to carry out experiments related to caustic dehydrofluorination utilizing KOH on at least July 16, July 29, and July 30, 2008. (EX2006, ¶¶ 22, 27; *see also* EX2037, ¶¶ 4, 5.)

PO Response, Paper 19, at 39

CONSTRUCTIVE REDUCTION TO PRACTICE

April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf (Paper 19, at 38)

4. As is customary, Dr. Selma Bektesevic prepared and provided me with detailed instructions on or near July 16, 2008, in the regular course of business, so that I could perform caustic dehydrofluorination experiments utilizing KOH ("July 16 Instructions"). As instructed, I performed experiments in line with the July 16, 2008 Instructions using the lab setup described in the Project Permit Change Form (EX2009). I recall that pursuant to the July 16 Instructions, I filled

5. Dr. Bektesevic subsequently prepared and provided instructions for myself and my colleague Steven Phillips on or near July 29, 2008, in the regular course of business, instructing us to perform additional caustic dehydrofluorination experiments utilizing KOH on July 29-30, 2008 ("July 29 Instructions"). As instructed, we performed experiments in line with the July 29 Instructions. I recall

Declaration of Angela Goodie, Exhibit 2037, ¶¶ 4, 5

CONSTRUCTIVE REDUCTION TO PRACTICE

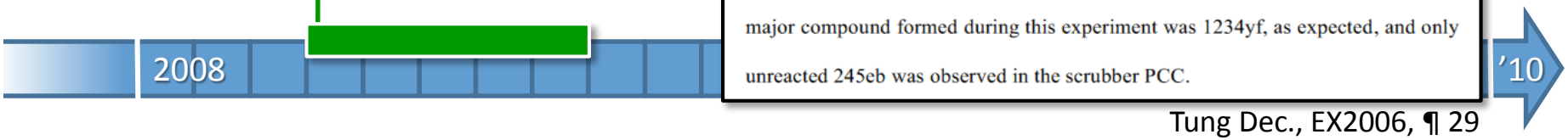
April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf (Paper 19, at 38)

4. Subsequent to the approval of the project permit and as noted on the invention disclosure form, I witnessed the performance of the caustic dehydrofluorination experiments utilizing KOH experiments by lab technicians Steven Phillips and Angela Mazurkiewicz (now Angela Goodie) on July 29-30, 2008 ("July 29 Instructions"). The July 29 Instructions that guided the experiments that I witnessed I understand have been submitted here as Exhibit 2012.

Declaration of Daniel Merkel, Exhibit 2038, ¶ 4

CONSTRUCTIVE REDUCTION TO PRACTICE

April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf (Paper 19, at 38)



As evidenced by Dr. Bektesevic's August 2008 Monthly Technology Report ("Bektesevic August 2008 Report"), several 1234yf experiments were conducted as well as experiments which reflected the CSTR concept being further applied to the reaction of 236ea and KOH to form 1225ye. (EX2006, at ¶¶ 29-32.)

PO Response, Paper 19, at 39

data). A true and accurate copy of Dr. Bektesevic's August 2008 Report which I received in the ordinary course of business at or near the time is submitted herewith as Exhibit 2013. Dr. Bektesevic's August 2008 Report indicates that the major compound formed during this experiment was 1234yf, as expected, and only unreacted 245eb was observed in the scrubber PCC.

Tung Dec., EX2006, ¶ 29

32. Dr. Bektesevic's August 2008 Report also summarizes experiments conducted which reflect the CSTR concept being further applied to the reaction of 236ea and KOH to form 1225ye. The GC data from this experiment is found in Appendix 7 thereto.

Tung Dec., EX2006

CONSTRUCTIVE REDUCTION TO PRACTICE

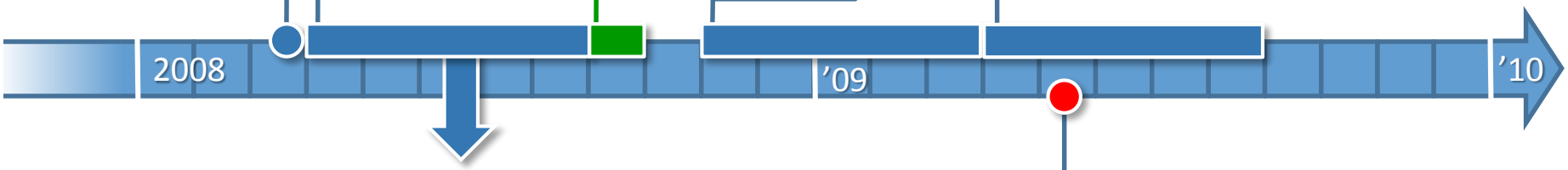
***Conception*: March 26, 2008**
Kopkalli E-mail (Paper 19, at 38; EX2007)

April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf (Paper 19, at 38)

September 2008
Further Evaluating the Dehydrofluorination Reaction Pathway, Specifically With Respect to the Use of 236ea (Paper 19, at 39)

October 2008 – March 2009
Further Experiments and Analysis Were Performed in Order to Improve the Quality of the Experimental Data and to Complete Mass Balance (Paper 19, at 40)

April – August 2009
Honeywell Pursuing Commercialization and Development of 1234yf Globally (Paper 19, at 40)



"The inventors . . . actually reduced to practice . . . on or before September 2008." (Paper 19, at 27)

May 15, 2009
PCT/GB2009/001263 ("Smith")

CONSTRUCTIVE REDUCTION TO PRACTICE

September 2008
Further Evaluating the Dehydrofluorination Reaction Pathway, Specifically With Respect to the Use of 236ea (Paper 19, at 39)

2008

The inventors continued in September 2008, as they further evaluated the dehydrofluorination reaction pathway, specifically with respect to the use of 236ea. (*Id.*, ¶ 34.) Indeed, Dr. Bektesevic had an experiment run on September 15, 2008 (which continued through September 19, 2008) wherein spent KOH was taken out of the reactor intermittently using a 5-inch dip tube and she provided instructions to lab technicians to perform additional caustic dehydrofluorination experiments utilizing KOH, including instructions for HCl titration. (*Id.*, at ¶¶ 36-37; *see also* EX2037, ¶ 6.) It was in September 2008, as a result of the experiments, that the "[p]reliminary results demonstrated that CSTR is a feasible option for commercial operation." (EX2006, at ¶¶ 39-40; *see also* EX2017.)

PO Response, Paper 19, at 39-40

CONSTRUCTIVE REDUCTION TO PRACTICE

September 2008
Further Evaluating the Dehydrofluorination Reaction Pathway, Specifically With Respect to the Use of 236ea (Paper 19, at 39)

2008

6. Dr. Bektesevic subsequently prepared and provided instructions to me on or near September 15, 2008, in the regular course of business, to perform additional caustic dehydrofluorination experiments utilizing KOH ("September 15 Instructions"). As instructed, we performed experiments in line with the September 15 Instructions. I recall that pursuant to the September 15 Instructions,

Declaration of Angela Goodie, Exhibit 2037, ¶ 6

CONSTRUCTIVE REDUCTION TO PRACTICE

***Conception*:**
March 26, 2008
Kopkalli E-mail
(Paper 19, at 38; EX2007)

April – August 2008
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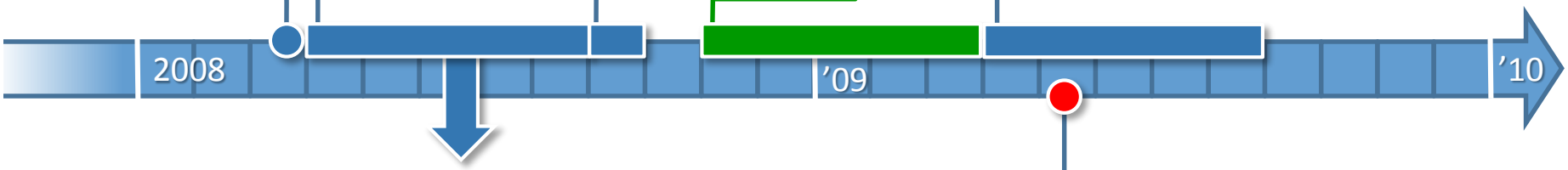
April – August 2009
Honeywell Pursuing Commercialization and Development of 1234yf Globally (Paper 19, at 40)



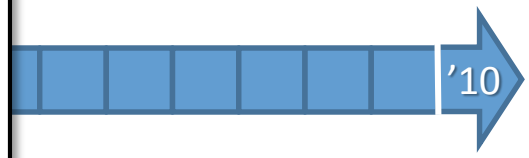
"The inventors . . . actually reduced to practice . . . on or before September 2008." (Paper 19, at 27)

May 15, 2009
PCT/GB2009/001263 ("Smith")

CONSTRUCTIVE REDUCTION TO PRACTICE

October 2008 – March 2009
Further Experiments and Analysis Were Performed in Order to Improve the Quality of the Experimental Data and to Complete Mass Balance (Paper 19, at 40)

As is typically done in an attempt to place the project in the best position for plant design by the engineering team, further experiments and analysis were performed in order to improve the quality of the experimental data and to complete mass balance. (EX2006, at ¶ 41.) Such further experiments and analysis included fine-tuning residence times, reaction temperatures, agitator speeds, conversion of reaction products, and kinetics of the reaction, as can be seen in Dr. Bektesevic's monthly reports between October 2008 and March 2009. (*Id.*, at ¶¶ 41-51; *see also* EX2018, EX2019, EX2021, EX2022, EX2024, and EX2026.)



PO Response, Paper 19, at 40

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***Conception*: March 26, 2008**
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April – August 2009
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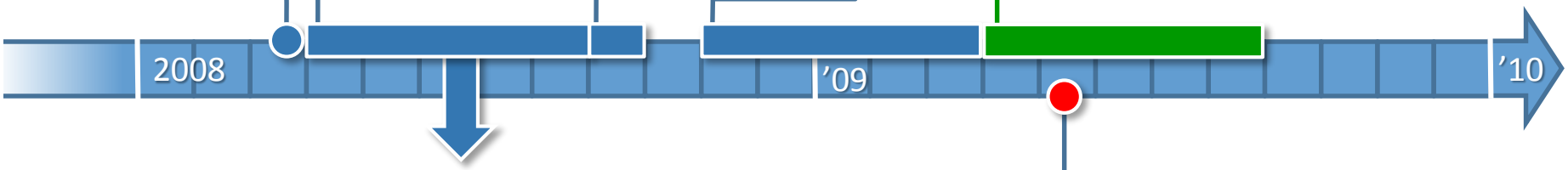


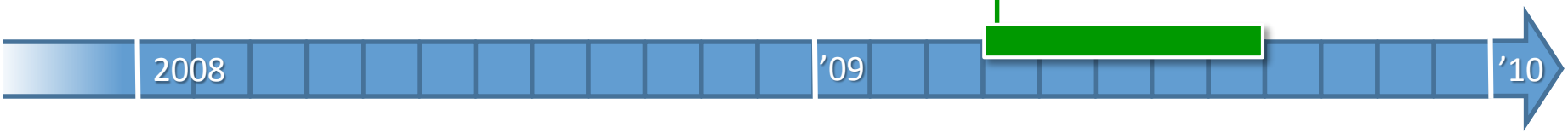
"The inventors . . . actually reduced to practice . . . on or before September 2008." (Paper 19, at 27)

May 15, 2009
PCT/GB2009/001263 ("Smith")

CONSTRUCTIVE REDUCTION TO PRACTICE

> Simultaneous to the aforementioned work, Honeywell was pursuing commercialization and development of 1234yf globally. A substantial portion of those efforts was developing a commercial plant and addressing the short, middle,

PO Response, Paper 19, at 40

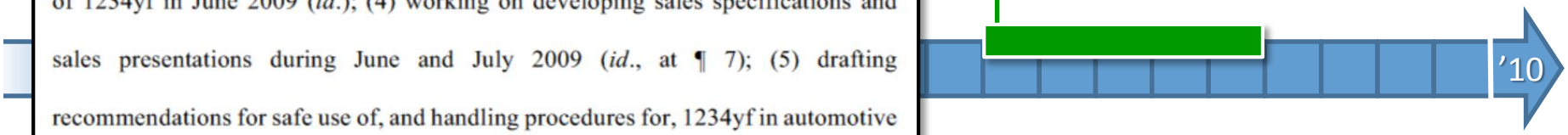


CONSTRUCTIVE REDUCTION TO PRACTICE

diligence.) The Declaration of Thomas Morris outlines representative activities that Honeywell was performing in furtherance of such development: (1) preparing and submitting a Forward Engineering Request Form in April 2009 to obtain approval to spend additional resources on engineering of the manufacturing plant for 1234yf (EX2036, ¶ 5); (2) establishing a path for a decision on a manufacturing plan for 1234yf, including pursuing converting an existing manufacturing facility that was owned by a major manufacturer of chemical products in May 2009 (*id.*, at ¶ 6); (3) visiting the potential site outside the U.S. to investigate options for the manufacture of 1234yf in June 2009 (*id.*); (4) working on developing sales specifications and sales presentations during June and July 2009 (*id.*, at ¶ 7); (5) drafting recommendations for safe use of, and handling procedures for, 1234yf in automotive air conditioning systems in August 2009 (*id.*, at ¶ 8); and (6) developed a working draft of a Whitepaper to brief Honeywell top executives regarding 1234yf and to obtain approval for business decisions related to the manufacture of 1234yf in September 2009 (*id.*, at ¶ 9).

PO Response, Paper 19, at 41

April – August 2009
Honeywell Pursuing Commercialization and Development of 1234yf Globally (Paper 19, at 40)



CONSTRUCTIVE REDUCTION TO PRACTICE

***Conception*: March 26, 2008**
Kopkalli E-mail (Paper 19, at 38; EX2007)

April – August 2008
Evaluate Utilizing a Caustic Reaction for the Dehydrofluorination Steps in Producing 1234yf (Paper 19, at 38)

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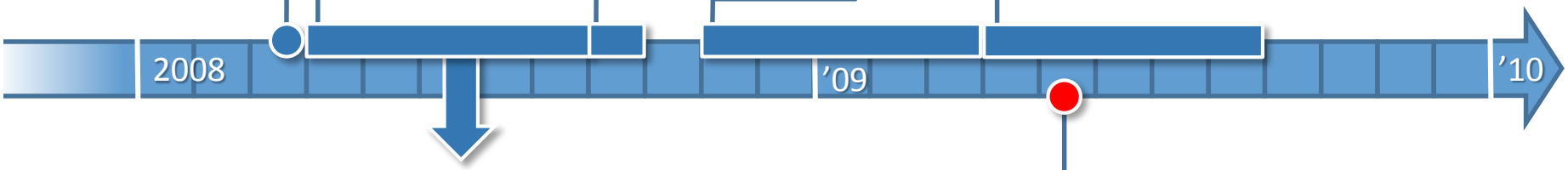
October 2008 – March 2009
Further Experiments and Analysis Were Performed in Order to Improve the Quality of the Experimental Data and to Complete Mass Balance (Paper 19, at 40)

April – August 2009
Honeywell Pursuing Commercialization and Development of 1234yf Globally (Paper 19, at 40)



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May 15, 2009
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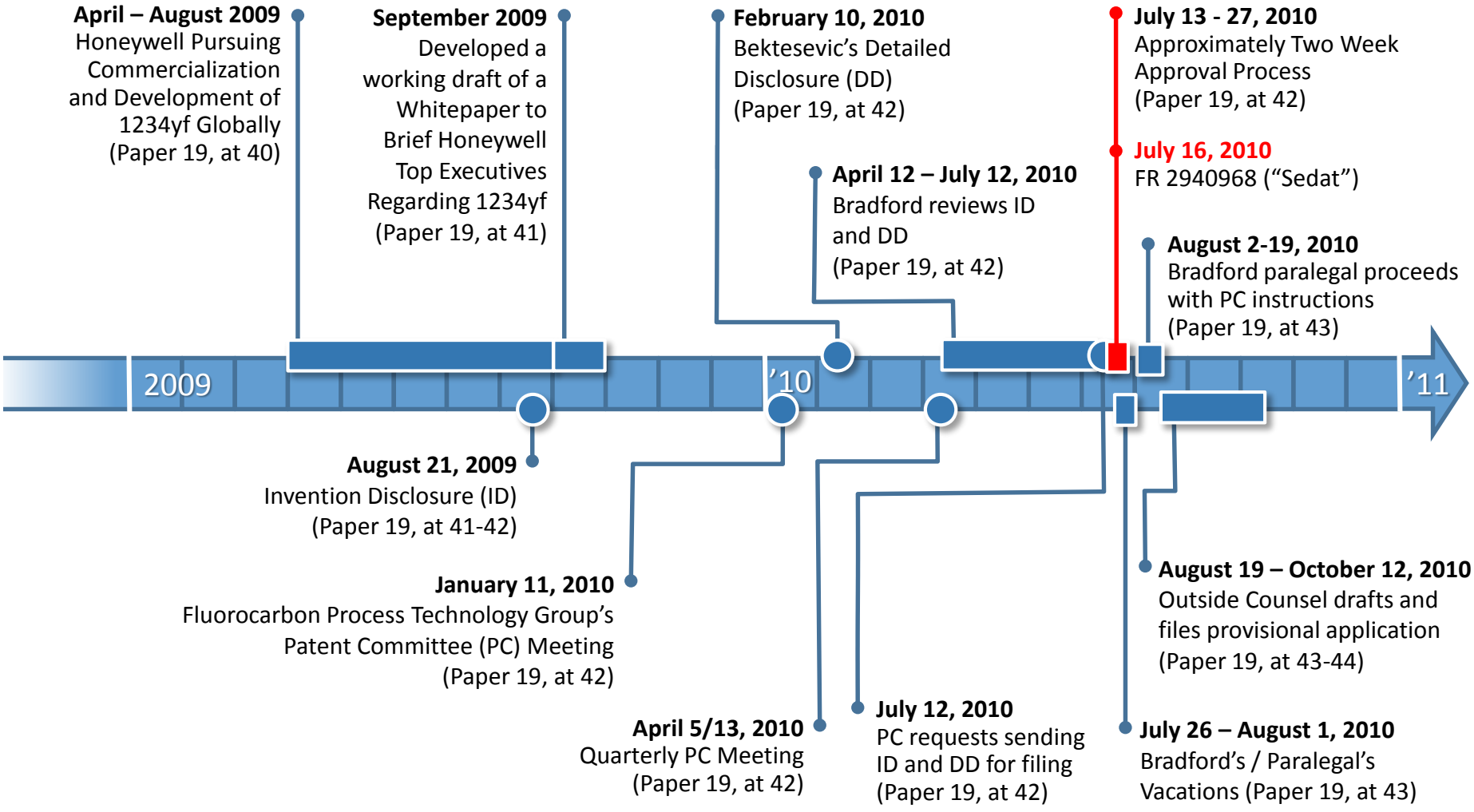

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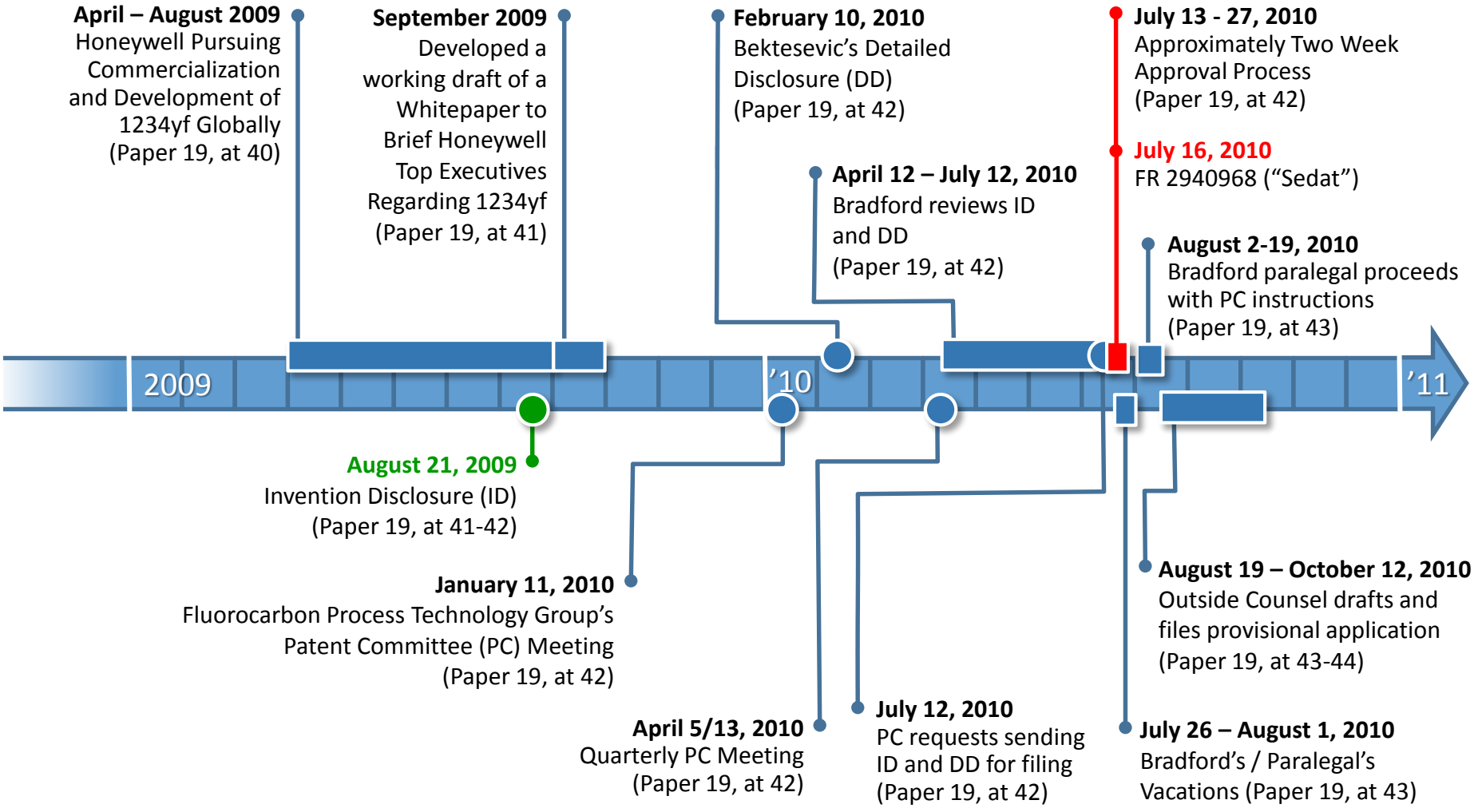


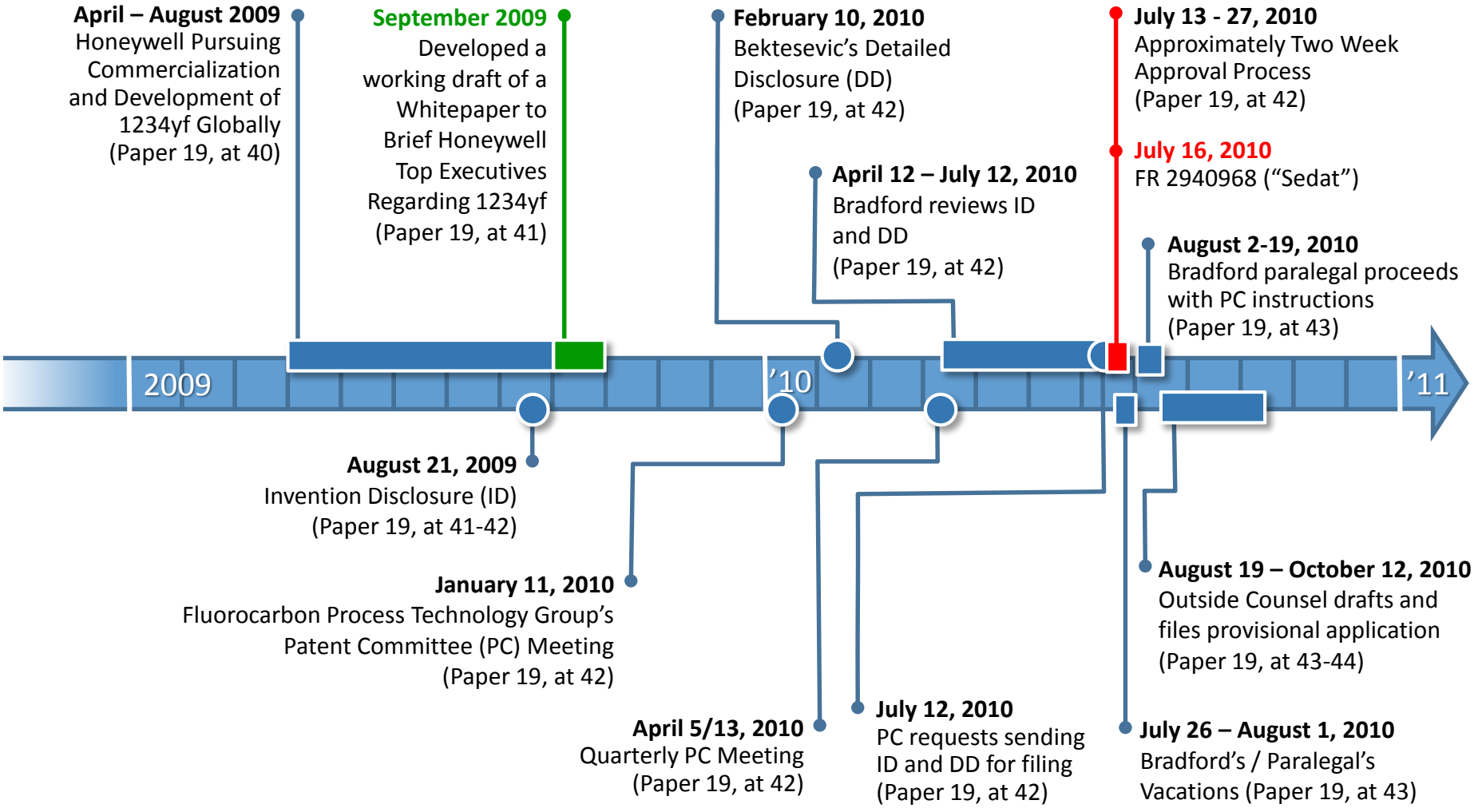
CONSTRUCTIVE REDUCTION TO PRACTICE

August 21, 2009
Invention Disclosure (ID)
(Paper 19, at 41-42)

> Consistent with Honeywell procedure, the invention disclosure form was submitted to the Honeywell Worldwide Invention Disclosure System ("WIDS") by the inventors on August 21, 2009 and was scheduled for review during the next patent committee ("PC") meeting. (EX2041, ¶¶ 7-8.) Thereafter, the Fluorocarbon Process Technology (4511) group held its next PC meeting on January 11, 2010. During this meeting, the PC requested that Dr. Bektesevic draft a detailed disclosure for the invention entitled "Integrated Continuous Process for Dehydrofluorination of 236ea and 245eb." (*Id.*, ¶ 10.) The detailed disclosure was received from Dr. Bektesevic on February 10, 2010. (*Id.*)

PO Response, Paper 19, at 41-42

CONSTRUCTIVE REDUCTION TO PRACTICE



CONSTRUCTIVE REDUCTION TO PRACTICE

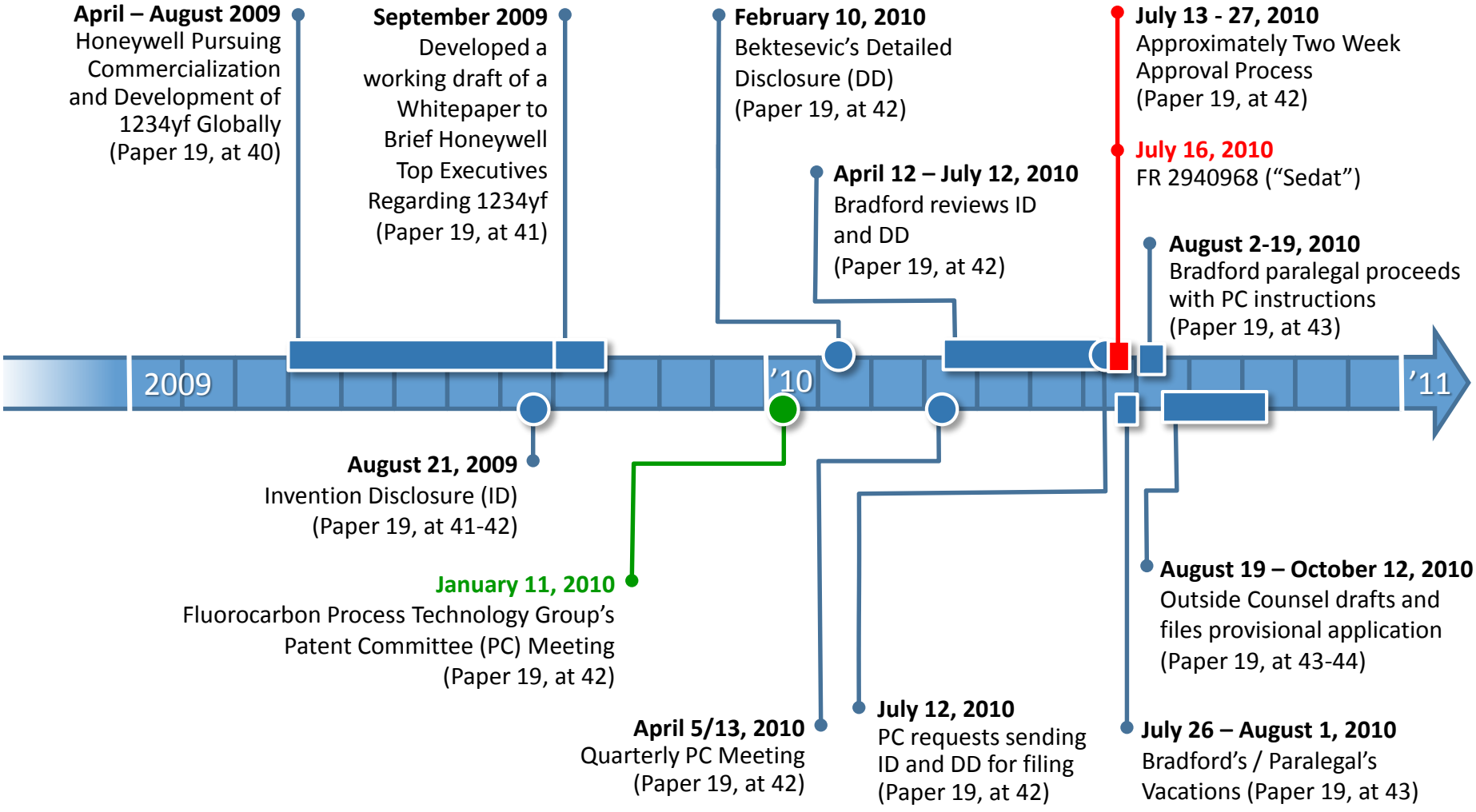
September 2009
Developed a working draft of a Whitepaper to Brief Honeywell Top Executives Regarding 1234yf (Paper 19, at 41)

2009

diligence.) The Declaration of Thomas Morris outlines representative activities that Honeywell was performing in furtherance of such development: (1) preparing and submitting a Forward Engineering Request Form in April 2009 to obtain approval to spend additional resources on engineering of the manufacturing plant for 1234yf (EX2036, ¶ 5); (2) establishing a path for a decision on a manufacturing plan for 1234yf, including pursuing converting an existing manufacturing facility that was owned by a major manufacturer of chemical products in May 2009 (*id.*, at ¶ 6); (3) visiting the potential site outside the U.S. to investigate options for the manufacture of 1234yf in June 2009 (*id.*); (4) working on developing sales specifications and sales presentations during June and July 2009 (*id.*, at ¶ 7); (5) drafting recommendations for safe use of, and handling procedures for, 1234yf in automotive air conditioning systems in August 2009 (*id.*, at ¶ 8); and (6) developed a working draft of a Whitepaper to brief Honeywell top executives regarding 1234yf and to obtain approval for business decisions related to the manufacture of 1234yf in September 2009 (*id.*, at ¶ 9).

PO Response, Paper 19, at 41

CONSTRUCTIVE REDUCTION TO PRACTICE



CONSTRUCTIVE REDUCTION TO PRACTICE

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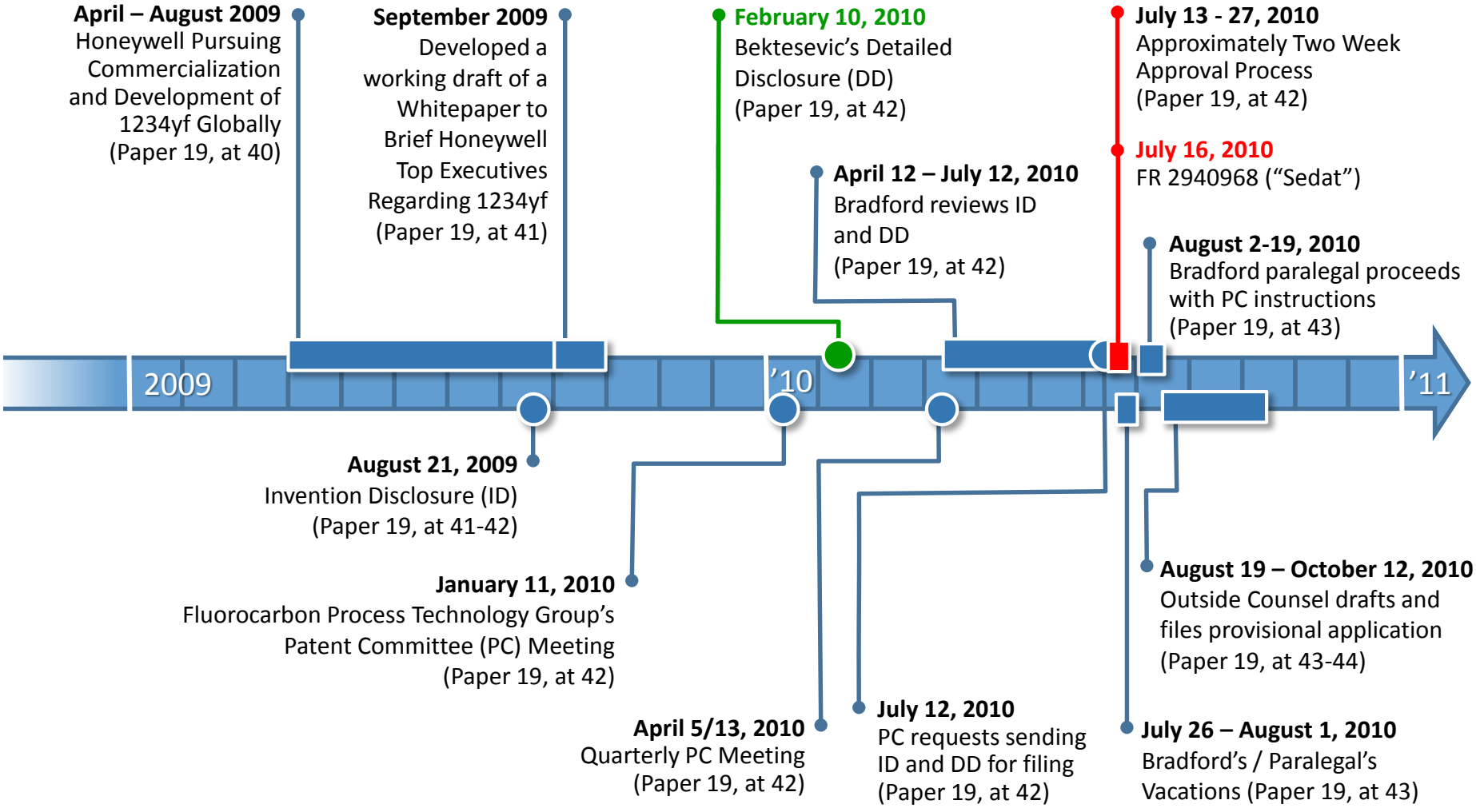
PO Response, Paper 19, at 41-42

2009

January 11, 2010
Fluorocarbon Process Technology Group's Patent Committee (PC) Meeting (Paper 19, at 42)

CONSTRUCTIVE REDUCTION TO PRACTICE



CONSTRUCTIVE REDUCTION TO PRACTICE

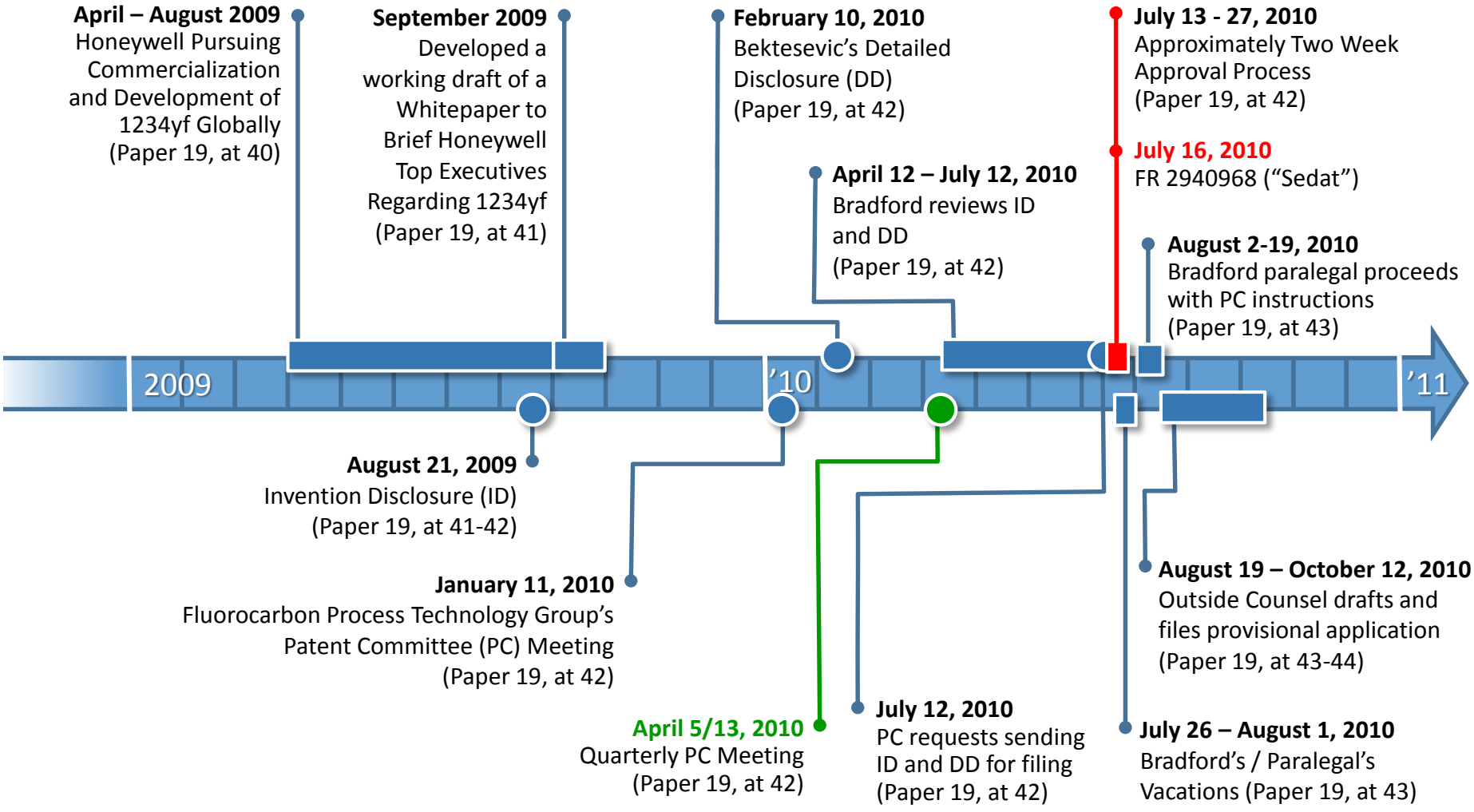
February 10, 2010
Bektesevic's Detailed Disclosure (DD)
(Paper 19, at 42)

Consistent with Honeywell procedure, the invention disclosure form was submitted to the Honeywell Worldwide Invention Disclosure System ("WIDS") by the inventors on August 21, 2009 and was scheduled for review during the next patent committee ("PC") meeting. (EX2041, ¶¶ 7-8.) Thereafter, the Fluorocarbon Process Technology (4511) group held its next PC meeting on January 11, 2010. During this meeting, the PC requested that Dr. Bektesevic draft a detailed disclosure for the invention entitled "Integrated Continuous Process for Dehydrofluorination of 236ea and 245eb." (*Id.*, ¶ 10.) The detailed disclosure was received from Dr. Bektesevic on February 10, 2010. (*Id.*)

'11

PO Response, Paper 19, at 41-42

CONSTRUCTIVE REDUCTION TO PRACTICE



CONSTRUCTIVE REDUCTION TO PRACTICE

At the subsequent quarterly PC meeting held on April 5 and 13, 2010, the PC requested that Mr. Bradford, in-house intellectual property counsel for Honeywell, review the invention disclosure form and the detailed disclosure prepared by Dr. Bektesevic with an eye to filing, as is customary for an intellectual property attorney at Honeywell. (*Id.*, ¶ 11.) Based on his review of the disclosures and consultation with business and technology leaders, Mr. Bradford's recommendation for the next quarterly PC meeting, held on July 12, 2010, was that Honeywell proceed with filing a provisional patent application for the invention. (*Id.*) At the July 12, 2010 PC meeting, the PC requested that Mr. Bradford send the invention disclosure form and detailed disclosure to outside counsel for filing. (*Id.*, ¶ 12; *see also* EX2042.)

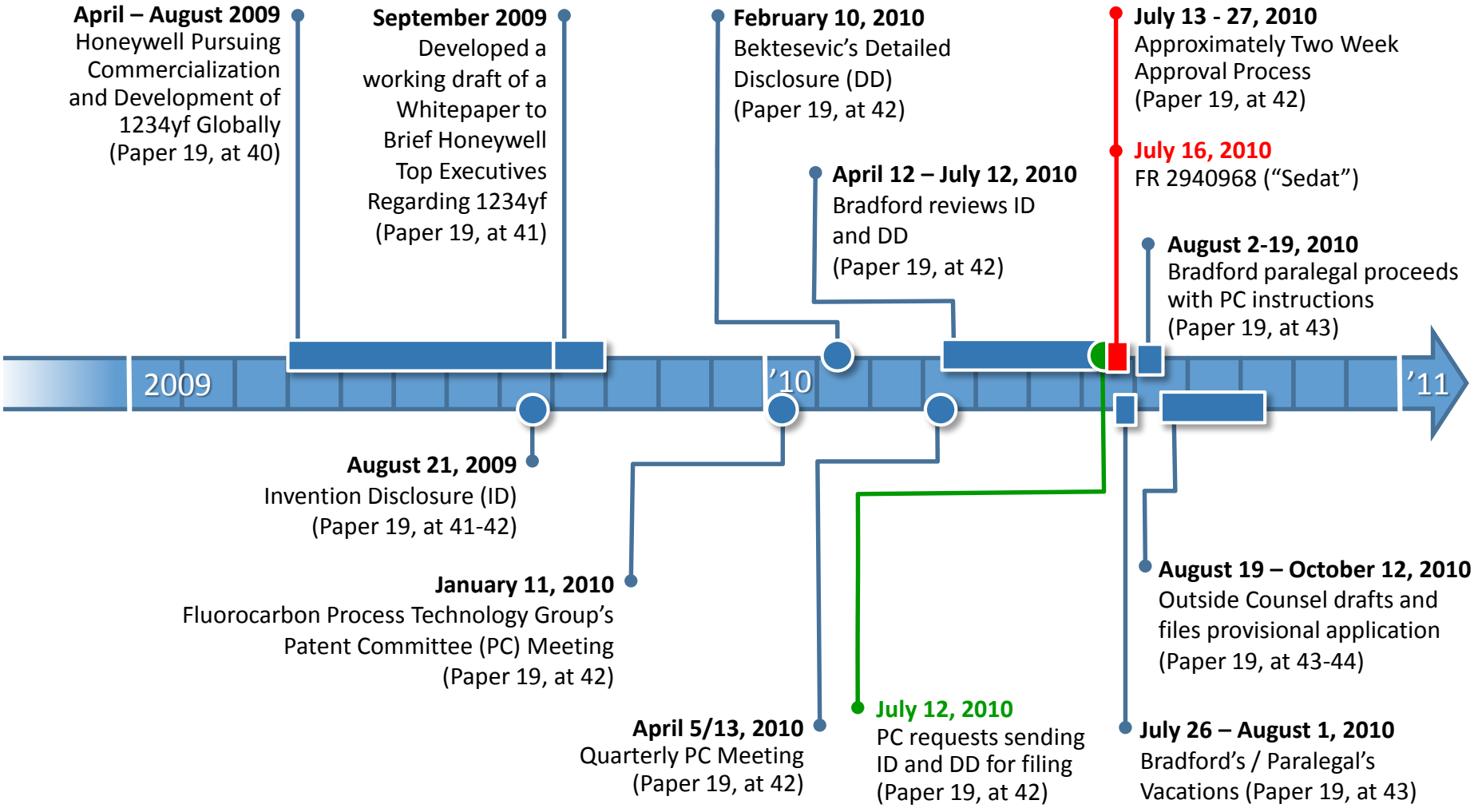
PO Response, Paper 19, at 42

2009

'11

April 5/13, 2010
Quarterly PC Meeting
(Paper 19, at 42)

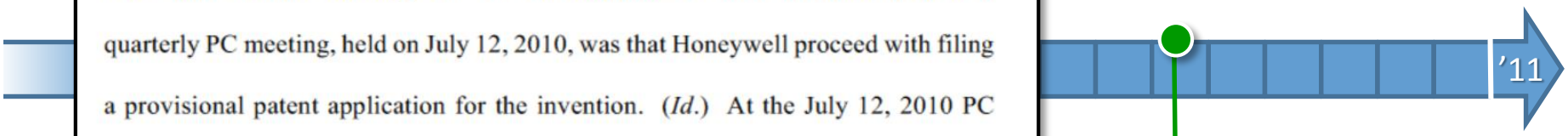
CONSTRUCTIVE REDUCTION TO PRACTICE



CONSTRUCTIVE REDUCTION TO PRACTICE

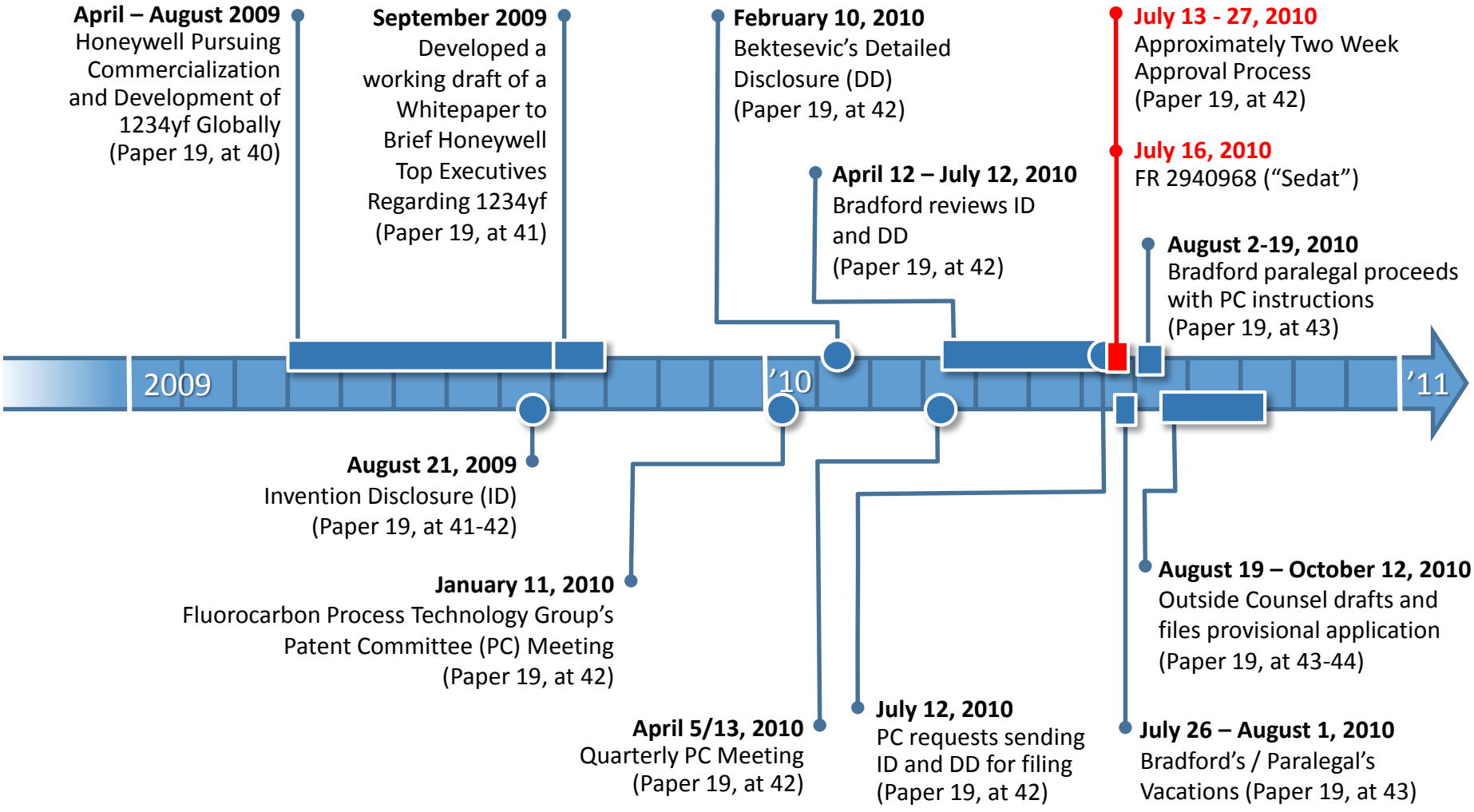
At the subsequent quarterly PC meeting held on April 5 and 13, 2010, the PC requested that Mr. Bradford, in-house intellectual property counsel for Honeywell, review the invention disclosure form and the detailed disclosure prepared by Dr. Bektesevic with an eye to filing, as is customary for an intellectual property attorney at Honeywell. (*Id.*, ¶ 11.) Based on his review of the disclosures and consultation with business and technology leaders, Mr. Bradford's recommendation for the next quarterly PC meeting, held on July 12, 2010, was that Honeywell proceed with filing a provisional patent application for the invention. (*Id.*) At the July 12, 2010 PC meeting, the PC requested that Mr. Bradford send the invention disclosure form and detailed disclosure to outside counsel for filing. (*Id.*, ¶ 12; *see also* EX2042.)

PO Response, Paper 19, at 42



July 12, 2010
PC requests sending ID and DD for filing (Paper 19, at 42)

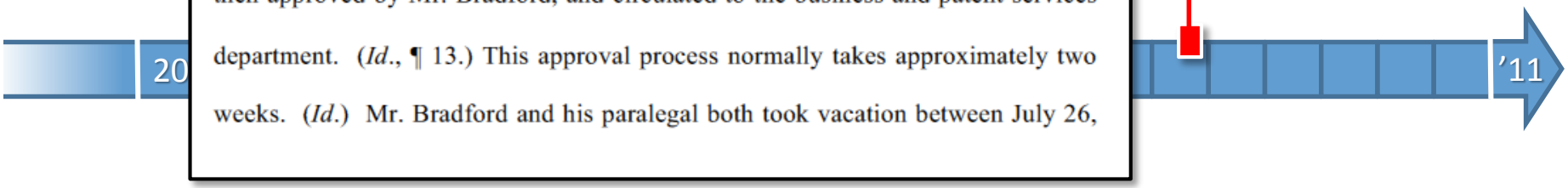
CONSTRUCTIVE REDUCTION TO PRACTICE



CONSTRUCTIVE REDUCTION TO PRACTICE

July 13 - 27, 2010
Approximately Two Week Approval Process
(Paper 19, at 42)

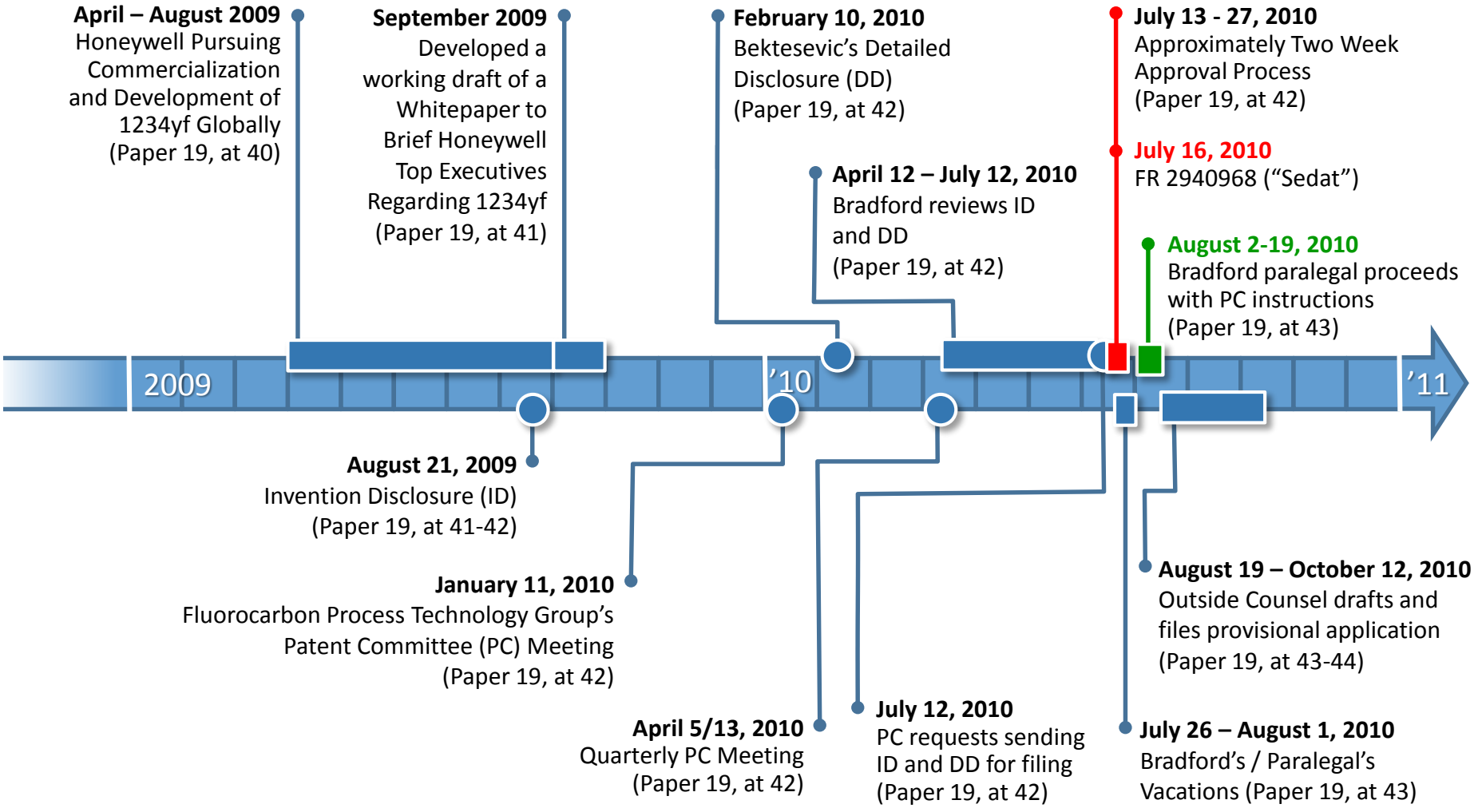
July 16, 2010
FR 2940968 ("Sedat")



> Mr. Bradford's paralegal prepared the minutes from the meeting, which were then approved by Mr. Bradford, and circulated to the business and patent services department. (*Id.*, ¶ 13.) This approval process normally takes approximately two weeks. (*Id.*) Mr. Bradford and his paralegal both took vacation between July 26,

PO Response, Paper 19, at 42-43

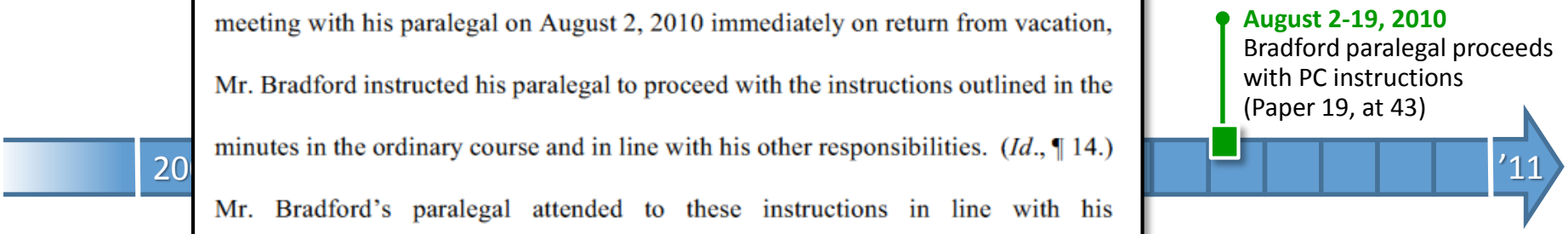
CONSTRUCTIVE REDUCTION TO PRACTICE



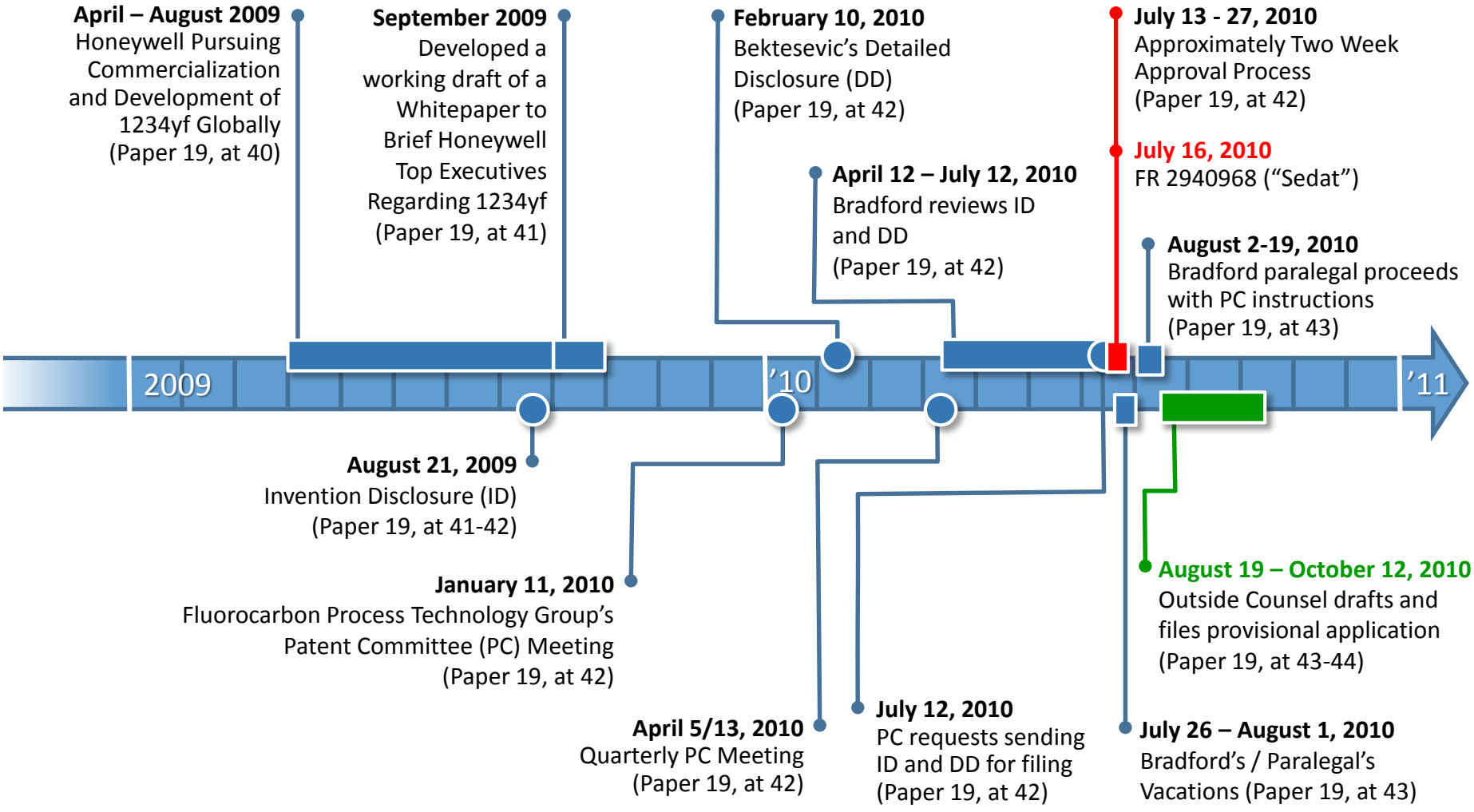
CONSTRUCTIVE REDUCTION TO PRACTICE

2010 and August 1, 2010, while the approval process was ongoing. After holding a meeting with his paralegal on August 2, 2010 immediately on return from vacation, Mr. Bradford instructed his paralegal to proceed with the instructions outlined in the minutes in the ordinary course and in line with his other responsibilities. (*Id.*, ¶ 14.) Mr. Bradford's paralegal attended to these instructions in line with his responsibilities, which included assisting Mr. Bradford with at least two technology transactions, and negotiating licensing and supply agreements. (*Id.*)

PO Response, Paper 19, at 43



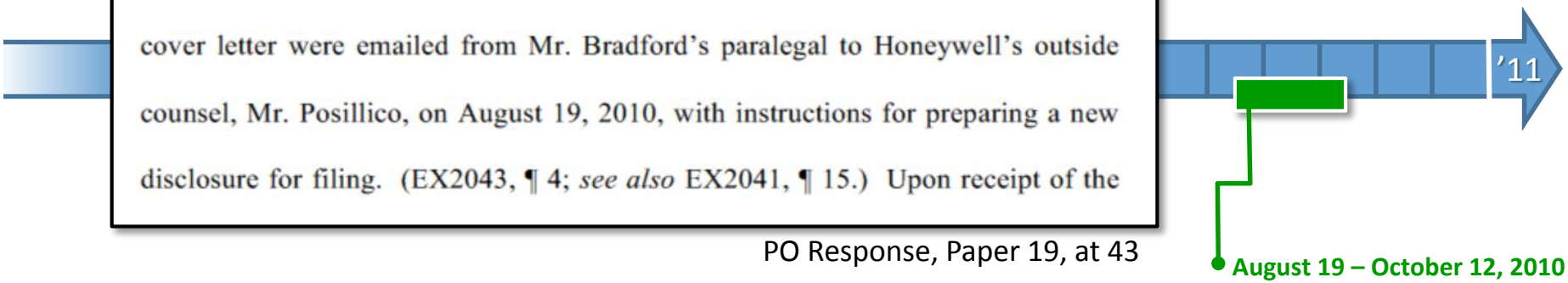
CONSTRUCTIVE REDUCTION TO PRACTICE



CONSTRUCTIVE REDUCTION TO PRACTICE

> Thereafter, the invention disclosure form, supporting documentation, and a cover letter were emailed from Mr. Bradford's paralegal to Honeywell's outside counsel, Mr. Posillico, on August 19, 2010, with instructions for preparing a new disclosure for filing. (EX2043, ¶ 4; *see also* EX2041, ¶ 15.) Upon receipt of the

PO Response, Paper 19, at 43

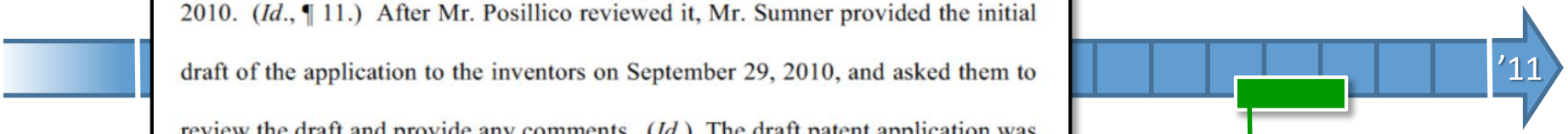


August 19 – October 12, 2010
Outside Counsel drafts and files provisional application (Paper 19, at 43-44)

CONSTRUCTIVE REDUCTION TO PRACTICE

Mr. Posillico's review of the time records indicate that Mr. Sumner picked up the drafting of the application in turn on September 22, 2010, and that the Fox attorneys devoted approximately 18 hours drafting the provisional application between September 22 and September 29, 2010. (*Id.*, ¶ 9.) Mr. Posillico confirms that Mr. Sumner had a full docket of matters at the time. (*Id.*, ¶ 10)

Mr. Sumner completed the first draft of the application on September 29, 2010. (*Id.*, ¶ 11.) After Mr. Posillico reviewed it, Mr. Sumner provided the initial draft of the application to the inventors on September 29, 2010, and asked them to review the draft and provide any comments. (*Id.*) The draft patent application was with the inventors from September 29, 2010 to October 12, 2010. This time period is not an unreasonable time period for the inventors to review the patent application, given the weekend and their general work requirements. (EX2006, at ¶ 59.) On October 12, 2010, Mr. Posillico received an email from Dr. Bektesevic which indicated that the inventors had discussed the application and that it was "OK to file invention [sic] as is." (*Id.*, ¶ 12.) The provisional application was filed that day.



August 19 – October 12, 2010
Outside Counsel drafts and files provisional application (Paper 19, at 43-44)

Sedat Is Distinguished

SEDAT IS DISTINGUISHED

- "A POSITA Would Not Rely on Sedat because it is Not Enabled." Paper 19, at 16.

Q. The language of Sedat goes on to discuss the dehydrofluorination reaction of step one may be carried at atmospheric pressure, but is preferred to work at a pressure above atmospheric pressure advantageously. This pressure is between 1.1 and 2.5 BarA.

A. Okay. So relatively low pressure, I would expect the -- are you asking me the 236 and 245?

Q. 236.

A. 236, I would expect 236 to be fairly volatile at that temperature, yes.

Q. And the same with 245?

A. Yes.

EX2004 at 148:19-149:6

The step (i) is generally carried out at a temperature such that the water formed during the dehydrofluorination reaction is removed, partly or completely, from the reaction medium via entrainment of the gas stream comprising the (hydro)fluoroolefin compound from the stirred reactor. This temperature is preferably between 80 and 180°C, advantageously between 125 and 180°C, and very particularly between 145 and 165°C.

The dehydrofluorination reaction of step (i) may be carried out at atmospheric pressure, but it is preferred to work at a pressure above atmospheric pressure. Advantageously, this pressure is between 1.1 and 2.5 bar.

EX1005 at 8-9

SEDAT IS DISTINGUISHED

- "Sedat is completely silent as to the presence of dissolved or entrained organics . . . ." Paper 19, at 21.
- "Sedat . . . teaches nothing regarding the separating of [dissolved or entrained] organics from the spent DHA or spent KOH in order to recover spent DHA or spent KOH." Paper 19, at 21.

Q. And there is no teaching of separating entrained or dissolved organics taught by Sedat?
MR. BEAUPRE: Objection to form.
A. There is no mention of entrained or dissolved organics in Sedat.

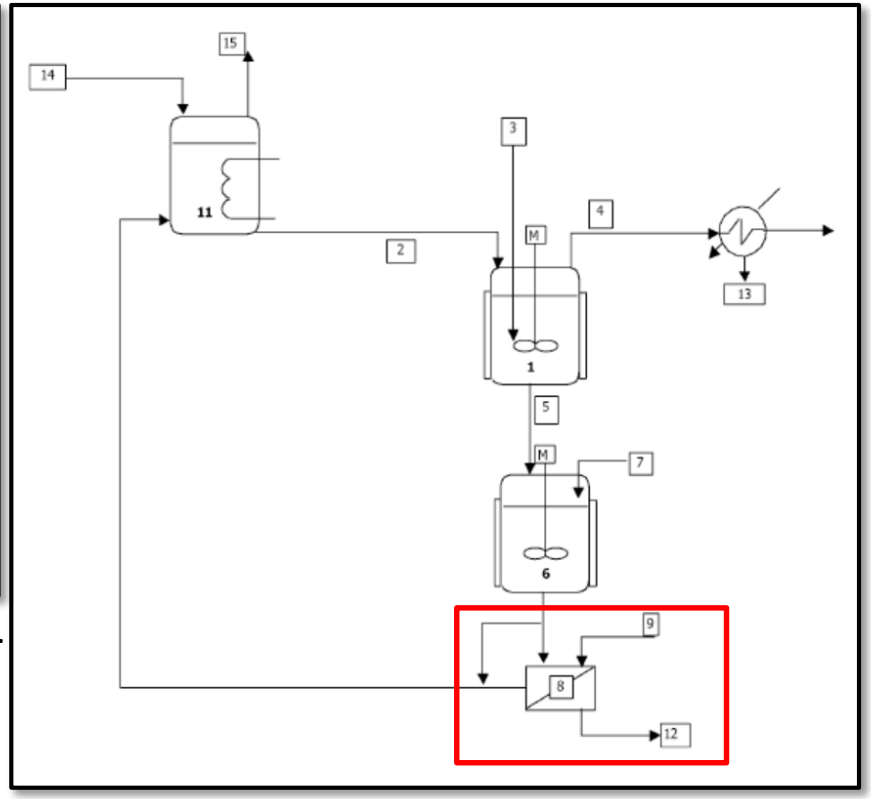
EX2004 at 166:12-16

SEDAT IS DISTINGUISHED

- "The only separation disclosed by Sedat appears to be the separation of calcium fluoride from converted KOH in filter (8) after regeneration of the spent KOH with calcium hydroxide in reactor (6) of Sedat's Figure 1." Paper 19, at 15.

Additionally, as further explained below, *Sedat* teaches recovery of spent KOH from the withdrawn dehydrohalogenation reaction medium as well as recovery of KOH regenerated from the metal salt by-product KF by: (1) introducing a suspension of calcium hydroxide to the reaction medium [*id.* at 10:17-19], which contains KOH and KF [*id.* at 8:13-17], resulting in the conversion of KF to KOH and the precipitation of CaF_2 [*id.* at 5:10-13], (2) separating CaF_2 from the reaction medium [*id.* at 10:21-23], and (3) collecting and concentrating an aqueous solution of KOH in an evaporator before recycling KOH back to the dehydrohalogenation reactor [*id.* at 11:1-4].

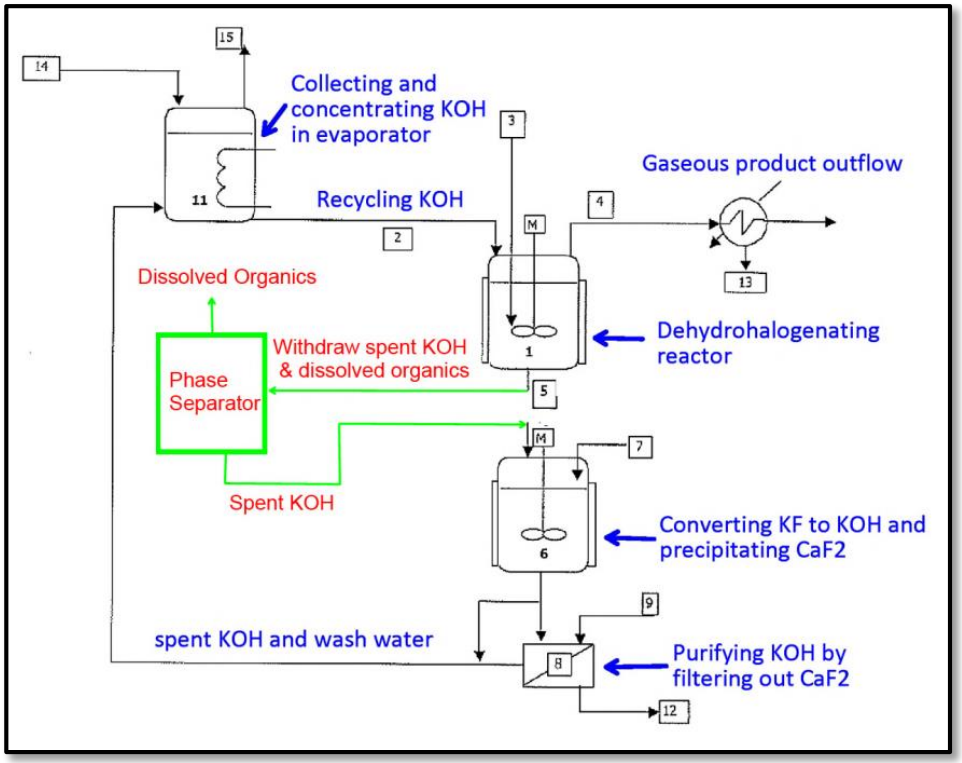
Paper 2, at 20.



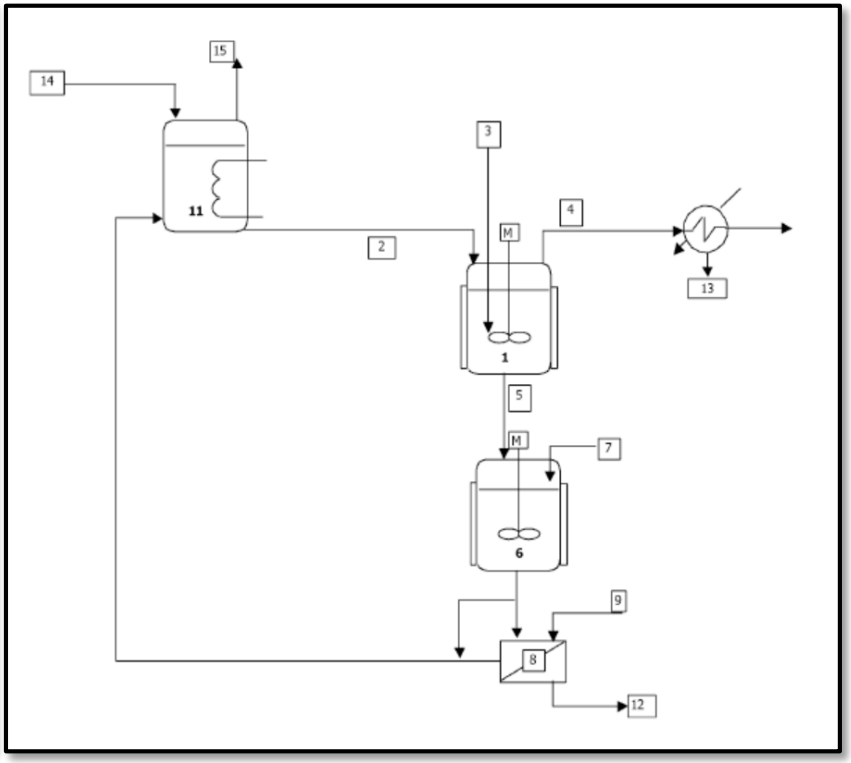
EX1004 at 12

SEDAT IS DISTINGUISHED

- "The only separation disclosed by Sedat appears to be the separation of calcium fluoride from converted KOH in filter (8) after regeneration of the spent KOH with calcium hydroxide in reactor (6) of Sedat's Figure 1." Paper 19, at 15.



Paper 40, at 4



EX1004 at 12

Smith Is Distinguished

SMITH IS DISTINGUISHED

- "Smith does not disclose the existence of, let alone any significant amount of . . . unreacted KOH." Paper 19, at 20.
- "Indeed, a review of Smith reveals that the disclosed process does not even contemplate the existence of a significant aqueous phase output from the dehydrofluorination reaction steps." Paper 19, at 22.

Q. Now, Smith doesn't disclose -- we discussed this yesterday -- Smith doesn't disclose the fact that unused or unreacted potassium hydroxide comes out of the reaction, is that correct?

MR. BEAUPRE: Same objection or objection, form.

A. He doesn't disclose what happens to any unreacted KOH.

Q. It doesn't disclose indeed that there even is unreacted KOH, correct?

MR. BEAUPRE: Objection, form.

A. He doesn't discuss whether there is or is not any unreacted KOH.

EX2005 at 208:15-209:2

Smith and Sedat
Are Distinguished

SMITH IN COMBINATION WITH SEDAT IS DISTINGUISHED

- "[Smith and Sedat are] completely silent as to the existence of spent KOH and any separation of dissolved organics from spent DHA or spent KOH in order to recover spent DHA or spent KOH." Paper 19, at 22.

Q. Now, Smith doesn't disclose -- we discussed this yesterday -- Smith doesn't disclose the fact that unused or unreacted potassium hydroxide comes out of the reaction, is that correct?

MR. BEAUPRE: Same objection or objection, form.

A. He doesn't disclose what happens to any unreacted KOH.

Q. It doesn't disclose indeed that there even is unreacted KOH, correct?

MR. BEAUPRE: Objection, form.

A. He doesn't discuss whether there is or is not any unreacted KOH.

EX2005 at 208:15-209:2

Q. And there is no teaching of separating entrained or dissolved organics taught by Sedat?

MR. BEAUPRE: Objection to form.

A. There is no mention of entrained or dissolved organics in Sedat.

EX2004 at 166:12-16

SMITH IN COMBINATION WITH SEDAT IS DISTINGUISHED

- "Smith does not disclose the existence of, let alone any significant amount of, unreacted DHA or unreacted KOH." Paper 19, at 20.

"Q. And Smith does not discuss anything having to do with spent KOH, correct?

A. No, I can't find anywhere where he does.

Q. And so there would be no mention of any desire to separate, purify, concentrate, regenerate or recycle spent KOH mentioned in Smith, correct?

A. I don't recall any mention of that specifically.

Q. And similarly, Smith also doesn't reference KF as a byproduct, does he?

A. I don't recall that.

Q. And, therefore, he doesn't mention ·regeneration using any KF?

A. No."

EX2004 at 145:10-147:5

SMITH IN COMBINATION WITH SEDAT IS DISTINGUISHED

- "[T]he POSITA would have had no . . . motivation to combine Sedat with any other reference . . . ." Paper 19, at 3.

Q. Did you ever refer to it as a rule of thumb when you were at DuPont?
MR. BEAUPRE: Objection to form.
A. I don't think I had to. It was pretty obvious when we were doing any of these process development programs for any chemical to develop the lowest cost process. And one develops the lowest cost process by recovering, recycling anything that makes sense.

EX2004 at 71:5-13

135. Petitioner further argues that "[r]ecovering and recycling all valuable reactants is a basic 'rule of thumb' in chemical process design." (Petition at p. 39, citing EX1006 at p. 116-117 and EX1002 at ¶ 100.). This is a narrow and incorrect assertion. Instead, the rule of thumb that I follow requires that you extract as much value out of the process, based on economics. In practice, this can mean much more or something entirely different than recovery and recycle. Instead, in my experience, the overall project and process economics need to be considered. Indeed, capital investment, fixed and variable costs, need to be evaluated in order to decide whether it necessary or practical to recover or recycle products of a reaction. It may be more economical to dispose or even sell materials to maximize value, instead of recovering or recycling.

EX2002, ¶ 135

Masayuki Is Distinguished

MASAYUKI IS DISTINGUISHED

- "[The process in Masayuki] fails to separate the entrained organic components from the aqueous phase in order to recover spent KOH." IPR2015-00916, Paper 21, at 17.

> Note that conventionally precipitation of dissolved alkali metal chloride accompanying the evaporation of this water as well as precipitation of polymer due to small amounts of 3,4-dichloro-butene-1, chloroprene and polymer contained in the aqueous phase are problematic. Though it is possible to dissolve precipitants using water, as is typically done to avoid trouble arising from the precipitation of alkali metal chloride, trouble caused by polymer requires that the apparatus used be dismantled. As a result, concentration of alkali metal hydroxide aqueous solution containing polymerizable organics, as is performed in the present invention, has not been previously carried out.

IPR2015-00916, EX1005 at 9:15-27

MASAYUKI IS DISTINGUISHED

- "[The process in Masayuki] fails to separate the entrained organic components from the aqueous phase in order to recover spent KOH." IPR2015-00916, Paper 21, at 17.

was performed at 60°C and 225 mmHg (abs). The aqueous phase was extracted via Conduit 4 such that the level
35 of Reactor 19 remained constant and the slightly 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. The aqueous phase containing solid sodium chloride

IPR2015-00916, EX1005 at 10:33-11:1.

91. Claim 30 depends from claim 21, and claim 41 depends from claim 36. Both claims recite "…wherein the spent KOH is purified using at least one separation method." *Masayuki* discloses separating aqueous spent alkali metal hydroxide from a lipid phase (e.g., organic catalyst) using a decanter, thereby purifying the spent alkali metal hydroxide. Ex. 1005 at 10:33-37 and Figure. A

IPR2015-00916, EX1002, ¶ 91

MASAYUKI IS DISTINGUISHED

- "[The process in Masayuki] fails to separate the entrained organic components from the aqueous phase in order to recover spent KOH." IPR2015-00916, Paper 21, at 17.

Q. Smith. Smith in combination with either of them, either Sedat or Masayuki, did you consider, so we don't have to go back and reask the question a second time, did you consider the concentrations of KOH in opining as to Sedat in combination with Smith or Masayuki in combination with Smith, with either of them?

MR. BEAUPRE: Objection, form.

A. Not specifically, I was looking at some method of enabling the preparation of the haloalkanes and that comes from Smith, and I was looking at a method of dehydrohalogenating those alkanes to 1234,

and that was the reason for the Sedat and Masayuki references over Smith. And in looking at those references, I had a high degree of confidence that combining those references would enable me to develop a process. One of the steps in that development would have been looking at concentrations.

Q. But you just didn't at this time look at the concentrations?

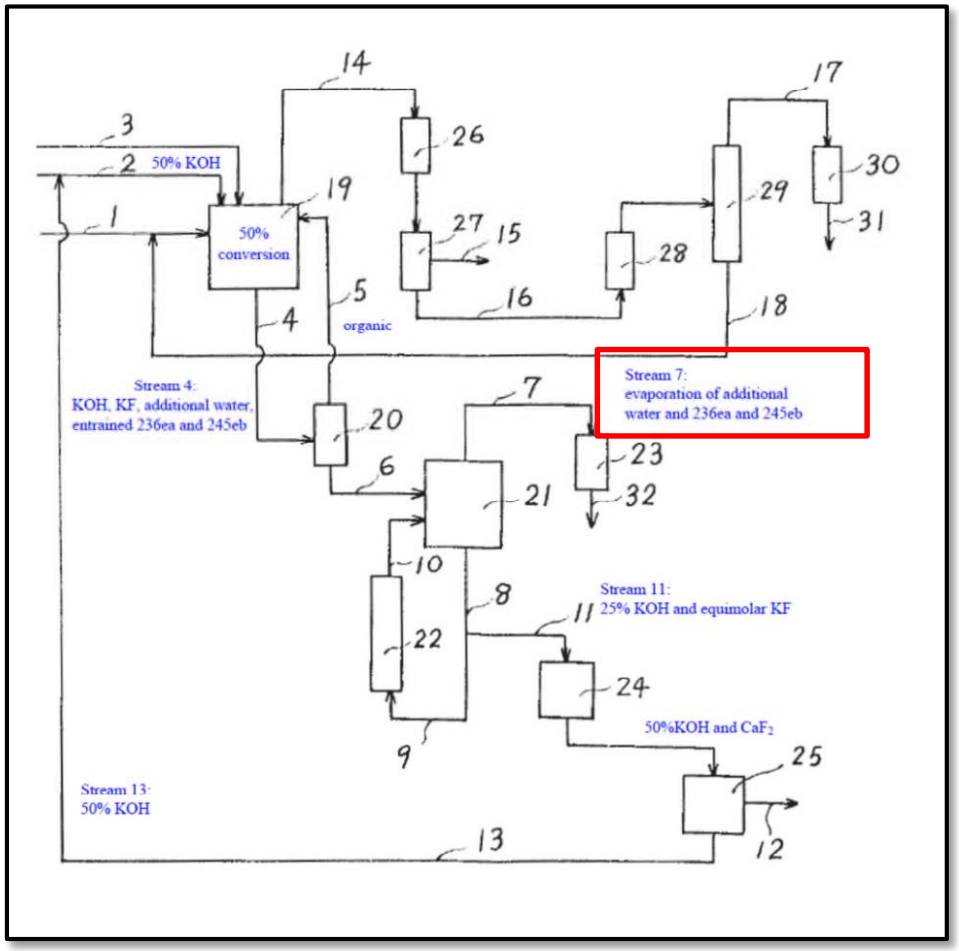
MR. BEAUPRE: Objection, form.

A. Not as a POSITA in 2010. I was just looking for prior art references.

IPR2015-00916, EX2004 at 132:14-133:11

MASAYUKI IS DISTINGUISHED

- "[The process in Masayuki] fails to separate the entrained organic components from the aqueous phase in order to recover spent KOH." IPR2015-00916, Paper 21, at 17.
- "…236ea and 245eb could be evaporated out of the enricher…" IPR2015-00916, Paper 42, at 9.



IPR2015-00916, Paper 42, at 12

MASAYUKI IS DISTINGUISHED

- "Masayuki only . . . provides an operable process with regards to sodium hydroxide (NaOH) . . . . [and] fails when used with potassium hydroxide (KOH)." IPR2015-00916, Paper 21, at 29.

> 130. Accordingly, the art of record specifically relating to KOH teaches an inoperable process when combined as asserted by Petitioner.

IPR2015-00916, EX2002, ¶ 130

The Combination Is Distinguished

SMITH IN COMBINATION WITH MASAYUKI AND HARRISON IS DISTINGUISHED

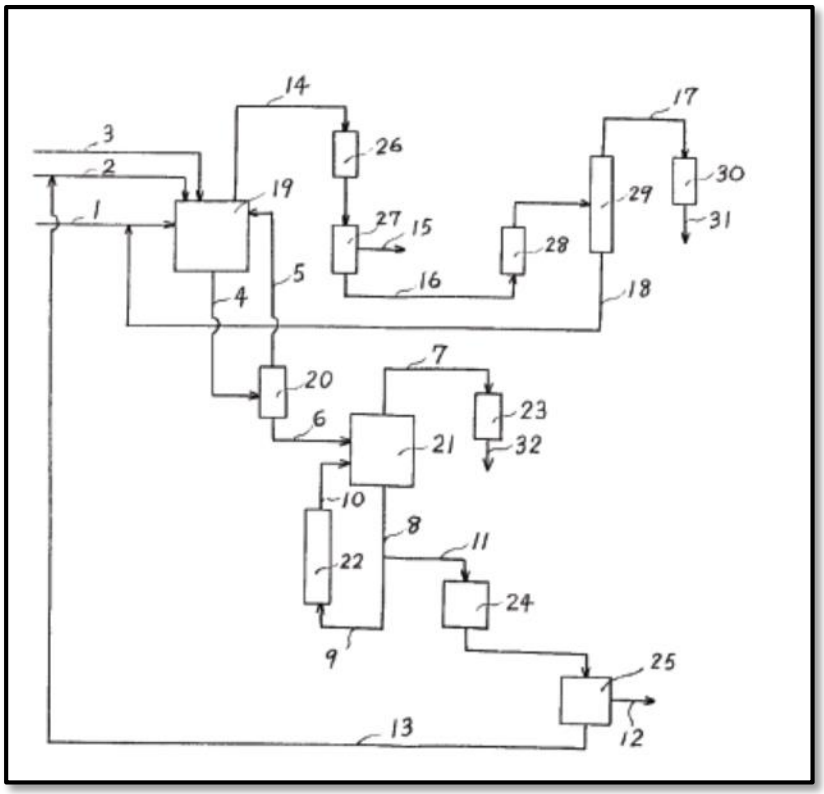
- "[Petitioner] fails to prove that a POSITA would have been motivated to look beyond Smith." IPR2015-00916, Paper 21, at 48.
- "As with Harrison, Smith is completely silent as to the existence of spent KOH and any separation of dissolved organics from spent KOH in order to recover spent KOH." IPR2015-00916, Paper 21, at 22.
- ". . . , Masayuki only purportedly provides an operable process with regards to sodium hydroxide (NaOH)" IPR2015-00916, Paper 21, at 29.

SMITH IN COMBINATION WITH MASAYUKI AND HARRISON IS DISTINGUISHED

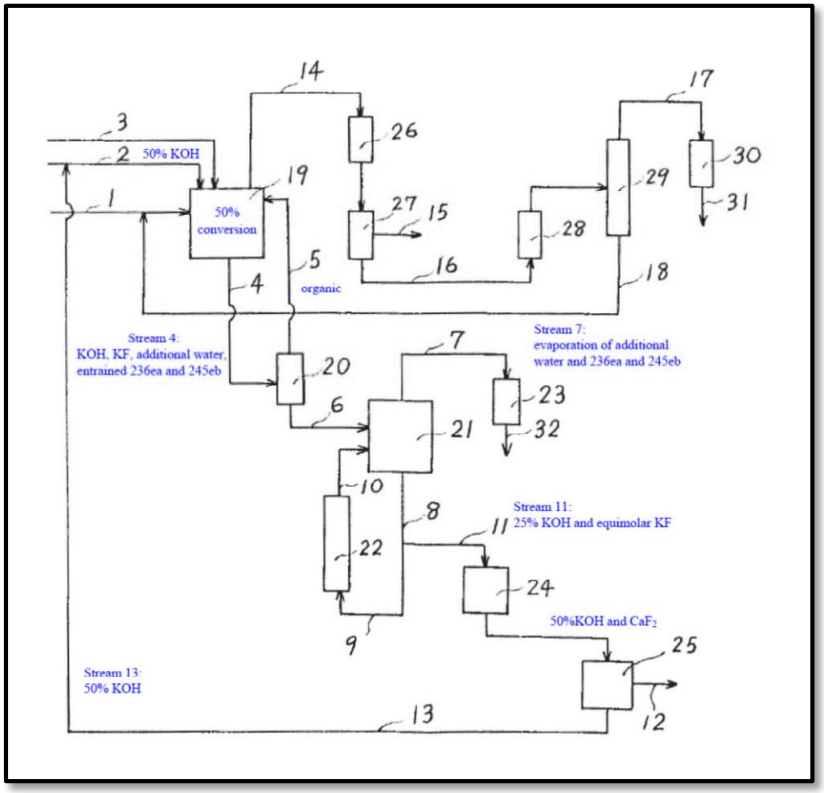
- "[T]he addition of the slaked lime from Harrison to the slurry tank of Masayuki while utilizing KOH . . . would render the process inoperable . . ." IPR2015-00916, Paper 21, at 27.
- "The POSITA reading Harrison would understand that the concentrations of both potassium hydroxide and potassium fluoride must be sufficiently low, and the amount of water must be sufficiently high (i.e., 80% water or higher), for the recited reaction to occur." IPR2015-00916, Paper 21, at 30.
- "Dr. Manzer made it clear that he did not perform any modeling or consider any concentration of materials." IPR2015-00916, Paper 21, at 27.

SMITH IN COMBINATION WITH MASAYUKI AND HARRISON IS DISTINGUISHED

- "[Petitioner's expert] admit[ted] that the addition of Harrison would result in disruption to the combination." IPR2015-00916, Paper 21, at 33.



IPR2015-00916, EX1005 at 13



IPR2015-00916, Paper 42, at 12