

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

PHARAOH ENERGY SERVICES, LLC
Petitioner

v.

FLEX-CHEM HOLDING COMPANY, LLC
Patent Owner

Case No. IPR2024-00822
Patent No. 10,697,282
Issued: June 30, 2020
Application No.: 16/226,464
Filed: December 19, 2018

Title: METHOD FOR REMEDIATION OF SUBTERRANEAN FORMED
METAL-POLYMER COMPLEXES USING A METAL-COMPLEXING
AGENT

**PETITION FOR *INTER PARTES* REVIEW
OF U.S. PATENT NO. 10,697,282
(CLAIMS 1–22)**

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EXHIBIT LIST

<u>Exhibit No.</u>	<u>Description</u>
1001	U.S. Patent No. 10,697,282
1002	Prosecution History of U.S. Patent No.10,697,282
1003	Expert Declaration of Dr. Jeffrey H. Harwell
1004	United States Patent No. 9,410,405 B2 to Ayers et al.
1005	United States Patent No. 6,436,880 B1 to Frenier
1006	Proof of Service of Summons and Complaint, <i>Flex-Chem Holding Company LLC v. Pharaoh Energy Services, LLC</i> , U.S. District Court for the Western District of Oklahoma, Case No. 5:23-cv-00316-JD
1007	U.S. Patent Application No. 14/525,038 filed October 27, 2014
1008	U.S. Provisional Application 61/895,873 filed October 25, 2013
1009	U.S. Provisional Application 61/898,107 filed October 31, 2013

1010	Flex-Chem Claim Construction Brief, <i>Flex-Chem Holding Company LLC v. Pharaoh Energy Services, LLC</i> , U.S. District Court for the Western District of Oklahoma, Case No. 5:23-cv-00316-JD
1011	U.S. Pat. No. 6,593,279 to Von Krosigk et al., entitled “Acid based micro-emulsions,” issued July 15, 2003.
1012	U.S. Pat. No. 5,203,413 to Zerhboub, entitled “Product and Process for Acid Diversion in the Treatment of Subterranean Formations,” issued April 20, 1993
1013	U.S. Patent. No. 1,877,504A to Grebe, entitled “Treatment of Deep Wells,” issued September 13, 1932
1014	Borchardt, <i>Chemicals Used in Oil Field Operations</i> , 1989 (clean copy obtained April 16, 2024)
1015	Schlumberger Online Glossary, (screenshots assembled April 16, 2014)

I. INTRODUCTION

Pharaoh Energy Services, LLC (“Petitioner”) petitions for *inter partes* review under 35 U.S.C. §§ 311–319 and 37 C.F.R. § 42.100 *et seq.* of all claims (1–22) of U.S. Patent No. 10,697,282 (“the ’282 Patent”) (Ex. 1001) (the “Challenged Claims”). This Petition sets forth two grounds for invalidity, which are all likely to prevail based on the accompanying evidence, and as such, this Petition should be granted and the Challenged Claims canceled.

II. MANDATORY NOTICES UNDER 37 C.F.R. § 42.8

A. Real Parties-in-Interest Under 37 C.F.R. § 42.8(b)(1)

Other than Petitioner there are no other real parties-in-interest.

B. Related Matters Under 37 C.F.R. § 42.8(b)(2)

The ’282 Patent is the subject of a patent infringement lawsuit brought by the Patent Owner Flex-Chem Holding Company, LLC (“Patent Owner”) and its purported exclusive licensee Flex-Chem Services Corp, against Petitioner in U.S. District Court for the Western District of Oklahoma, Case No. 5:23-cv-00316-JD (“Related Litigation”). Petitioner was served a copy of the Summons and Complaint on April 18, 2023. (A copy of the Proof of Service of the Summons and Complaint filed by Patent Owner in the Related Litigation is attached as Exhibit 1006. On April 17, 2024, Petitioner filed a Petition for *Inter Partes* Review of U.S. Patent No. 9,944,843, which is also a patent-in-suit in the Related Litigation.

C. Lead and Back-Up Counsel Under 37 C.F.R. § 42.8(b)(3)

Petitioner provides the following designation of counsel:

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D. Service Information Under 37 C.F.R. § 42.8(b)(4)

Petitioner may be served at the addresses shown above. Petitioner consents to electronic service: iprfilings@ryanwhaley.com. Pursuant to 37 C.F.R. § 42.10(b), filed with this Petition is a Power of Attorney.

III. PAYMENT OF FEES PURSUANT TO 37 C.F.R. §§ 42.15 AND 42.103

Per 37 C.F.R. §42.103, the Office is authorized to charge \$43,750 and any additional fees to Deposit Account No. 606468.

IV. GROUND FOR STANDING PURSUANT TO 37 C.F.R. § 42.104(A)

Petitioner certifies that the '282 Patent is available for *inter partes* review and that Petitioner is not barred or estopped from requesting *inter partes* review challenging the claims of the '282 Patent on the grounds identified herein. Neither Petitioner, nor any party in privity with Petitioner: (1) has filed a civil action challenging the validity of the Challenged Claims of the '282 Patent; (ii) has been served a complaint alleging infringement of the '282 Patent more than one year prior to the present date; or (iii) is estopped from challenging the claims on the grounds identified in the Petition. The Challenged Claims also have not been the subject of a prior *inter partes* review or a finally concluded district court litigation.

V. IDENTIFICATION OF CHALLENGE PURSUANT TO 37 C.F.R. § 42.104(B) AND STATEMENT OF RELIEF REQUESTED

A. Claims for Which Review is Requested

Petitioner respectfully requests review and cancellation of certain claims 1–22 of the '282 Patent as unpatentable.

B. Citation of Prior Art

United States Patent No. 9,410,405 B2 to Ayers et al. (“Ayers”) (Ex. 1004). Ayers was filed on May 29, 2014 as U.S. Patent Application No. 14/361,685 as a National Stage Entry of PCT/US2011/062697 filed on November 30, 2011, and issued as U.S. Patent No. 9,410,405 B2 on August 9, 2016. Ayers qualifies as prior art under 5 U.S.C. § 102(a)(2).

United States Patent No. 6,436,880 B1 to Frenier (“**Frenier**”) (Ex. 1004). Frenier was filed on May 3, 2000 as U.S. Patent Application No. 09/563,557 and issued as U.S. Patent No. 6,436,880 B1 on August 20, 2002. Frenier qualifies as prior art under 35 U.S.C. § 102(a)(1).

C. Statutory Grounds of Challenge

Petitioner asks the Board to institute an inter partes review under 35 U.S.C. § 314(a) as there is a reasonable likelihood that at least one or more of the Challenged Claims should be canceled as unpatentable on the following evidence and grounds:

Ground	Basis	Claims	References
1.	§ 102	1–3, 11–14, and 19	Ayers
2.	§ 103	1–22	Ayers alone and/or in view of Frenier

For each of these grounds, in Section VII, the Petition demonstrates at least a reasonable likelihood, based on a preponderance of the evidence (35 U.S.C. § 316(e)), that Petitioner will prevail in establishing that at least one or more of the Challenged Claims are unpatentable.

D. This Petition Presents Art and Arguments Not Previously Considered by the Office

The Office has not previously considered the prior art, the arguments presented herein, or any substantially similar art or arguments regarding the '282 Patent. Petitioner relies on the following patents and printed publications:

E. There Have Been No Other Petitions Filed

None of the Challenged Claims have been the subject of any other Petition for *inter partes* review.

F. Person of Ordinary Skill in the Art

Patent Owner has described a person of ordinary skill in the art (“POSITA”) at the time of the alleged invention of the ‘282 Patent had at least a bachelor’s degree in chemistry, chemical engineering, petroleum engineering, geology, or the equivalent, and at least two years of experience in oil and natural gas well treatments, or an equivalent amount of relevant work and/or educational experience. For the purposes of this Petition, Petitioner is prepared to adopt the level of ordinary skill in the art set forth by Patent Owner.

Dr. Jeffrey H. Harwell, PH.D., meets these criteria and concurrently submits a Declaration (Ex. 1003) under 37 C.F.R. § 1.68 in support of this petition.

G. Supporting Evidence Under 37 C.F.R. § 42.104(b)(5)

An Exhibit List identifying the exhibits relied upon is included immediately following the Table of Contents, *supra*.

VI. SUMMARY OF THE ’282 PATENT

A. Effective Filing Date of the ’282 Patent

The ’282 Patent issued on June 30, 2020, from U.S Patent Application No. 16/226,464, filed December 19, 2018 (“the ’464 Application”). The ’464

Application is a continuation of U.S. Patent Application No. 14/525,038 (“the ’038 Application”)(Ex. 1008) filed on October 27, 2014, now U.S. Patent No. 10,202,834. The ’038 Application claims priority to U.S. Provisional Application No. 61/895,873 filed October 25, 2013 (“the ’873 Provisional”) (Ex. 1009) and to U.S. Provisional Application No. 61/898,107 filed October 31, 2013 (“the ’107 Provisional”) (Ex. 1010). Therefore, solely for the purposes of this filing, the earliest effective filing date of the ’282 Patent is October 25, 2013.

B. The Specification

The ’282 Patent describes “[f]ormulations and methods for remediating subterranean-formed metal-polymer complexes, such as polyacrylamide or other gelable polymers that crosslink with metal ions, from wells in subterranean formations, including wells in nano-darcy shale formations.” Ex. 1001 at 2:20–24.¹ The disclosed method includes injecting a metal-polymer complex remediation mixture containing a metal complexing agent into a subterranean formation adjacent to a well a pressure below the fracture pressure of the formation. *Id.* at 2:25–29. After allowing sufficient contact time, the mixture is pumped from the subsurface. *Id.* at 2:29–30. According to the ’282 Patent, application of this method results in the remediation of polymer-containing, subterranean-formed metal-polymer

¹ The notation N:XX–YY refers to column (or page) number N, and lines XX to YY of a reference.

complexes in shale formations. *Id.* at 2:30–33.. Although the '282 Patent fails to conclusively set forth the mechanism of action underlying its purportedly inventive concept, it suggests that the metal complexing agent is likely forming complexes with the metals in the metal-polymer complexes, and particularly divalent metal ions. *Id.* at 2:33–37.

The specification of the '282 Patent describes a “method for remediation a subterranean-formed metal-polymer complex in a pre-existing well in a subterranean shale formation” generally including the following steps:

- (i) providing a metal-polymer complex remediation mixture comprising between 0.1% and 95% by weight metal complexing agent;
- (ii) injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the subterranean formation until at least some of the metal-polymer complex remediation mixture contacts the subterranean-formed metal-polymer complex, wherein the subterranean-formed metal polymer complex forms from a previously injected fracturing fluid and the metal of the subterranean-formed metal-polymer complex includes metal naturally present within the subterranean formation;
- (iii) maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex for a contact time of between about 1 minute and about 100 days, thereby allowing the metal complexing agent to cause the subterranean-formed metal-polymer complex to dissociate and dissolve but not precipitate the metal and thereby creating a low viscosity flow back fluid comprising the spent metal-polymer complex remediation mixture and the metal-polymer complex components; and
- (iv) removing the low viscosity flow back fluid from the well after the contact time, thereby improving hydrocarbon production of the well.

Ex. 1001, 2:35–53.

With regards to the metal complexing agent, the '282 Patent provides an expansive definition: “any chemical that can bind with a metal regardless of the binding mechanism and includes sequestration agents, reducing agents, **chelating agents**, ligands, porphyrins, pigments, peptides, saccharides and/or nucleic acid.” *Id.* at 5:16–21 (emphasis added). Notably, the use of chelating agents in well treatment solutions has been well-known prior to the effective filing date of the '282 Patent, as will be discussed below.

The specification of the '282 Patent further provides that “[t]he metal-polymer complex remediation mixture can contain the metal complexing agent as well as multiple chemical additives as desired. The additives may include biocide, scale inhibitor, clay control additive, oxygen scavenger and surfactant that assist fluid recovery.” *Id.* at 6:56–60. Additionally, the '282 Patent discloses that the additives may include “a corrosion inhibitor”; “a surfactant”; “a biocide”; “a dispersing agent”; “a colloidal silica deposition inhibitor”; “a mutual solvent”; and “[a]cid...in order to control the pH of the remediation mixture.” *See id.* at 7:5–8:27. The use of these additives in well treatment mixtures was also well-established prior to the effective filing date of the '282 Patent.

C. The '282 Patent's Prosecution History

The '464 Application that became the '282 Patent was filed on December 19, 2018, with two independent claims and twenty total claims. (Ex. 1002 at pp. 285–289). By preliminary amendment, claims 1–20 were canceled and claims 21–42 were added. (*Id.* at pp. 269–275). The '464 Application claims priority to the '038 Application, the '873 Provisional, and the '107 Provisional. *Id.* at pp. 318.

On June 24, 2019, the Office issued a Non-Final Rejection rejecting all pending claims: Claims 21–25, 28, and 36–40 were rejected as under 35 U.S.C. § 103 as being unpatentable over Lockhart et al. (U.S. 4,986,356) (“Lockhart”) in view of Moses et al. (U.S. 5,678,632) (“Moses”) and Dunleavy et al. (U.S. 5,038,864). *Id.* at 177–181. Claims 31–34 were rejected as under 35 U.S.C. § 103 as being unpatentable over Lockhart in view of Moses, Dunleavy, and further, in view of Hanlon et al. (U.S. 4,609,475) (“Hanlon”). *Id.* at p. 181. Claims 26, 29, and 30 were rejected as under 35 U.S.C. § 103 as being unpatentable over Lockhart in view of Moses, Dunleavy, and further, in view of Nasr-El-Din et al, (U.S. 2014/0116710) (“Nasr-El-Din”). *Id.* at pp. 181–82. Claim 27 was rejected as under 35 U.S.C. § 103 as being unpatentable over Lockhart in view of Moses, Dunleavy, Nasr-El-Din, and further, in view of Hanlon. *Id.* at pp. 182–83. Claim 35 was rejected under 35 U.S.C. § 103 as being unpatentable over Lockhart in view of Moses, Dunleavy, and further, in view of Lai et al. (U.S. 6,265,355) (“Lai”). *Id.* at pp. 183–84. Claim 41 was rejected as under 35 U.S.C. § 103 as being unpatentable over Lockhart in view of

Moses, Dunleavy, and Nasr-El-Din. *Id.* at pp. 184–87. Claim 42 was rejected as under 35 U.S.C. § 103 as being unpatentable over Lockhart in view of Moses, Dunleavy, Nasr-El-Din, and, further, in view of Lai. *Id.* at pp. 187–88.

Claims 21–40 were also rejected as under 35 U.S.C. § 103 as being unpatentable over Horton et al. (U.S. 2011/0053811) (“Horton”) in view of Moses and Lai. [*Id.* at pp. 188–198]. Further, claims 41 and 42 were rejected as under 35 U.S.C. § 103 as being unpatentable over Horton in view of Welton et al. (U.S. 8,071,511) (“Welton”), Moses, Lai, and Nasr-El-Din. [*Id.* at pp. 198–203].

On November 22, 2019, Patent Owner responded to the objections and rejections contained in the June 24, 2019 Office action. In response to the § 103 rejections over Lockhart, the Patent Owner argued that the claimed method of the ’464 Application relates to remediating subterranean-formed metal-polymer complexes “*within the subterranean formation*, not merely within a wellbore.” *Id.* at p. 136 (emphasis in original). Patent Owner distinguished Lockhart, arguing that “Lockhart does not disclose or suggest *metal naturally present within the subterranean formation complexing with polymers in fracturing fluids*.” *Id.* at 137 (emphasis in original).

On December 23, 2019, the Office issued a Final Rejection largely maintaining the objections and rejections from the previous Non-Final Rejection. *See Id.* at 91–113. Examiner found much of Patent Owner’s responses to the

arguments in the Non-Final Rejection unpersuasive. *Id.* at pp. 108–113. With regards to Patent Owner’s arguments concerning Lockhart, the Examiner “noted that the features upon which applicant relies (i.e., wherein the metal polymer complexes form from a fracturing fluid) are not recited in the rejected claim(s).” *Id.* at 109.

Patent Owner initiated an interview with the Examiner, which took place on April 2, 2020. *Id.* at p. 82. According to the Examiner Interview Record, amendments proposed by the Patent Owner were discussed. *Id.* Patent Owner proposed amending Independent Claim 21 (now Independent Claim 1), to include the language: “wherein the subterranean-formed metal polymer complex forms from a fracturing fluid...” *Id.* at p. 49. In the Interview, “[t]he Examiner suggested to add the phrase **-previously injected-** prior to “a fracturing fluid” for clarification, and agreed such amendments would overcome the rejections of record.” *Id.* at p. 82 (emphasis added).

On April 16, 2020, Patent Owner responded to the December 23, 2019 Final Rejection, which included the amendments and Examiner suggestions discussed in the Applicant Initiated Interview. *Id.* at pp. 47–67. The Examiner issued a Notice of Allowance on April 29, 2020. *Id.* at p. 15.

**D. How the Challenged Claim(s) are To Be Construed under 37
C.F.R. § 42.104(b)(3)**

In the Related Litigation, Patent Owner has proposed construing claim terms of the '282 Patent using the plain and ordinary meaning given to the term by a person of ordinary skill in the art (POSITA) at the relevant time. *See*, Ex. 1010. Petitioner does not necessarily agree that a POSITA in October 2013 would have construed the claim terms as proposed by Patent Owner. However, for purposes of this Petition (and any subsequent PTAB proceedings), Petitioner hereby adopts the plain and ordinary meaning of the claim terms.

VII. DETAILED EXPLANATION OF GROUNDS FOR CANCELLATION OF THE CHALLENGED CLAIMS

A. Ground I: Claims 1–3, 11–14, and 19 are Anticipated under 35 U.S.C. § 102 by Ayers

i. Overview of Ayers

Ayers, entitled “Compositions and Methods for Enhanced Hydrocarbon Recovery,” discloses “a method of using a fluid formulation for increasing flow, production, or recovery of oil and gas hydrocarbons from a subterranean formation.” Ex. 1004, Abstract. Ayers discloses a fluid formulation for a composition including a peroxygen, surfactant, alkali metal chelate, and a cosolvent. *Id.* at 1:51–53.

Ayers describes “[a] method for enhancing hydrocarbon recovery in a subterranean formation having a blockage or accumulation of material” generally including the steps of: (a) injecting a treatment mixture into a formation (including a chelate), (b) creating gas pressure, (c) removing blockage, and (d) wherein the pH

of the mixture is less than 10 and the chelate comprises between 0.2% and about 5% by weight of the mixture. *Id.* at 49:10–29. With regards to the alkali metal chelate, Ayers explains that “[t]he alkali metal chelate may be, for example, a mono-, di-, tri- or tetra-sodium ethylenediaminetetraacetic acid (EDTA), a mono-, di-, tri- or tetra-potassium ethylenediaminetetraacetic acid (EDTA), sodium ethylenediamine-N,N'-disuccinic acid (EDDS) or combination thereof. In some embodiments, the alkali metal chelate is sodium EDTA.” *Id.* at 21:27–36.

ii. Anticipation

“When, as by a recitation of ranges or otherwise, a claim covers several compositions, the claim is ‘anticipated’ if one of them is in the prior art.” *Titanium Metals Corp. v. Banner*, 778 F.2d 775, 227 USPQ 773 (Fed. Cir. 1985). When a patent claims a range, as in this case, that range is anticipated by a prior art reference if the reference discloses a point within the range. *Id.* If the prior art discloses its own range, rather than a specific point, then the prior art is only anticipatory if it describes the claimed range with sufficient specificity such that a reasonable fact finder could conclude that there is no reasonable difference in how the invention operates over the ranges. *Ineos USA LLC v. Berry Plastics Corp.*, 783 F.3d 865, 869 (Fed. Cir. 2015). When the prior art discloses a range which touches or overlaps the claimed range, but no specific examples falling within the claimed range are

disclosed, the reference anticipates the claims if the claimed subject matter is disclosed in the reference with "sufficient specificity." *See* MPEP 2131.03.

For a disclosure to be implicit or inherent it must be a necessary feature or result of an embodiment sufficiently described and enabled in the prior art reference. However, it is not necessary that a POSITA would have recognized that implicit or inherent disclosure at the time of invention. *See, e.g., MEHL/Biophile Int'l Corp. v. Milgraum*, 192 F.3d 1362, 1365 (Fed. Cir. 1999). If the claimed composition is identical to a composition disclosed by a prior art reference, it does not matter that the applicant recognized additional properties of that composition. *Titanium Metals Corp. of America v. Banner*, 778 F.2d at 782.

When a reference or material from various documents is explicitly incorporated by reference, it is "effectively part of the host document as if [it] were explicitly contained therein." *Arbutus Biopharma Corp. v. ModernaTX, Inc.*, 65 F.4th 656, 662–63 (Fed. Cir. 2023).

1. Independent Claim 1

a. Element 1[Preamble]: "A method for remediating a subterranean-formed metal-polymer complex in a pre-existing well in a subterranean shale formation, the method comprising:"

To the extent the preamble is limiting, Ayers discloses this limitation. Ayers discloses "a method of using a fluid formulation for increasing flow, production, or recovery of oil and gas hydrocarbons from a subterranean formation." Ex. 1004 at

Abstract. The method in Ayers aims to alleviate reductions in the productivity of pre-existing oil and gas wells in subterranean formations. *See* Ex. 1003 at ¶ 79. Ayers identifies the following as potential causes of reduced flow, production, or recovery of oil and gas: “[t]he formation of immobile emulsions, gels or polymers, the incomplete recovery of stimulation or fracking liquids, and the formation of larger particle blockages...also can lead to clogging and reduction of oil and gas recovery from subterranean formations.” *Id.* at 5:41–47. Ayers further identifies that “damage or blockage causing polymers can include...cross-linked polymers...and/or other viscosity increasing polymers.” *Id.* at 24:1–6.

Accordingly, to the extent the preamble is limiting, Ayers discloses this limitation.

b. Element 1[a]: “providing a metal-polymer complex remediation mixture comprising between 0.1% and 95% by weight metal complexing agent;”

Ayers discloses a “fluid formulation” or “composition,” i.e., a metal-polymer complex remediation mixture, including “a peroxygen, a surfactant, an alkali metal chelate, and a cosolvent.” *Id.* at 1:51–53. Ayers explains that “an alkaline chelate can be added to the formation to **chelate metals associated with the formation matrix** or pore liquids.” *Id.* at 29:61–62 (emphasis added). According to the ’282 Patent, “a metal-complexing agent may be **any chemical that can bind with a**

metal regardless of the binding mechanism and includes...chelating agents....”

Ex. 1001 at 5:16–21 (emphasis added). Here, the alkali metal chelate component of the fluid formulation discloses a metal complexing agent. *See* Ex. 1003 at ¶ 86.

Ayers provides that the chelate component of its fluid formulation may be, *inter alia*, EDTA, EDDS, or salts thereof. *See id.* at 21:27–36. The specification of ’282 Patent includes EDTA and EDDS, and salts thereof as a “suitable complexing agent.” *See* Ex. 1001 at 5:60–6:9; *see also id.* at 6:10–38.

Ayers further discloses that the chelate concentration “may be between about **0.2 and about 5 percent by weight**,” which is a specific range within the claimed range (i.e., between 0.1% and 95% by weight). Ex. 1004 at 36:27–29. As such, Ayers anticipates this limitation of Element 1[a]. *See Atofina v. Great Lakes Chem. Corp.*, 441 F.3d 991, 999 (Fed. Cir. 2006).

Accordingly, Ayers discloses the limitations of Element 1[a].

- c. Element 1[b]: “injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the subterranean formation until at least some of the metal-polymer complex remediation mixture contacts the subterranean-formed metal-polymer complex, wherein the subterranean-formed metal polymer complex forms from a previously injected fracturing fluid and the metal of the subterranean-formed metal-polymer complex includes metal naturally present within the subterranean formation;”**

Ayers discloses that “[t]he composition may be introduced into the wellbore or subterranean formation by, for example, pumping, displacing, or locating the composition at a desired location...**at a rate and pressure less than the reservoir hydraulic fracture pressure.**” Ex. 1004 at 36:65–37:3 (emphasis added). Also, Ayers clarifies that the fluid formulation is “injected.” *See id.* at 42:43–49. Accordingly, Ayers discloses injecting the remediation mixture into the well at a pressure less than a fracture pressure of the subterranean formation.

Ayers also discloses that “[t]he introduced **composition may be brought into contact with, for example, blockage or damage in the wellbore or subterranean formation.**” *Id.* at 38:29–31. Ayers further explains that said blockage or damage may result from previously injected fracturing fluid:

Inevitably, hydraulic fracturing also typically results in some level of formation damage and blockage as the **fracturing fluids intrude the natural formation and have negative interactions** in the way of...**forming immobile emulsions or gels....**

Id. at 8:27–35. As such, Ayers discloses that subterranean formations of metal-polymer complexes may result due to previous injections of fracturing fluid.

It would be obvious to a POSITA that, for example, a fracturing fluid containing polyacrylamide, a common polymer additive, may intrude the natural formation and have negative interactions, including forming metal-polymer complexes with metal ions naturally present in the formation. *See* Ex. 1003 at ¶ 98.

Ayers discloses the presence of transition metals in the formation. *See id.* at 28:20–21 (“[t]he presence of inorganic transition metals, either **naturally present** in pore liquids (oil or water) or the formation”)(emphasis added).

Moreover, Ayers recognizes that, “[o]ver time **stranded polymer** in the well bore, filter cake, and **in adjacent subterranean formations** can **form matrices that block oil flow....**” *Id.* at 13:28–32 (emphasis added).

Further, Ayers discloses that, in some embodiments, “the damage or blockage may include viscosity increasing polymers. **The polymer may have been introduced as a separate treatment fluid.** The polymer may include, for example...**cross-linked polymers...**other viscosity increasing polymers, or combinations thereof.” *Id.* at 38:46–53 (emphasis added).

Accordingly, Ayers discloses the limitations of Element 1[d].

d. Element 1[c]: “maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex for a contact time of between about 1 minute and about 100 days, thereby allowing the metal complexing agent to cause the subterranean-formed metal-polymer complex to dissociate and dissolve but not precipitate the metal and thereby creating a low viscosity flow back fluid comprising the spent metal-polymer complex remediation mixture and the metal-polymer complex components; and”

Ayers discloses maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex. *Id.* at 22:20–24

(“[t]he **composition can be allowed to contact** the wellbore or a portion of a wellbore or **subterranean formation or hydrocarbon-bearing subterranean formation** for a sufficient period of time...”). As discussed above, Ayers discloses the existence of subterranean-formed metal-polymer complexes occurring within the subterranean formation. Accordingly, those in-situ formed metal-polymer complexes would necessarily be encountered by the composition disclosed in Ayers. *Id.*

Further, Ayers discloses contacting the remediation mixture with the metal-polymer complex for a contact time between about 1 minute and 100 days. *See Id.* at 22:32–39. In one Example, Ayers discloses a contact time of 8 to 16 hours, which is a specific range within the claimed range (i.e., between about 1 minute and about 100 days). *Id.* at 43:27. As such, Ayers anticipates this limitation of Element 1[a]. *See Atofina v. Great Lakes Chem. Corp.*, 441 F.3d 991, 999 (Fed. Cir. 2006).

Further, Ayers makes clear that the disclosed composition can be “[a]llowed to contact blockage or damage in...a portion of a subterranean formation for **a sufficient time...to sufficiently alter, remove, degrade, and/or dissolve the permeability damage or blockage...**” Ex. 1004 at 23:30–36 (emphasis added). Accordingly, any contact time between the composition in Ayers, which includes a chelating agent, and a subterranean-formed metal-polymer complex (i.e., the permeability blockage or damage), for a time sufficient to degrade and/or dissolve

the damage and or blockage would inherently meet the limitation of at least “about” 1 minute of contact time. Ex. 1003 at ¶ 108.

Furthermore, Ayers explains that the disclosed composition induces the dissociation and dissolution of subterranean-formed metal-polymer complexes. *See id.* at 23:30–36. The chelating agents disclosed in Ayers would prevent metal ion precipitation by forming complexes that keep the metals dissolved in solution. *See* Ex. 1004 at 1:63–65 (“The alkali metal chelate can serve the purpose of scavenging ionic or **bound phases of metals in a formation**[.]”) (emphasis added).

Ayers further discloses that the dissociation and dissolution of the metal-polymer complexes generate a low viscosity flowback fluid comprising the spent metal-polymer complex remediation mixture and metal-polymer complex components. Ayers explains that the disclosed composition will “liquefy” encountered inorganic and organic materials, the “liquified material” then displaced through the formation and into the well for subsequent removal. *See id.* at 28:25–35; *see also id.* at 42:36–38.

According to Ayers, Figure 3 shows an example of materials evacuated from a well subsequent to treatment with the disclosed composition. Ayers notes that the evacuated materials from Figure 3 depict “**flowable characteristics** that readily enable pumping as a liquid.” *Id.* at 42:36–38. A POSITA at the relevant time would understand that to disclose a low viscosity flow back fluid. Ex. 1003 at ¶ 112.

Accordingly, Ayers discloses the limitations of Element 1[c].

- e. **Element 1[d]: “removing the low viscosity flow back fluid from the well after the contact time, thereby improving hydrocarbon production of the well.”**

Ayers discloses that “[t]he composition may be removed from the **wellbore**, for example, by pumping or flushing.” *Id.* at 37:41–44 (emphasis added). Ayers further explains that “after the composition is introduced into the wellbore or subterranean formation, the wellbore or **subterranean formation is contacted** for a period of time sufficient **to increase flow, production, or recovery of hydrocarbons.**” *Id.* at 36:60–64 (emphasis added); *see also id.* at 22:20–24. Accordingly, Ayers discloses removing the low viscosity flow back fluid after the contact time, thereby improving hydrocarbon production of the well. *See also id.* at 42:36–38.

Accordingly, Ayers discloses the limitations of Element 1[d].

2. Dependent Claim 2

- a. **“The method of claim 1, wherein the metal complexing agent is citric acid, formic acid, or mixtures thereof.”**

First, Ayers discloses that “[i]n some embodiments, an organic acid, mineral acid, or a base is added to the composition to adjust the pH. The acid may be, for example, hydrochloric, hydrofluoric, **formic, citric** or acetic acid.” *Id.* at 35:66–36:2 (emphasis added). While Ayers highlights the pH-adjusting function of these acids,

the ability of these acids to perform other functions due to their inherent properties remain unrestricted. Crucially, a POSITA, prior to the effective date of the '282 Patent, would have recognized as inherent citric and formic acid's well-established chelating properties, rendering them suitable metal complexing agents within the disclosed subterranean treatment process. Ex. 1003 at ¶ 120.

Additionally, Ayers discloses that the use of citric acid, formic acid, or mixtures thereof, within fluid formulations for treating subterranean formations to achieve increased permeability and enhance hydrocarbon production: "Matrix stimulation treatments using acids, or acids in combination with water, surfactants, or other chemicals, are **very common** and also referred to as 'acid treatments' or 'acidizing'. Hydrochloric acid (HCl), acetic acid, **citric, formic** and hydrofluoric acid **are commonly used** acids for matrix treatment." *Id.* at 11:35–40 (emphasis added). It is common knowledge within the relevant industry that citric acid, formic acid, and mixtures thereof, possess effective chelating properties. Ex. 1003 at ¶ 122.

Moreover, given the acknowledged purpose of the claimed invention—to enhance hydrocarbon recovery—it would be inherent to select a material capable of binding with metal ions, which are often present in subterranean formations and can impede fluid flow. Ex. 1003 at ¶ 122.

Thus, Ayers discloses the limitations of Dependent Claim 2.

3. Dependent Claim 3

- a. **“The method of claim 1, wherein the metal complexing agent is selected from the group consisting of (EDTA), (PDTA), (NTA), (HEDTA), (DTPA), (HEIDA), (CDTA), (DPAS), (EDDHA), glucoheptonic acid, gluconic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, phthalic acid, terephthalic acid, aconitic acid, carballylic acid, trimesic acid, isocitric acid, citric acid, salts thereof, and mixtures thereof.”**

Ayers discloses that “[c]ompositions and methods of embodiments according to the present invention include or require the use of a chelate, such as a mono-, di-, tri-, or tetra-sodium ethylenediaminetetraacetic acid (EDTA), a mono-, di-, tri-, or tetra-potassium ethylenediaminetetraacetic acid (EDTA)...For example, the chelate can be sodium EDTA.” *Id.* at 21:27–36..

Accordingly, Ayers discloses the limitations of Dependent Claim 3.

4. Dependent Claim 11

- a. **“The method of claim 1, wherein the metal-polymer complex remediation mixture further comprises a pH modifier.”**

Ayers discloses that “pH control of the composition can be enabled by use of alkalinity increasing pH buffers, such as caustic soda, sodium carbonate, sodium bicarbonate, lime or hydrated lime or with organic or inorganic acids in the case of lowering pH.” *Id.* at 28:1–4; *see also id.* at 20:53–54.

Accordingly, Ayers disclose the limitations of Dependent Claim 11.

5. Dependent Claim 12

- a. **“The method of claim 11, wherein the metal-polymer complex remediation mixture further comprises between 0.1 and 30% of one or more acids as a pH modifier.”**

Petitioner incorporates the arguments above concerning Dependent Claim 11. Ayers discloses that the pH modifier may be one or more acids: “[i]n some embodiments, an **organic acid, mineral acid**, or a base is added to the composition to adjust the pH.” *Id.* at 35:66–67 (emphasis added).

Additionally, Ayers discloses that the composition described therein may “be augmented with less than 1% acids.” *Id.* at 32:28–29. This concentration falls squarely within the claimed range.

Further, Ayers discloses that “the pH may be adjusted by adding an acid or base to the composition,” and discloses, for example, that “[t]he composition may have a pH less than about 10.0.” *Id.* at 41:60–61; *id.* at 35:18–19. Consequently, Ayers inherently anticipates the inclusion of an acid pH modifier in an amount falling within the claimed range, as adjusting the pH requires the presence of an acid pH modifier in an amount sufficient to attain the desired pH level.

Accordingly, Ayers discloses the limitations of Dependent Claim 12.

6. Dependent Claim 13

- a. **“The method of claim 12, wherein the acid is selected from the group consisting of hydrochloric acid, aqua regia, arsenic acid,**

boric acid, carbonic acid, chloric acid, chromic acid, fluoroantimonic acid, fluoroboric acid, fluorosulfuric acid, fulminic acid, hexafluorophosphoric acid, hexafluorosilicic acid, hydrobromic acid, hydrofluoric acid, hydrogen iodide, hypochlorous acid, hypofluorous acid, hypophosphoric acid, iodic acid, nitric acid, nitrosyl-O-hydroxide, nitrous acid, orthocarbonic acid, perchloric acid, permanganic acid, perrhenic acid, pertechnetic acid, phosphoric acid, silicic acid, sulfuric acid, thiocyanic acid, titanitic acid, tungstic acid, xenic acid, and mixtures thereof.”

Petitioner incorporates the arguments above concerning Dependent Claim 11 and 12. Ayers discloses that “[t]he acid may be, for example, **hydrochloric, hydrofluoric**, formic, citric or acetic acid.” *Id.* at 35:67–36:4 (emphasis added).

Accordingly, Ayers discloses the limitations of Dependent Claim 13.

7. Dependent Claim 14

- a. “The method of claim 12, wherein the acid is **hydrochloric acid.**”

Petitioner incorporates the arguments above concerning Dependent Claim 11, 12, and 13. As discussed above, Ayers discloses wherein the acid is hydrochloric acid. *See id.* at 35:66–36:4.

8. Dependent Claim 19

- a. “The method of claim 1, wherein the contact time is between about 4 hours and about 4 days.”

With regards to contact time, Ayers discloses that “i]n some embodiments, the composition is allowed to remain in the wellbore or subterranean formation for a period of time before hydrocarbon extraction resumes.” Ex. 1004 at 8:59–61. Specifically, Ayers discloses that the contact time may be within a range that is “more than 4 hours” and “less than...4 days.” This disclosed range falls entirely within the claimed contact range described in the ’282 Patent. *See id.* at 38:59–67.

Further, Ayers discloses that the described composition can be “[a]llowed to contact blockage or damage in a portion of the wellbore or a portion of a subterranean formation for **a sufficient time...**to sufficiently alter, remove, **degrade, and/or dissolve the permeability damage or blockage...**” *Id.* at 23:30–36 (emphasis added). Accordingly, any contact time between the composition in Ayers, which includes a chelating agent (i.e., a metal complexing agent), and a subterranean-formed metal-polymer complex (i.e., the permeability blockage or damage), for a time sufficient to degrade and/or dissolve the damage and or blockage would seem to inherently meet the limitation of at least “about” 4 hours of contact time. Ex. 1003 at ¶ 140.

As such, Ayers discloses the limitations of Dependent Claim 19.

B. Ground II: Claims 1–22 are Obvious under 35 U.S.C. § 103 Over Ayers in View of Frenier

i. Overview of Frenier

Frenier, entitled “Well Treatment Fluids Comprising Chelating Agents,” is directed to “stimulation of hydrocarbon wells and in particular to acid fluids and methods of using such fluids in treating a subterranean formation having low permeability.” Ex. 1005, at 1:6–9. Frenier discloses a “well treatment fluid,” comprising: “a first acid; water; and a hydroxyethylaminocarboxylic acid.” *Id.* at 4:12–13. Generally, the methods include “the step of injecting a well treatment fluid composition via a wellbore into a subterranean formation... at a fluid pressure that is less than the minimum in situ rock stress....” *Id.* at 4:49–53. Frenier explains that the practice of using treatment fluids to improve the productivity of oil wells was disclosed over a century ago. *See id.* at 1:66–2:3.

Moreover, Frenier states “[t]he desired result of the treatment is an increase in the permeability of the formation, for example by the creation or enlargement of passageways through the formation, and therefore an increase in the rate of production of formation fluids such as oil and gas.” *Id.* at 7:34–38.

ii. Obviousness

“A party that petitions the Board for a determination of obviousness must show that a skilled artisan would have been motivated to combine the teachings of the prior art references to achieve the claimed invention, and that the skilled artisan would have had a reasonable expectation of success in doing so.” *Apotex Inc. v. Wyeth LLC*, IPR2014-00115, Paper 94, slip op. at 11 (Apr. 20, 2015) (internal

quotation omitted). The obviousness determination turns on underlying factual inquiries involving: (1) the scope and content of prior art, (2) differences between claims and prior art, (3) the level of ordinary skill in pertinent art, and (4) secondary considerations such as commercial success and satisfaction of a long-felt need. *See Procter & Gamble Co. v. Teva Pharm. USA, Inc.*, 566 F.3d 989, 994 (Fed. Cir. 2009).

iii. Motivation to Combine Ayers and Frenier

To the extent that it is needed, a POSITA would have been motivated to combine the teachings of Ayers and Frenier to produce obvious, beneficial, and predictable results. Ex. 1003 at ¶ 68. Ayers and Frenier are analogous prior art and both pertain to the same field of endeavor. Specifically, both seek to enhance hydrocarbon production using treatment mixtures that include chelating agents. As discussed in both Ayers and Frenier, the beneficial results of the disclosed operations include increasing the permeability of subterranean formations of presently low permeability. Increasing the permeability of the formations provides the benefit of allowing more hydrocarbons to be recovered from the well. The optimized performance of hydrocarbon-bearing wells would have been desirable to a POSITA. Ex. 1003 at ¶ 69.

Ayers discloses a composition for enhanced hydrocarbon recovery, acknowledging its potential use as a stimulation fluid. Ex. 1004 at 27:14–14 (“The

composition according to an embodiment of the present invention can be for a drilling fluid, treatment fluid, stimulation fluid, fracturing fluid, and/or a fluid used in an enhanced oil recovery technique.”). Notably, acid treatments are recognized as a well-established stimulation technique within the industry. *Id.* at 11:35–37 (“Matrix stimulation treatments using acids, or acids in combination with water, surfactants, or other chemicals, are very common and also referred to as ‘acid treatments’ or ‘acidizing’.”).

Frenier, on the other hand, specifically discloses a fluid formulation for an acid treatment for use in stimulation activities. Specifically, Frenier discloses an “acid fluid that is useful in stimulation and workover operations, and in particular, for the control of iron in acidizing operations.” Ex. 1005 at Abstract.

A POSITA at the time of filing would have been highly motivated to combine the teachings of Ayers and Frenier. Ex. 1003 at ¶ 73. First, a POSITA would have recognized that Ayers’ composition, aimed at enhancing hydrocarbon recovery, possesses properties that could be beneficial for stimulation fluids. Combining it with Frenier’s dedicated matrix acidizing fluid could create a synergistic effect, potentially leading to a more effective stimulation treatment. *Id.* Second, as Ayers acknowledges acid treatments as a stimulation technique, a POSITA would have been aware of the established benefits of incorporating components of an acid treatment mixture into a stimulation fluid. *Id.* Frenier’s matrix acidizing fluid

specifically addresses this need, and combining it with Ayers' composition offers a potential pathway to an improved stimulation fluid. Finally, a POSITA would have recognized that it was possible that Ayers' composition, while beneficial for enhancing hydrocarbon recovery, might lack some of the specific properties desired for optimal stimulation. *Id.* at ¶ 74. A POSITA would be motivated to combine the technologies to address these potential shortcomings and create a more versatile stimulation fluid. *Id.* Moreover, a POSITA would have a reasonable expectation of success in combining the teachings of Ayers and Frenier. *Id.*

1. Independent Claim 1

a. Element 1[Preamble]: “A method for remediating a subterranean-formed metal-polymer complex in a pre-existing well in a subterranean shale formation, the method comprising:”

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers's disclosure of Element 1[Preamble]. It would have been obvious to a POSITA to apply the disclosures of Ayers to a method for remediating a subterranean formed metal-polymer complex in a pre-existing well in a subterranean formation. Ex. 1003 at ¶ 148.

Moreover, Frenier discloses this limitation, to the extent the preamble is limiting. Frenier discloses “methods to **enhance the productivity of hydrocarbon wells** (e.g., oil wells) by...**removing (by dissolution) near-wellbore formation**

damage.” Ex. 1005 at 1:55–60 (emphasis added). A POSITA would have understood Frenier’s disclosure to apply to remediating a subterranean-formed metal-polymer complex. Ex. 1003 at ¶ 150.

Further, Frenier discloses [a]n acid fluid that is useful in stimulation and **workover operations**, and in particular for the control of iron in acidizing operations...” *Id.* at Abstract (emphasis added). A POSITA would have understood Frenier’s disclosure to apply to remediating pre-existing wells in addition to new wells. Ex. 1003 at ¶¶ 151–53.

Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Element 1[Preamble].

b. Element 1[a]: “providing a metal-polymer complex remediation mixture comprising between 0.1% and 95% by weight metal complexing agent;”

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers’s disclosure of Element 1[a].

Moreover, Frenier discloses a “well treatment fluid,” i.e., a remediation mixture, comprising: “a first acid; water; and a hydroxyethylaminocarboxylic acid.” Ex. 1005 at 4:12–13. Frenier further explains that “[e]xamples of first acids include non-oxidizing mineral acids...Other examples include non-oxidizing organic acids such as formic acid, acetic acid, and mixtures thereof.” *Id.* at 5:27–31. However,

Frenier also discloses that “hydroxyethylaminocarboxylic acids have the advantage of being effective in acidizing treatment of a formation **without the use of a first acid.**” *Id.* at 6:31–33 (emphasis added). “Preferably, the hydroxyethylaminocarboxylic acid is selected from hydroxyethylethylenediaminetriacetic acid (HEDTA), hydroxyethyliminodiacetic acid (HEIDA), or a mixture thereof.” *Id.* at 4:13–17. Thus, Frenier discloses various treatment mixtures wherein the metal complexing agent may be a traditional chelating or sequestering agent such as HEDTA or HEIDA, or may be a lower pH organic acid such as acetic acid. A POSITA would have known and understood the same. Ex. 1003 at ¶ 164.

Frenier provides examples of “typical concentration ranges” for its treatment mixture with and without a first acid, including between 1% and 30% by weight metal complexing agent, which falls squarely within the claimed range set forth in Element 1[a]. Ex. 1005 at 6:25–47. Frenier further discloses treatment mixtures having between 15% and 28% and between 10% and 30% by weight metal complexing agent. *See* claims 1, 9, and 28 at *id.*, 11:27–34; 11:54–63; 13:3–20.

A POSITA, prior to the effective date of the ’282 Patent, would have recognized that the specific proportion of metal complexing agent within the claimed remediation mixture is likely to vary depending on several factors, including the unique characteristics of the subterranean formation being treated. *See* Ex. 1005 at

6:16–19 (“The proportion of the various components of a composition of the present invention will vary depending on the characteristics of the formation to be treated, the acid to be used, and other factors well known in the art.”).

The broad ranges disclosed in Ayers and Frenier provide ample room for a POSITA to make adjustments. Ex. 1003 at ¶ 169. Given the vastness of these concentration ranges and the well-understood functionality of metal complexing agents, a POSITA could readily arrive at successful formulations through routine experimentation. *Id.* The established functions of metal complexing agents provide a predictable framework for optimizing the composition for a particular application. *Id.* A POSITA, familiar with these functionalities and the specific needs of a treatment project, could readily adjust the ratios within the claimed broad ranges to achieve the desired outcome. *Id.* This level of optimization would not require inventive leaps but rather routine experimentation based on established scientific principles. Ex. 1003 at ¶ 170.

Accordingly, Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Element 1[a].

- c. Element 1[b]: “injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the subterranean formation until at least some of the metal-polymer complex remediation mixture contacts the subterranean-formed metal-polymer complex, wherein the subterranean-formed metal polymer complex forms from a previously injected fracturing**

fluid and the metal of the subterranean-formed metal-polymer complex includes metal naturally present within the subterranean formation;”

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers’s disclosure of Element 1[b].

Moreover, Frenier discloses “a method of acid-treating a subterranean formation.” Ex. 1005 at 4:44–45. “The method includes the step of injecting a well treatment fluid composition via a wellbore **into a subterranean formation.**” *Id.* at 4:45–47 (emphasis added). Frenier further explains that, “the injection step is **preferably performed at a fluid pressure that is less than the minimum in situ rock stress...**” *Id.* at 4:49–53 (emphasis added).

Accordingly, Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Element 1[b].

d. Element 1[c]: “maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex for a contact time of between about 1 minute and about 100 days, thereby allowing the metal complexing agent to cause the subterranean-formed metal-polymer complex to dissociate and dissolve but not precipitate the metal and thereby creating a low viscosity flow back fluid comprising the spent metal-polymer complex remediation mixture and the metal-polymer complex components; and”

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers’s disclosure of Element 1[c].

Furthermore, given the vastness of time within the range, a POSITA would recognize the optimal contact time by experimentation and trial and error since it has been held, “[w]here the general conditions of a claim are disclosed in the prior art, it is not inventive to discover the optimum or workable ranges by routine experimentation.” *In re Aller*, 220 F.2d 454, 456, 105 USPQ 233, 235 (CCPA 1955). *See* Ex. 1003 at ¶ 189.

Moreover, Frenier discloses that “[a]fter the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow more complete reaction between the acid and the formation material.” Ex. 1005 at 7:30–34. Frenier also explains that “[p]arameters such as...**shut-in time**...must be determined for each particular treatment since each of these parameters depends upon the extent of the damage, formation geology (e.g., permeability), formation temperature, depth of the producing zone, etc. **A well-treatment designer of ordinary skill is familiar with the essential features of matrix acidizing treatments.**” *Id.* at 7:39–46 (emphasis added). Notably, in the examples provided in Frenier, the treatment solutions were maintained in contact with the formation samples for 24 and 72 hours. Ex. 1005 at 9:27–66.

Here, as with Ayers above, a POSITA would recognize the optimal contact time by experimentation and trial and error since it has been held, “[w]here the general conditions of a claim are disclosed in the prior art, it is not inventive to

discover the optimum or workable ranges by routine experimentation.” *In re Aller*, 220 F.2d at 456.

e. Element 1[d]: “removing the low viscosity flow back fluid from the well after the contact time, thereby improving hydrocarbon production of the well.”

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers’s disclosure of Element 1[d].

Moreover, Frenier discloses removal of the low viscosity flow back fluid from the well after the contact time, thereby improving hydrocarbon production of the well. Frenier plainly states, “the present invention relates to methods to **enhance the productivity of hydrocarbon wells** (e.g., oil wells) by creating alternate flowpaths by removing portions of a wellbore coating, dissolving small portions of the formation, **or removing (by dissolution) near-wellbore formation damage.**” Ex. 1005 at 1:55–60.

Frenier also cites, U.S. Patent. No. 1,877,504A to Grebe, entitled “Treatment of Deep Wells,” issued September 13, 1932 (“Grebe”) (Ex. 1013). Grebe is directed to “increasing or restoring” the flow of an oil well in a limestone formation “by treating with a quantity of an acid, such as hydrochloric acid. The acid has the effect of attacking and dissolving the rock, thereby enlarging the cavity at the bottom of the well, or the channels and pores in the rock through which oil flows to the well.” Ex. 1013 at page 1, lines 45–52. Grebe further discloses, “[i]n carrying out our

improved method we employ a mineral acid, preferably hydrochloric acid, inasmuch as the latter **upon reacting with the calcareous rock forms water soluble salts which remain in solution and are removed from the well with the spent acid.**” *Id.* at lines 59–74 (emphasis added).

Accordingly, Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Element 1[d].

2. Dependent Claim 2

- a. **“The method of claim 1, wherein the metal complexing agent is citric acid, formic acid, or mixtures thereof.”**

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers’s disclosure of Dependent Claim 2.

Moreover, Frenier cites U.S. Pat. No. 4,888,121 to Dill, entitled “Compositions and Method for Controlling Precipitation When Acidizing Sour Wells” (“Dill”). According to Frenier, Dill “discloses an acidizing composition that includes an acid such as CHI; **an iron sequestering agent such as citric acid, EDTA, or nitrilotriacetic acid (NTA); and a sulfide modifier such as formaldehyde.**” Ex. 1005 at 3:39–45 (emphasis added).

Thus, a POSITA would have known and understood, or at a minimum it would have been obvious, to use citric acid as the metal complexing/sequestering agent prior to the effective date of the ’282 Patent. Ex. 1003 at ¶¶ 208–10.

Accordingly, Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Dependent Claim 2.

3. Dependent Claim 3

- a. **“The method of claim 1, wherein the metal complexing agent is selected from the group consisting of (EDTA), (PDTA), (NTA), (HEDTA), (DTPA), (HEIDA), (CDTA), (DPAS), (EDDHA), glucoheptonic acid, gluconic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, phthalic acid, terephthalic acid, aconitic acid, carballylic acid, trimesic acid, isocitric acid, citric acid, salts thereof, and mixtures thereof.”**

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers’s disclosure of Dependent Claim 3. Ayers discloses wherein the metal complexing agent is selected from a group including EDTA and salts thereof. Ex. 1004 at 21:27–36.

Moreover, a POSITA would have known and understood, or at a minimum it would have been obvious, to use EDTA, or a salt thereof, as a metal complexing agent prior to the effective date of the ’282 Patent. Ex. 1003 at ¶ 214.

Accordingly, Ayers, as informed by the knowledge of a POSITA, discloses Dependent Claim 3.

4. Dependent Claim 4

- a. **“The method of claim 1, further wherein the metal complexing agent is selected from the**

group consisting of (GLDA), (MGDA), (β -ADA), ethylenediaminedisuccinic acid, (EDDS), (IDS), (HIDS), polyamino disuccinic acids, (BCA6), (BCA5), (MCBA5), (TCA6), (MIDA), (IDA), (ADA), hydroxymethyliminodiacetic acid, (CEAA), (CMAA), diethylenetriamine-N,N''-disuccinic acid, triethylenetetramine-N,N'''-disuccinic acid, 1,6-hexamethylenediamine-N,N'-disuccinic acid, tetraethylenepentamine-N,N''''-disuccinic acid, 2-hydroxypropylene-1,3-diamine-N,N''-disuccinic acid, 1,2-propylenediamine-N,N'-disuccinic acid, 1,3-propylenediamine-N,N'-disuccinic acid, cis-cyclohexanediamine-N,N'-disuccinic acid, trans-cyclohexanediamine-N,N'-disuccinic acid, ethylenebis(oxyethylenenitrilo)-N,N'-disuccinic acid, glucoheptanoic acid, cysteic acid-N,N-diacetic acid, cysteic acid-N-monoacetic acid, alanine-N-monoacetic acid, N-(3-hydroxysuccinyl) aspartic acid, N-[2-(3-hydroxysuccinyl)]-L-serine, aspartic acid-N,N-diacetic acid, aspartic acid-N-monoacetic acid, salts thereof, and mixtures thereof."

Ayers discloses that "[c]ompositions and methods of embodiments according to the present invention include or require the use of a chelate, such as...sodium ethylenediamine-N,N-disuccinic acid (EDDS)." *Id.* at 21:27–36. As such, Ayers discloses wherein the metal complexing agent is selected from a group including EDDS and salts thereof, such as sodium EDDS.

Moreover, it would have been obvious to a POSITA before the effective filing date of the '282 Patent to select one of the acids, salts, or mixtures listed in Dependent Claim 4 in light of the disclosure in Ayers. Ex. 1003 at ¶ 218.

Accordingly, Ayers, as informed by the knowledge of a POSITA, discloses
Dependent Claim 4.

5. Dependent Claim 5

- a. **“The method of claim 1, further wherein the metal complexing agent is selected from acetic acid, acrylates, dihydroxymaleic acid, salts of dihydroxymaleic acid, (EDTA), salts of EDTA, erythorbic acid, erythroboric acid, formic acid, gluconodeltalactone, (GLDA), salts of GLDA, (HEDTA), salts of HEDTA, (HEIDA), salts of HEIDA, (MGDA), salts of MGDA, (NTA), and mixtures thereof.”**

Petitioner incorporates herein its statements and arguments above from with respect to Ayers’s disclosure of Dependent Claim 3. Ayers discloses wherein the metal complexing agent is selected from a group including EDTA and salts thereof. Ex. 1004 at 21:27–36.

Moreover, it would have been obvious to a POSITA before the effective filing date of the ’282 Patent to select one of the acids, salts, or mixtures listed in Dependent Claim 5 in light of the disclosure in Ayers. Ex. 1003 at ¶ 122.

Accordingly, Ayers, as informed by the knowledge of a POSITA, discloses
Dependent Claim 5.

6. Dependent Claim 6

- a. **“The method of claim 1, wherein the metal-polymer complex remediation mixture comprises between 10% and 80% by weight**

metal complexing agent, one or more additives, and mutual solvent.”

Ayers discloses a remediation mixture that includes, *inter alia*, a chelating agent and additional components such a surfactant and a cosolvent. Ex. 1004 at 21:27–36. Ayers explains that the composition may contain a chelating agent having a concentration between about 0.2 and about 5.0 percent by weight (*id.* at 36:20–22); a surfactant at a concentration between 0.01 and about 50 percent by weight (*id.* at 36:5–7); and a cosolvent having a concentration between about 1 to 40 percent by weight (*id.* at 36:46–48). Accordingly, Ayers discloses a remediation mixture containing a metal complexing agent, one or more additives, and a mutual solvent, with the combined weight percentage of those components falling between 10% and 80%. Ex. 1003 at ¶¶ 224–233.

Additionally, Ayers incorporates by reference U.S. Patent No. 6,593,279 to Von Krosig et al. (“Von Krosig”), which, according to Ayers, teaches a composition for cleaning oil sludges from oil well cuttings, well formations, and down hole wells. *See id.* at 12:18–20. The Von Krosig reference discloses a composition requiring a “co-solvent,” and discloses numerous “co-solvents” to be used. Ex. 1011 at 6:7–14. A POSITA would understand that the aforementioned listing of “co-solvents” disclose mutual solvents, i.e., a chemical additive for stimulation treatments that is soluble in water, oil, and acid-based treatment fluids. Ex. 1003 at ¶ 227.

Similarly, Frenier discloses various treatment mixtures wherein the metal complexing agent may be a traditional chelating or sequestering agent such as HEDTA or HEIDA. *See* Ex. 1005 at 4:23–27; 5:27–3. Frenier further discloses that, if a solvent is used in the disclosed formulation, “the solvent typically comprises an alcohol or a glycol ether.” *Id.* at 6:14–15. A POSITA at the time of filing would have understood glycol ether to disclose a mutual solvent. Ex. 1003 at ¶ 231. Frenier further discloses treatment mixtures having between 15% and 28% and between 10% and 30% by weight metal complexing agent. Ex. 1005 at 11:27–34; 11:54–63; 13:3–20. Frenier further discloses that composition additives, e.g., wetting agents, non-emulsifiers, or solvents, may comprise between 0.1–10% and 0.1–5%. *See id.* at 6:25–45.

Ayers, alone or with Frenier, discloses a remediation mixture containing a metal complexing agent, one or more additives, and a mutual solvent, with the combined weight percentage of those components falling between 10% and 80%. Ex. 1003 at ¶ 233. Additionally, the broadness of the claimed ranges and the predictability associated with optimizing the composition based on well-known functionalities render the invention obvious to a POSITA. Ex. 1003 at ¶ 237.

Accordingly, Ayers, alone or with Frenier, as informed by the knowledge of a POSITA, discloses Dependent Claim 6.

7. Dependent Claim 7

- a. **“The method of claim 6, wherein the metal complexing agent comprises between 10% and 80% by weight citric acid, formic acid, or mixtures thereof, between 0.1 and 30% of one or more acids as a pH modifier, and mutual solvent.”**

Petitioner incorporates herein its statements and arguments above with respect to Ayers’s disclosure of Dependent Claim 6.

Ayers discloses that “[t]he pH of the composition can be adjusted if need to avoid pH changes in the formation.” *Id.* at 20:53–54. Specifically, Ayers discloses that “an organic acid, mineral acid, or a base is added to the composition to adjust the pH [such as] hydrochloric, hydrofluoric, formic, citric or acetic acid.” *Id.* at 35:66–36:2. Accordingly, it would have been obvious to a POSITA at the time of filing that one or more acids may be used as a pH modifier. Ex. 1003 at ¶ 243.

Further, Frenier discloses a treatment fluid including formic acid. *Id.* at 5:27–31. Although Frenier refers to formic acid as a possible “first acid,” formic acid’s inherent properties as a metal complexing agent is not diminished. It would have been obvious to a POSITA at the time of filing to select formic acid for use as a metal complexing agent. Ex. 1003 at ¶ 245.

Frenier discloses that its treatment mixtures may contain metal complexing agents in a concentration between 1% and 30% by weight, which covers much of the claimed ranges set forth in Dependent Claim 7. Ex. 1005. at 6:25–47. Frenier further discloses treatment mixtures having between 15% and 28% and between

10% and 30% by weight metal complexing agent. Ex. 1005 at 11:27–34; 11:54–63; 13:3–20.

Frenier further discloses a treatment mixture including pH modifiers. *Id.* at 5:56–63 (“pH control agents”). Frenier explains that pH of the treatment fluid can be controlled by an organic acid or a mineral acid. *Id.* at 4:57–60. Frenier also provides examples of treatment mixtures comprising 0.1% to 15% by weight pH modifier. *Id.* at 6:40–45, and composition additives, such as solvents, comprising between 0.1–10% and 0.1–5% by weight. *See id.* at 6:25–45.

Accordingly, Ayers and Frenier, informed by the knowledge of a POSITA, discloses every limitation of dependent claim 7.

8. Dependent Claim 8

- a. “The method of claim 1, wherein the metal-polymer complex remediation mixture comprises between 0.1% and about 60% by weight metal complexing agent.”**

Ayers discloses a formulation containing a metal complexing agent having a concentration between about 0.2 and about 5.0 percent by weight, which falls squarely within the claimed range. Ex. 1004 at 36:27–29. Similarly, Frenier discloses a formulation containing a metal complexing agent having a concentration between 1% and 30% by weight, which falls squarely within the range set forth in Dependent Claim 8. Ex. 1005 at 6:25–47.

Accordingly, Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Dependent Claim 8

9. Dependent Claim 9

- a. **“The method of claim 1, wherein the metal-polymer complex remediation mixture further comprises a mutual solvent and an additive selected from the group consisting of acid inhibitor, corrosion inhibitor, scale inhibitor, clay control additive, surfactant, biocide, and mixtures thereof.”**

Frenier discloses a treatment mixture that may include a mutual solvent, such as one comprising a glycol ether, (Ex. 1005 at 6:14–15) and one or more additives, including surfactants, corrosion inhibitors, stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. *See* Ex. 1005 at 5:56–62.

Accordingly, Frenier discloses a remediation mixture further comprising a mutual solvent and an additive selected from a group including surfactants and corrosion inhibitors.

10. Dependent Claim 10

- a. **“The method of claim 1, wherein the metal-polymer complex remediation mixture comprises: between 1% and 95% by weight metal complexing agent;”**

Petitioner incorporates herein its statements and arguments above with respect to Ayers’s and Frenier’s disclosure of Element 1[a].

b. “between 1 and 10,000 parts per million (ppm) of a corrosion inhibitor;”

Frenier discloses a treatment mixture having one or more additives, explicitly including corrosion inhibitors. Ex. 1005 at 5:56–65. Frenier provides examples of treatment mixtures containing additives, such as corrosion inhibitors, at a concentration between 0.1–10% and 0.1–5% by weight. *Id.* at 6:25–45.

A POSITA at the time of filing would understand that a treatment mixture containing additives, including a corrosion inhibitor, at a concentration between 0.1–10% and 0.1–5% by weight discloses a treatment mixture containing between 1 and 10,000 ppm of a corrosion inhibitor. Ex. 1003 at ¶ 258. Even if Frenier doesn't explicitly mention 1-10,000 ppm for the corrosion inhibitor, disclosing a broader range that includes it suggests that such concentrations are encompassed by the invention. *Id.* at ¶ 259.

Further, as noted by the Examiner, due to the absence of evidence of the criticality of the use of corrosion inhibitors, “it would have been obvious to one having ordinary skill in the art before the effective filing date of the claimed invention to include an amount of...corrosion inhibitor...as desired and when needed in order to prevent any corrosion...within the well and/or formation.” Ex. 1002 at pp. 194–95

c. “between 1 and 10,000 parts per million (ppm) of a biocide;”

As mentioned above, Frenier discloses a treatment mixture including the use of various additives, and many “such additives are well known in the art.” *Id.* at 5:56–63. Indeed, the use of biocides in matrix acidizing treatment solutions was well known in the art prior to the effective date of the ’282 Patent, and it would have been at least obvious to a POSITA to include such additives into the treatment mixture to control microbial growth. Ex. 1003 at ¶ 261. Ayers also explicitly discloses the use of biocides in the treatment mixture. Ex. 1004 at 24:36–39.

As noted by the Examiner, due to the absence of evidence of the criticality of the use of biocide within the amounts as claimed, it would have been obvious to a POSITA before the effective filing date of the ’282 Patent to include an amount of biocide as desired and when need to prevent bacterial growth within the well and/or formation as based on the actual conditions encountered in the formation and desired treatments to provide thereto. Ex. 1002 at pp. 194–95; Ex. 1003 at ¶ 263.

d. “between 1 and 10,000 parts per million (ppm) of a colloidal silica deposition inhibitor;”

Frenier discloses a treatment mixture including the use of various additives. *Id.* at 5:56–63 (“[A]dditives are well known in the art.”). Indeed, the use of colloidal silica deposition inhibitors in matrix acidizing treatment solutions was well known in the art prior to the effective date of the ’282 Patent, and it would have been at least obvious to a POSITA to include such additives into the treatment mixture to control

colloidal silica deposition. Ex. 1003 at ¶ 265. For instance, Ayers explains that “[t]he formation of larger particle blockages that may include precipitates, flocculants and/or **colloids** with clays, minerals, paraffins, asphaltenes, and condensates also can lead to clogging and reduction of oil and gas recovery from subterranean formations.” Ex. 1004 at 5:41–47 (emphasis added).

Moreover, as noted by the Examiner, due to the absence of evidence of the criticality of the use of colloidal silica deposition inhibitors within the amounts as claimed, it would have been obvious to a POSITA before the effective filing date of the ’282 Patent to include an amount of colloidal silica deposition inhibitor as desired and when need to prevent colloidal silica buildup within the well and/or formation as based on the actual conditions encountered in the formation and desired treatments to provide thereto. *See* Ex. 1002 at pp. 194–95; Ex. 1003 at ¶ 267.

e. “between 1 and 500 gallons mutual solvent per thousand gallons (gpt) of water;”

As discussed above, Frenier discloses a treatment mixture including various additives, including a mutual solvent. Frenier provides examples of treatment mixtures containing additives, such as mutual solvents, at a concentration between 0.1–10% and 0.1–5% by weight. *See id.* at 6:25–45.

A POSITA at the time of filing would understand that a treatment mixture containing additives, including a mutual solvent, at a concentration between 0.1–10% and 0.1–5% by weight discloses a treatment mixture containing between 1 and

500 gallons of mutual solvent per thousand gallons of water. Even if Frenier doesn't explicitly mention said range, disclosing a broader range that includes it suggests that such concentrations are encompassed by the invention.

Moreover, as noted by the Examiner, due to the absence of evidence of the criticality of the use of corrosion inhibitors, “it would have been obvious to one having ordinary skill in the art before the effective filing date of the claimed invention to include an amount of...mutual solvent...as desired and when needed.” Ex. 102 at pp. 194–95.

11. Dependent Claim 11

- a. “The method of claim 1, wherein the metal-polymer complex remediation mixture further comprises a pH modifier.”**

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers’s disclosure of Dependent Claim 11.

Further, Frenier discloses a treatment mixture having additives, including a pH modifier. Ex. 1005 at 5:56–63 (“pH control agents”).

Ayers, alone or in combination with Frenier, disclose wherein the metal polymer complex remediation mixture further comprises a pH modifier.

12. Dependent Claim 12

- a. “The method of claim 11, wherein the metal-polymer complex remediation mixture further comprises between 0.1 and 30% of one or more acids as a pH modifier.”**

Petitioner incorporates herein its statements and arguments above from Dependent Claim 11 and the arguments from Ground I with respect to Ayers's disclosure of Dependent Claim 12.

Moreover, Frenier explicitly discloses treatment mixtures comprising 0.1% to 15% by weight pH modifier. *Id.* at 6:40–45, and composition additives, such as solvents, comprising between 0.1–10% and 0.1–5% by weight. *See id.* at 6:25–45.

Ayers alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Dependent Claim 12.

13. Dependent Claim 13

- a. “The method of claim 12, wherein the acid is selected from the group consisting of hydrochloric acid, aqua regia, arsenic acid, boric acid, carbonic acid, chloric acid, chromic acid, fluoroantimonic acid, fluoroboric acid, fluorosulfuric acid, fulminic acid, hexafluorophosphoric acid, hexafluorosilicic acid, hydrobromic acid, hydrofluoric acid, hydrogen iodide, hypochlorous acid, hypofluorous acid, hypophosphoric acid, iodic acid, nitric acid, nitrosyl-O-hydroxide, nitrous acid, orthocarbonic acid, perchloric acid, permanganic acid, perrhenic acid, pertechnetic acid, phosphoric acid, silicic acid, sulfuric acid, thiocyanic acid, titanilic acid, tungstic acid, xenic acid, and mixtures thereof.”**

Petitioner incorporates herein its statements and arguments above from Dependent Claim 12 and the arguments from Ground I with respect to Ayers's disclosure of Dependent Claim 13.

Moreover, Frenier explains that pH of the treatment fluid "can be controlled by organic acid, a mineral acid, or a base." *Id.* at 4:57–60. For instance, Frenier identifies "a non-oxidizing mineral acid, such as **hydrochloric acid, hydrofluoric acid**, or a mixture thereof." *Id.* at 4:18–20 (emphasis added).

Ayers alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Dependent Claim 13.

14. Dependent Claim 14

- a. **"The method of claim 12, wherein the acid is hydrochloric acid."**

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers's disclosure of Dependent Claim 13.

15. Dependent Claim 15

- a. **"The method of claim 1, wherein the injecting operation further comprises: alternately injecting a first amount of metal-polymer complex remediation mixture and a second amount of a diverting mixture into the well."**

Ayers discloses that "[p]olymers such as xanthan gum, guar gum, and hydrolyzed polyacrylamide can be added to Component A or optionally can be injected in a polymer drive **following** the addition of the...composition according to

an embodiment of the present invention.” Ex. 1004 at 30:42–48 (emphasis added). A POSITA would have found the use of the aforementioned polymers as diverting materials to be obvious. Ex. 1003 at ¶ 285. These polymers are well-established and documented materials used in the relevant industry for diversion purposes. Ex. *Id.* at ¶ 286. Their function of creating temporary downhole plugs or increasing fluid viscosity to influence flow paths is common knowledge within the field. *Id.* at ¶ 287. Moreover, a POSITA would recognize that many other known diverting materials exist. *Id.* at ¶ 288.

Further, Frenier discloses that, during the matrix acidizing process, “[p]ackers can be used in the wellbore to control the formation zones into which the treatment fluid is injected from the wellbore.” Ex. 1005 at 7:27–29. It would have been obvious to a POSITA to alternately inject the treatment mixture and a diverting mixture to better control the placement of the treatment fluid into the formation zones and avoid so-called “thief zones.” Ex. 1003 at ¶ 290.

Moreover, Frenier incorporates by reference in its entirety U.S. Pat. No. 5,203,413 to Zerhboub, entitled “Product and Process for Acid Diversion in the Treatment of Subterranean Formations.” (“Zerhboub”). Ex. 1005 at 7:46–48; Ex. 1012. Zerhboub discloses:

[A] treatment fluid injected into a subterranean formation is **diverted from a zone of relatively high permeability to a zone of relatively lower permeability** by a process of **first injecting a foam** comprising an inert fluid, a gas

and at least one surfactant and **subsequently injecting a treatment fluid** comprising an active fluid and at least one surfactant used in the preceding foam injection step. This sequential treatment is repeated as necessary...

Ex. 1012 at 1:52–59 (emphasis added). Zerhboub explains that the injected foam is a diverting mixture. *Id.* at 4:1–5; 4:36–37 (“The principle of diversion by means of a foam is also known.”).

Ayers, alone or with Frenier, as informed by the knowledge of a POSITA discloses Dependent Claim 15.

16. Dependent Claim 16

- a. **“The method of claim 1, wherein the injecting operation further comprises: injecting the metal-polymer complex remediation mixture until pressure within the well reaches a predetermined target that does not cause substantial fracturing of the formation.”**

Ayers discloses that “[t]he composition may be introduced into the wellbore or subterranean formation...at a rate and **pressure less than the reservoir hydraulic fracture pressure.**” Ex. 1004 at 36:65–37:3 (emphasis added).

Further, in Example 6 of Ayers, where the disclosed composition was used to “remediate biological, clay and other blockages”—and not for the purpose of fracturing—the composition was introduced into the well bore and “[a]ll injection wells were shut in around 750–790 psi surface pressure.” *Id.* at 47:9–10; 47:43–45. In that example, the mixture was introduced into the wellbores until a predetermined

target pressure was achieved, which was adequate for remediating blockages in the formation without causing substantial fracturing. While the injection of the treatment mixture in that example ceased upon reaching a predetermined surface pressure, a POSITA would recognize that surface pressure can provide information indicative of well pressure. Ex. 1003 at ¶ 295.

Moreover, in Example 6, it is disclosed that along with surface pressure, downhole reservoir pressure was also being monitored, noting that surface pressure eventually adjusted to be “approximately equal to downhole reservoir pressures.” *See* Ex. 1004 at 47:54–62.

Similarly, Frenier discloses that the injection step of the treatment mixture disclosed therein may be “performed at a fluid pressure that is **less than the minimum in situ rock stress....**” Ex. 1005 at 4:49–52.

A POSITA would have found it obvious to inject a remediation mixture into a well until the pressure within the well reaches a predetermined target pressure that avoids substantial fracturing of the formation. Ex. 1003 at ¶ 298. A POSITA would understand the detrimental effects of exceeding the formation’s fracture pressure, including the damage it could do to the wellbore, creation of unwanted flow paths, and its negative impact on treatment effectiveness. *Id.* at ¶ 299. Monitoring well pressure during treatment is a well-established practice in the relevant industry, and a POSITA would be aware of the importance of such monitoring, especially to

prevent the detrimental effects mentioned earlier. *Id.* A POSITA would also recognize the established correlation between increasing wellbore pressure and the risk of fracturing the formation and, as such, it would have been obvious to a POSITA to select a target pressure that would not cause substantial fracturing of the formation. *Id.* at ¶ 300.

Ayers, alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses this dependent claim.

17. Dependent Claim 17

- a. **“The method of claim 1, wherein the injecting operation further comprises: injecting the metal-polymer complex remediation mixture until the pressure within the well reaches a predetermined target pressure calculated based on the fracture pressure of the subterranean formation; and upon reaching the target pressure, shutting in the well.”**

Ayers discloses that “[t]he composition may be introduced into the wellbore or subterranean formation...at a rate and **pressure less than the reservoir hydraulic fracture pressure.**” Ex. 1004 at 36:65–37:3 (emphasis added).

Ayers also provides examples that disclose this limitation. For instance, Example 3 of Ayers discloses the use of a metal-polymer complex remediation mixture for the purpose of “[c]ausing secondary porosity and increased permeability.” *Id.* at 44:62–65. In that example, the treatment mixture was allowed to gravity flow into the wellbore before a water chase was pumped behind the

mixture to displace the mixture into the subterranean formation. *See id.* at 45:6–11. Ayers explains that “[t]he water chase was pumped at a rate of 2 to 8 gpm at a pressure of 6 psi to 100 psi; the rates and **pressures remained well below reservoir fracturing pressures.**” *Id.* at 45:11–14 (emphasis added).

In another instance, as detailed in Example 6, a metal-polymer complex remediation mixture was injected into wells and into the subterranean formation for the purpose of “remediat[ing] biological, clay and other blockages,” *Id.* at 47:9–12. The injection process involved pumping the mixture into the wellbore and displacing the mixture into subterranean formation using a water chase, after which “[a]ll injection wells **were shut in at around 750–790 psi surface pressure.**” *Id.* at 47:43–45 (emphasis added). Accordingly, Ayers discloses every limitation of claim 17.

Also, Frenier discloses the injection step “performed at a fluid pressure that is **less than the minimum in situ rock stress....**” Ex. 1005 at 4:49–52. As such, Frenier discloses reaching a predetermined target pressure calculated based on the fracture pressure of the formation, the calculation taking into account, for example, in situ rock stress, a parameter closely related to formation fracture pressure.

Furthermore, Zerhoub (incorporated into Frenier) provides that the process disclosed therein “includes a shut-in step between each sequential foam injecting and treatment fluid injecting step.” Ex. 1012 at 1:63–65.

A POSITA, prior to the effective date of the '282 Patent, would have found it obvious to inject a remediation mixture into a well until the pressure reaches a predetermined target pressure calculated based on the fracture pressure of the formation, and then shut-in the well, in view of Ayers and Frenier. Ex. 1003 at ¶ 309.

Pressure monitoring is a standard practice during formation treatments, and a POSITA understands the importance of fracture prevention due to its detrimental effects. Ex. 1003 at ¶ 310. A POSITA would recognize that the fracture pressure of a formation is a critical parameter to consider when injecting fluids downhole. *Id.* at ¶ 311 A POSITA would find it obvious to calculate a target pressure based on the formation's fracture pressure, and to shut-in the well after reaching the calculated target pressure. *Id.* at ¶ 312.

18. Dependent Claim 18

- a. **“The method of claim 1, wherein the injecting operation further comprising: monitoring at least one of pH and well pressure during the maintaining operation; and”**

Ayers discloses this limitation. In Example 6, Ayers notes that after the injection step, monitoring of, at least, well pressure occurred during the maintaining operation:

The morning following injector well treatments (12 to 24 hrs after injector well treatments, then shut in), surface pressures at the injector wells were 350 for injector #1, 380

for #2 and 300 for #3. **These pressures were approximately equal to downhole reservoir pressures....**

Id. at 47:54–62 (emphasis added). As such, Ayers discloses monitoring well pressure during the maintaining operation.

Frenier discloses the use of “pressure transient analysis” during the maintaining operation: “Pressure transient analysis is a common technique for estimating the extent of damage.” Ex. 1005 at 6:49–54. A POSITA, prior to the effective date of the ’282 Patent, would have known that pressure transient analysis includes monitoring well pressure over time during the maintaining operation. Ex. 1003 at ¶ 315.

Monitoring both pH and well pressure during treatment and formation contact with the mixture is standard practice in the relevant industry. Similarly, monitoring well pressure helps identify potential pressure buildups that could lead to unwanted fracturing. Finally, it is understood in the industry that monitoring both parameters allows for early detection of potential issues that could compromise wellbore integrity and worker safety. Ex. 1003 at ¶ 317.

b. “initiating the removing operation based on results of the monitoring operation.”

Ayers discloses this limitation. More particularly, Ayers discloses, in Example 3, that:

The pressure in the test wellbore was observed to begin rising steadily after 2 to 3 hours following treatment injection...Gas was continually observed...over the next 30 hours, at which time poly tubing was lowered to the bottom of the wellbore and clean water was pumped into the wellbore at sufficient rate to flush solids from the wellbore and onto the ground surface surrounding the test wellbore.

Ex. 1004 at 45:23–33. Accordingly, Ayers discloses initiating the removing operation based on the results of the monitoring operation.

Moreover, a POSITA, before the effective date of the '282 Patent, would have found it obvious to initiate the removing operation based on the results of a monitoring operation. In the relevant industry, monitoring pressure and pH during and after treatment is a well-established practice. Ex. 1003 at ¶ 319. A POSITA would understand that fluctuations in pressure or pH observed during monitoring operations could indicate formational changes, prompting the POSITA to consider initiating removal procedures. *Id.* at ¶ 320.

19. Dependent Claim 19

- a. “The method of claim 1, wherein the contact time is between about 4 hours and about 4 days.”**

Petitioner incorporates herein its statements and arguments above from Ground I with respect to Ayers’s disclosure of Dependent Claim 19..

Similarly, Frenier discloses that “[a]fter the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow

more complete reaction between the acid and the formation material.” Ex. 1005 at 7:30–34. Frenier also explains that “shut-in time... must be determined for each particular treatment [but] a well-treatment designer of ordinary skill is familiar with the essential features of matrix acidizing treatments.” *Id.* at 7:39–46. Here, a POSITA would recognize the optimal contact time by experimentation and trial and error since it has been held, “it is not inventive to discover the optimum or workable ranges by routine experimentation.” *In re Aller*, 220 F.2d at 456; Ex. 1003 at ¶ 324.

As such, Ayers, either alone or in combination with Frenier, discloses the limitation wherein the contact time is between four (4) hours and about four (4) days.

20. Dependent Claim 20

a. “The method of claim 19, wherein the contact time is between 1 to 4 days.”

Petitioner incorporates its arguments for Dependent Claim 19, above. For the reasons discussed above, Ayers, either alone or in combination with Frenier, discloses the limitation wherein the contact time is between one (1) day to four (4) days.

21. Independent Claim 21

a. “Element 21[Preamble]: “A method for remediating a subterranean-formed metal-polymer complex in an existing, low-producing well in a hydrocarbon-bearing subterranean shale formation, comprising:”

Petitioner incorporates its arguments concerning Element 1[Preamble], above. To the extent the preamble is limiting, Ayers discloses this limitation. Ayers discloses “a method of using a fluid formulation for increasing flow, production, or recovery of oil and gas hydrocarbons from a subterranean formation.” Ex. 1004 at Abstract. The method in Ayers aims to alleviate reductions in the productivity of pre-existing oil and gas wells in subterranean formations. Ex. 1003 at ¶ 331.

b. Element 21[a] “observing a well production decrease;”

Ayers discloses, with regards to Example 6, that “[p]rior to this treatment example, water injection/extraction wells were continuously operating, with a **reported** increase in injection well head pressure and decreased volumetric injection flowrates, with a **decreasing and low yield of oil production in the extraction wells over time.**” *Id.* at 47:17–22 (emphasis added); *see also id.* at 47:25–28 (“It was **reported by the well operators** that there had been a **decrease in volumetric injection flowrates, with a decreasing and low yield of oil** at less than 1.3 BOPD.”) (emphasis added). As such, Ayers discloses the limitation of observing a well production decrease, as claimed in Element 21[a].

c. Element 21[b] “providing a metal-polymer complex remediation mixture containing between 1% and 95% by weight metal complexing agent;”

Petitioner incorporates its statements and arguments concerning Element 1[a], above. Ayers and Frenier disclose every limitation of Element 21[b]. Ex. 1003 at ¶¶ 335–348.

- d. Element 21[c] “injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the subterranean shale formation until at least some of the metal-polymer complex remediation mixture contacts the subterranean-formed metal-polymer complex, wherein the subterranean-formed metal polymer complex forms from a previously injected fracturing fluid and the metal of the subterranean-formed metal-polymer complex includes metal naturally present within the subterranean formation;”**

Petitioner incorporates its statements and arguments concerning Element 1[b], above. Ayers and Frenier disclose every limitation of Element 21[c]. Ex. 1003 at ¶¶ 349–356.

- e. Element 21[d] “maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex for a contact time of between about 1 minute and about 100 days, thereby allowing the metal complexing agent to cause the subterranean-formed metal-polymer complex to dissociate and dissolve, thereby creating a low viscosity flow back fluid comprising the spent metal-polymer complex remediation mixture and the metal-polymer complex components; and”**

Petitioner incorporates its statements and arguments concerning Element 1[c], above. Ayers and Frenier disclose every limitation of Element 21[d]. Ex. 1003 at ¶¶ 357–368.

- f. Element 21[e] “removing the low viscosity flow back fluid from the well after the contact time, thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method.”**

Petitioner incorporates its statements and arguments concerning Element 1[d], above. Ayers and Frenier disclose every limitation of Element 21[e]. Ex. 1003 at ¶¶ 369–374. The additional words “relative to the hydrocarbon production immediately prior to performance of the method” are superfluous given that “improvements” are always measured to prior performance. *Id.* at ¶ 373.

22. Dependent Claim 22

- a. “22. The method of claim 21, wherein the injecting operation further comprises: alternately injecting a first amount of metal-polymer complex remediation mixture and a second amount of a diverting mixture into the well.”**

Ayers discloses this limitation. Ayers discloses that “[p]olymers such as xanthan gum, guar gum, and hydrolyzed polyacrylamide can be added to Component A or optionally can be injected in a polymer drive **following** the addition of the...composition[.]” *Id.* at 30:42–48 (emphasis added). A POSITA, prior to the

effective date of the '282 Patent, would find the use of such polymers to be obvious. Ex. 1003 at ¶ 376. These polymers are well-established and documented materials used in the relevant industry for diversion purposes. *Id.* at ¶ 377. Their function of creating temporary downhole plugs or increasing fluid viscosity to influence flow paths is common knowledge within the field. *Id.* at 378. Moreover, a POSITA would recognize that the brief list of xanthan gum, guar gum, and hydrolyzed polyacrylamide are just a few examples within a broader category of known diverting materials. *Id.* at ¶ 379.

Further, Frenier discloses that, during the matrix acidizing process, “[p]ackers can be used in the wellbore to control the formation zones into which the treatment fluid is injected from the wellbore.” Ex. 1005 at 7:27–29. In light of that disclosure, it would have been obvious to a POSITA to alternately inject the treatment mixture and a diverting mixture to better control the placement of the treatment fluid into the formation zones and avoid so-called “thief zones.” Ex. 1003 at ¶ 381.

Moreover, Frenier incorporates by reference in its entirety U.S. Pat. No. 5,203,413 to Zerhboub, entitled “Product and Process for Acid Diversion in the Treatment of Subterranean Formations.” (“Zerhboub”). *See id.* at 7:46–48; Ex. 1012 Zerhboub discloses alternately injecting a first amount of treatment mixture and a second amount of a diverting mixture into the well. Specifically, Zerhboub discloses:

a treatment fluid injected into a subterranean formation is
diverted from a zone of relatively high permeability to

a zone of relatively lower permeability by a process of **first injecting a foam** comprising an inert fluid, a gas and at least one surfactant and **subsequently injecting a treatment fluid**... This sequential treatment is repeated as necessary utilizing the increasing volumes of foam in each subsequent foam injecting step.

Ex. 1012 at 1:52–59 (emphasis added). Zerhboub explains that the injected foam is a diverting mixture. *Id.* at 4:1–5; 4:36–37 (“The principle of diversion by means of a foam is also known.”).

Accordingly, Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA discloses this dependent claim.

VIII. CONCLUSION

Petitioner respectfully submits there is a reasonable likelihood it will prevail in establishing that the Challenged Claims are unpatentable and requests that trial be instituted.

Respectfully submitted,

Date: April 18, 2024

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Patent 10,697,282

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CERTIFICATION OF WORD COUNT

This Petition complies with the type-volume limitations as mandated in 37 C.F.R. § 42.24, totaling 13,959 words. Counsel has relied upon the word count feature provided by Microsoft Word.

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CERTIFICATE OF SERVICE

I hereby certify pursuant to 37 C.F.R. §§ 42.6(e) and 42.105 that on this 18th day of April 2024, a true and correct copy of the foregoing Petition for *Inter Partes* Review of U.S. Patent 10,697,282 was served via Federal Express upon the following counsel of record for Patent Owner Flex-Chem Holding Company, Inc.:

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UNITED STATES

I further hereby certify that on this 18th day of April, 2024, a true and correct copy of the foregoing Petition for Inter Partes Review of U.S. Patent 10,697,282 was served via email upon the following litigation counsel for Patent Owner Flex-Chem Holding Company, Inc.:

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