

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

SHELL OIL COMPANY,
Petitioner,

v.

EXXONMOBIL RESEARCH AND ENGINEERING COMPANY,
Patent Owner.

Case IPR2016-00009
Patent 6,923,949 B1

Before GRACE KARAFFA OBERMANN, *Vice Chief Administrative
Patent Judge*, LORA M. GREEN and ELIZABETH M. ROESEL,
Administrative Patent Judges.

ROESEL, *Administrative Patent Judge*.

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

In this *inter partes* review, instituted pursuant to 35 U.S.C. § 314, Shell Oil Company (“Petitioner”) challenges the patentability of claims 1–4 of U.S. Patent No. 6,923,949 B1 (Ex. 1001, “the ’949 patent”), assigned to ExxonMobil Research and Engineering Company (“Patent Owner”).

We have jurisdiction under 35 U.S.C. § 6. This final written decision is issued pursuant to 35 U.S.C. § 318(a) and 37 C.F.R. § 42.73.

For the reasons that follow, we determine that Petitioner has not met its burden to show by a preponderance of the evidence that claims 1–4 of the ’949 patent are unpatentable.

I. BACKGROUND

A. *Procedural History*

On October 19, 2015, Petitioner filed a Petition requesting *inter partes* review of claims 1–4 of ’949 patent. Paper 2 (“Pet.”). On January 29, 2016, Patent Owner filed a Preliminary Response. Paper 9 (“Prelim. Resp.”).

On April 5, 2016, we instituted *inter partes* review of claims 1–4. Paper 10 (“Institution Decision” or “Dec.”).

On April 19, 2016, Petitioner filed a request for rehearing of our Institution Decision. Paper 14. On May 17, 2016, we denied rehearing. Paper 22 (“Rehearing Decision” or “Rhg. Dec.”)

On June 27, 2016, Patent Owner filed a Patent Owner Response. Paper 25 (“PO Resp.”). On September 16, 2016, Petitioner filed a Reply to Patent Owner’s Response. Paper 30 (“Pet. Reply”).

With the Petition, Petitioner filed a Declaration of Raul F. Lobo, Ph.D. (Ex. 1004) and a Declaration of David Cooper (Ex. 1006). Patent Owner cross-examined Mr. Cooper and Dr. Lobo on June 2 and 3, 2016,

respectively, and filed transcripts of the deposition testimony as Exhibits 2009 and 2010, respectively.

With the Patent Owner Response, Patent Owner filed a Declaration of John B. Higgins, Ph.D. Ex. 2013. Petitioner cross-examined Dr. Higgins on August 18, 2016, and filed a transcript of the deposition testimony as Exhibit 1025.

With the Reply, Petitioner submitted a Reply Declaration of Dr. Lobo. Ex. 1026. On October 18, 2016, Patent Owner again cross-examined Dr. Lobo and filed a transcript of the deposition testimony as Exhibit 2017. On November 7, 2016, Patent Owner filed a Motion for Observations on the Cross-Examination of Dr. Lobo. Paper 35 (“PO Obs.”). On November 21, 2016, Petitioner filed a Response to Patent Owner’s Motion for Observations on the Cross-Examination of Dr. Lobo. Paper 39 (“Pet. Resp.”).

An oral hearing was held December 16, 2016. A transcript of the hearing was entered in the record. Paper 44 (“Tr.”).

B. Related Proceedings

The parties identify no related matters pursuant to 37 C.F.R. § 42.8(b)(2). Pet. 1; Paper 5 (Patent Owner’s mandatory notices).

C. The ’949 Patent (Ex. 1001)

The ’949 patent relates to synthetic zeolites, which are useful for catalyzing hydrocarbon reactions. Ex. 1001, 1:15–17. According to the ’949 patent, “[c]ertain zeolitic materials are ordered, porous crystalline aluminosilicates having a definite crystalline structure as determined by X-ray diffraction.” *Id.* at 1:17–20. Examples of known synthetic aluminosilicates include: zeolite A, zeolite X, zeolite Y, zeolite ZK-5,

zeolite ZK-4, zeolite ZSM-5, zeolite ZSM-11, zeolite ZSM-12, ZSM-23, ZSM-48, and ZSM-50. *Id.* at 1:57–67, 2:14, 2:65.

The '949 patent is directed to one of these synthetic zeolites—ZSM-48. *Id.* at Abstract, 1:7–8. According to the '949 patent, ZSM-48 having a silica to alumina ratio ($\text{SiO}_2/\text{Al}_2\text{O}_3$) in the range 150 to 600 is known. *Id.* at 2:48–50. The '949 patent states that a lower silica to alumina ratio is desirable for certain types of catalysts, but prior attempts to synthesize such ZSM-48 have been unsuccessful and resulted in the formation of impurities, such as ZSM-50 and Kenyaite. *Id.* at 2:50–57.

According to the '949 patent, highly pure ZSM-48 crystals with a silica to alumina ratio of less than 150 result from the disclosed synthesis, which promotes incorporation of more aluminum into the framework of the crystals and suppresses the formation of impurities and fibrous morphology crystals. *Id.* at 3:38–51.

D. Illustrative Claim

The '949 patent includes 12 claims. The Petition challenges claims 1–4, which are directed to “pure phase ZSM-48 crystals.” Ex. 1001, 9:57. The Petition does not challenge claims 5–9, which are directed to a method for making ZSM-48 crystals, or claims 10–12, which are directed to a method for preparing pure phase ZSM–48.

Claim 1 is reproduced below with bracketing lettering and paragraph breaks added for ease of reference:

1. [a] Pure phase ZSM-48 crystals
[b] having a $\text{XO}_2/\text{Y}_2\text{O}_3$ ratio of less than about 150/1 and
[c] free from ZSM-50 and Kenyaite impurities
[d] having a diameter of less than about 1 micron and

[e] being substantially free of fibrous morphology.
Id. at 9:57–60.

E. Instituted Ground

We instituted *inter partes* review on a single ground:

Whether claims 1–4 are unpatentable under 35 U.S.C. § 102(b) as anticipated by Valyocsik, EP 0142317 A2, published May 22, 1985, Ex. 1002 (“Valyocsik”).

II. DISCUSSION

A. Claim Construction

In an *inter partes* review, claim terms in an unexpired patent are given their broadest reasonable interpretation in light of the specification of the patent in which they appear. 37 C.F.R. § 42.100(b); *Cuozzo Speed Techs., LLC v. Lee*, 136 S. Ct. 2131, 2144–46 (2016). Under that standard, we generally give claim terms their ordinary and customary meaning, as understood by a person of ordinary skill in the art in the context of the entire disclosure. *In re Translogic Tech., Inc.*, 504 F.3d 1249, 1257 (Fed. Cir. 2007). We construe claim terms only to the extent necessary to resolve the controversy. *Wellman, Inc. v. Eastman Chem. Co.*, 642 F.3d 1355, 1361 (Fed. Cir. 2011) (quoting *Vivid Techs., Inc. v. Am. Sci. & Eng’g, Inc.*, 200 F.3d 795, 803 (Fed. Cir. 1999)).

The Institution Decision provides the following claim constructions:

Claim term	Claim construction
“free from ZSM-50 and Kenyaite impurities”	free from ZSM-50 and Kenyaite impurities, as determined, for example, by x-ray diffraction (XRD) analysis or comparing an XRD pattern to Figure 1 of the '949 patent
“having a diameter of less than about 1 micron”	modifies “ZSM crystals”

Neither party identifies any error in the above constructions or proposes any modifications or additional constructions. Accordingly, for the reasons set forth in the Institution Decision, Dec. 5–8, we adopt these constructions for purposes of determining whether Petitioner has met its burden to establish unpatentability of the challenged claims of the '949 patent.

B. Person of Ordinary Skill in the Art

Petitioner asserts that a person of ordinary skill in the art would have a Ph.D. in chemistry, physical chemistry, chemical engineering, or a closely related field and at least five years' experience synthesizing and characterizing zeolites, including high-silica zeolites. Pet. 7–8 (citing Ex. 1004 ¶ 22).

Patent Owner does not specifically contest Petitioner's assertion regarding the level of ordinary skill in the art.

We accept Petitioner's assertion regarding the level of ordinary skill in the art, which is supported by Dr. Lobo's testimony. Ex. 1004 ¶ 22.

Based upon their stated qualifications, we consider both Dr. Lobo and Dr. Higgins qualified to opine from the viewpoint of a person of ordinary

skill in the art regarding the subject matter of the '949 patent. Ex. 1004 ¶¶ 6–17; Ex. 1005 (Lobo CV); Ex. 2012 ¶¶ 1–15; Ex. 2011 (Higgins CV).

C. Anticipation by Valyocsik

Petitioner contends that claims 1–4 of the '949 patent are anticipated by Valyocsik (Ex. 1002). Pet. 18–27.

1. Valyocsik (Ex. 1002)

Valyocsik discloses crystalline silicate ZSM-48 and a method for its preparation. Ex. 1002, Title, Abstract. According to Valyocsik, the disclosed method produces a zeolite product having “enhanced purity and catalytic activity when compared with ZSM-48 synthesized by known techniques.” *Id.* at 1.¹ Valyocsik states that one known method requires a silica to alumina ratio “of at least about 500 to avoid product contamination with other zeolites, notably ZSM-5 and ZSM-11.” *Id.* According to Valyocsik, the disclosed method produces “relatively pure ZSM-48” using a silica to alumina ratio of “much less than 500, for example as low as 100” in the reaction mixture. *Id.* at 3. Valyocsik notes that the silica to alumina ratio is “critical, since at less than about 100 a different crystalline silicate tends to form in place of the ZSM-48.” *Id.* Valyocsik describes the XRD pattern for ZSM-48 in tabular form, listing the characteristic peaks and their intensities. *Id.* at 2. According to Valyocsik, this pattern “distinguishes ZSM-48 from closely related materials such as ZSM-12.” *Id.* at 5–6.

Valyocsik discloses fifteen examples illustrating the synthesis of crystalline silicate ZSM-48. *Id.* at 10–14. Valyocsik introduces these

¹ Our citations to Valyocsik refer to the page numbers of the original document, rather than the page numbers added by Petitioner.

examples by stating that the disclosed method “enables pure ZSM-48 to be produced with a higher tetrahedral aluminum content (and hence much higher catalytic activity) than has been the case with conventional methods.” *Id.* at 10. The synthesis reaction and results of Examples 1–15 are summarized in Tables 1 and 2 of Valyocsik. *Id.* at 12, 14. Table 1 discloses the reaction mixture composition, reaction time, and resulting zeolite products, *id.* at 11–12, and Table 2 discloses the analytical compositions of these products, *id.* at 13–14.

2. *Parties’ Contentions*

Petitioner contends that Valyocsik discloses a ZSM-48 product meeting each limitation of claims 1–4 of the ’949 patent. Pet. 21. Petitioner relies on the express disclosures of Valyocsik for the limitations of claim 1, elements [a] through [c] and dependent claims 2–4, including “pure phase ZSM-48 crystals” that are “free from ZSM-50 and Kenyaite impurities.” *Id.* at 21–22, 26–27 (claim chart); Pet. Reply 1–6. Petitioner contends that the crystal diameter and morphology limitations of claim 1, elements [d] and [e], are inherently disclosed by Valyocsik. Pet. 23–27. According to Petitioner, Mr. Cooper reproduced Valyocsik Example 13 seven times. *Id.* at 23. Dr. Lobo reviewed the results of Mr. Cooper’s experiments and concluded that the product of Valyocsik Example 13 necessarily meets the crystal diameter and morphology limitations of claim 1. *Id.* (citing Ex. 1004 ¶¶ 77-102). In addition, Petitioner and Dr. Lobo assert that XRD patterns for Mr. Cooper’s reproductions of Valyocsik Example 13 (Ex. 1009) show “pure phase” ZSM-48 that is “free from ZSM-50 and Kenyaite impurities.” Ex. 1004 ¶ 96; Pet. Reply 7, 13–16; Ex. 1026 ¶¶ 19, 21.

Patent Owner contends that Petitioner fails to prove that Valyocsik discloses, either expressly or inherently, ZSM-48 that is “free from ZSM-50 and Kenyaite impurities,” as recited in claim element 1[c]. PO Resp. 1–2, 4, 11–14, 35. Relying on Dr. Higgins’ analysis of Petitioner’s XRD data (Ex. 1009), Patent Owner contends that the data do not show that Mr. Cooper’s reproductions of Valyocsik Example 13 were free from ZSM-50 and Kenyaite impurities. *Id.* at 3, 24–35 (citing Ex. 2013, ¶¶ 25–28, 33–41). Patent Owner further contends that, on cross examination, Dr. Lobo admitted that Petitioner’s XRD data (Ex. 1009) includes peaks consistent with the presence of ZSM-50. PO Obs. 4 (citing Ex. 2017, 61:1–75:2.)

3. *Legal Standard for Anticipation*

Anticipation is established when a single prior art reference describes, either expressly or inherently, each and every element of the claim. *In re Robertson*, 169 F.3d 743, 745 (Fed. Cir. 1999). “An anticipatory reference, however, need not duplicate word for word what is in the claims.

Anticipation can occur when a claimed limitation is ‘inherent’ or otherwise implicit in the relevant reference.” *Standard Havens Prod., Inc. v. Gencor Indus., Inc.*, 953 F.2d 1360, 1369 (Fed. Cir. 1991). In addition, to be anticipating, a prior art reference must be enabling and must describe the claimed invention sufficiently to have placed it in possession of a person of ordinary skill in the field of the invention. *Helifix Ltd. v. Blok-Lok, Ltd.*, 208 F.3d 1339, 1346 (Fed. Cir. 2000); *In re Paulsen*, 30 F.3d 1475, 1479 (Fed. Cir. 1994).

“[A]nticipation by inherent disclosure is appropriate only when the reference discloses prior art that must necessarily include the unstated limitation.” *Rexnord Indus., LLC v. Kappos*, 705 F.3d 1347, 1355 (Fed. Cir.

2013) (citation omitted). “To establish inherency, the extrinsic evidence ‘must make clear that the missing descriptive matter is necessarily present in the thing described in the reference.’” *Robertson*, 169 F.3d at 745 (quoting *Continental Can Co. v. Monsanto Co.*, 948 F.2d 1264, 1268 (Fed. Cir. 1991)). Inherency “may not be established by probabilities or possibilities. The mere fact that a certain thing may result from a given set of circumstances is not sufficient.” *In re Oelrich*, 666 F.2d 578, 581 (CCPA 1981). For example, where the practice of a prior art example sometimes, but not always, yields the claimed product, anticipation is not established. *Glaxo Inc. v. Novopharm Ltd.*, 52 F.3d 1043, 1047–48 (Fed. Cir. 1995).

4. Analysis

Our analysis focuses on the limitation of claim 1 that requires ZSM-48 crystals that are “free from ZSM-50 and Kenyaite impurities.” After considering the arguments and evidence presented by both parties, we find that Petitioner has not met its burden to prove that Valyocsik discloses, either expressly or inherently, ZSM-48 crystals that are free from ZSM-50 impurity. In view of this finding, we need not address whether Valyocsik discloses ZSM-48 crystals that are free from Kenyaite impurity.

Regarding the claim limitation, “free from ZSM-50 and Kenyaite impurities,” Petitioner advances two arguments: (1) that Valyocsik implicitly discloses this limitation, without the need to consider the evidence relating to Mr. Cooper’s reproductions of Example 13 (Pet. Reply 1–6; Tr. 10:11–11:4, 15:20–24); and (2) that the experimental evidence supports a finding that Valyocsik inherently discloses this limitation (Pet. Reply 7–17). We address each of these arguments below under the subheadings, “implicit disclosure” and “inherent disclosure.”

i. Implicit Disclosure

Petitioner does not contend that Valyocsik expressly discloses ZSM-48 crystals that are free from ZSM-50 impurity. Instead, relying on the testimony of Dr. Lobo, Petitioner contends that a person of ordinary skill in the art would have understood Valyocsik Examples 10–14 as disclosing ZSM-48 that is free from ZSM-50 impurity. Pet. 21–22 (citing Ex. 1004 ¶¶ 72–76); Pet. Reply 2–6; Ex. 1026 ¶¶ 3–6, 8.

As discussed in the Institution Decision, Dec. 10–12, Dr. Lobo bases his opinion on three kinds of data: (1) Valyocsik’s express disclosures regarding purity of the product and desire to avoid contamination with other zeolites and crystalline silicates (Ex. 1002, 1, 3, 10; Ex. 1004 ¶ 72); (2) data in Valyocsik Table 1 relating to Examples 1–15 (Ex. 1002, 12; Ex. 1004 ¶¶ 74–76); and (3) XRD patterns for Mr. Cooper’s reproductions of Valyocsik Example 13 (Ex. 1009; Ex. 1004 ¶ 96). In his reply declaration, Dr. Lobo opines that it would have been contrary to accepted scientific practice for Valyocsik to identify ZSM-48 as “pure,” if it contained ZSM-50, which Dr. Lobo asserts was known to be a potential impurity in ZSM-48. Ex. 1026 ¶¶ 3–6, 8.

Petitioner contends that the express disclosures of Valyocsik and the testimony of Dr. Lobo are sufficient to meet its burden of proof, without the need to consider the evidence relating to Mr. Cooper’s reproductions of Example 13. Pet. Reply 1–6; Tr. 10:11–11:4, 15:20–24. We disagree for several reasons.

First, as noted in the Rehearing Decision (Rhg. Dec. 6), Petitioner’s contention is not supported by Dr. Lobo’s opinion, which is based, in part, on Mr. Cooper’s experiments. Ex. 1004 ¶¶ 19, 77–93, 96. We decline

Petitioner's invitation to carve up Dr. Lobo's testimony by not relying on the portion that is based Mr. Cooper's reproductions of Valyocsik Example 13 and characterizing the balance as "unrebutted." *Cf.* Pet. Reply 2. The preponderance of the evidence standard requires us to consider the record as a whole, including **all** of the evidence relevant to a proposition of unpatentability. The XRD patterns for Mr. Cooper's reproductions of Example 13 (Ex. 1009) are relevant to whether Valyocsik discloses ZSM-48 crystals that are free from ZSM-50 impurity. The relevance of XRD analysis for determining the purity of crystalline material is undisputed. Ex. 1004 ¶¶ 39, 73; Ex. 1026 ¶¶ 12–14. Having placed the XRD patterns (Ex. 1009) in the record and relied upon them in the Petition and Lobo Declaration, Petitioner cannot now argue that we should not consider this evidence.

Second, we do not agree with Petitioner's characterization of Dr. Lobo's testimony as "unrebutted." Pet. Reply 2. Patent Owner submits the testimony of Dr. Higgins, who reviewed Petitioner's XRD patterns and reached a different conclusion from that stated in Dr. Lobo's initial declaration. *Compare* Ex. 2013 ¶¶ 33–41, *with* Ex. 1004 ¶ 96. Patent Owner also submits the deposition testimony of Dr. Lobo, who admitted on cross-examination that Petitioner's XRD data (Ex. 1009) includes peaks consistent with the presence of ZSM-50. Ex. 2017, 61:1–62:20, 67:22–75:2. Dr. Higgins' testimony and the deposition testimony of Dr. Lobo **are** a rebuttal to Dr. Lobo's opinion, which is based, in part, on an analysis of the XRD patterns of Exhibit 1009. Ex. 1004 ¶¶ 19, 96.

Under the circumstances of this case, we give weight to the expert testimony regarding what a person of ordinary skill in the art would have

understood from the express disclosures of Valyocsik, but we also give weight to the expert testimony based on an analysis of experimental data showing what Valyocsik actually discloses to a person of ordinary skill in the art. The latter category of expert testimony is relevant not only to inherency, but also to whether Valyocsik's disclosures are sufficient to put a person of ordinary skill in the art in possession of the claimed subject matter. *Helifix*, 208 F.3d at 1346; *In re Paulsen*, 30 F.3d at 1479.

Third, based on the full record developed during trial, we are not persuaded that Petitioner has shown that a person of ordinary skill in the art would have understood Valyocsik Examples 10–14 as disclosing ZSM-48 that is free from ZSM-50 impurity, without considering XRD data for these examples. The Petition relies on Valyocsik's identification of the product of Example 13 as “ZSM-48 (100%)” to argue that Example 13 meets the purity limitations of claim 1. Pet. 22 (citing Ex. 1002, 12, Table 1). At the hearing, however, Petitioner conceded that the “100%” notation in Valyocsik Table 1 refers to crystallinity, not purity, of the product. Tr. 12:24–13:10, 13:24–14:3.

Petitioner relies on additional disclosures in Valyocsik, including the statement that the disclosed method “enables pure ZSM-48 to be produced” (Ex. 1002, 10), Valyocsik's stated objective of avoiding contamination with other zeolites and crystalline silicates (*id.* at 1, 3), and Valyocsik Table 1, which discloses that Examples 8 and 15 resulted in the formation of crystalline silicates other than ZSM-48, but that the product of Examples 10–14 was ZSM-48 (*id.* at 12). See Ex. 1004 ¶¶ 72, 74–76.

Those disclosures must be viewed from the perspective of a person of ordinary skill in the art at the time of the invention. The evidence

establishes that such a person would rely on XRD analysis to determine the identity and purity of crystalline material, such as a zeolite. Ex. 1004 ¶¶ 39, 73; Ex. 1026 ¶¶ 12–14. Dr. Lobo explains that, by analyzing an XRD pattern, a person of ordinary skill in the art can identify the crystalline structure, e.g., ZSM-48, and determine whether any impurities are present. Ex. 1004 ¶ 39. In his reply declaration, Dr. Lobo explains how such an analysis would have been conducted. Ex. 1026 ¶¶ 12–14. According to Dr. Lobo, a person of ordinary skill in the art would analyze an XRD pattern for a synthesized material, first, to determine whether the intended zeolite was produced, and next, to determine whether the XRD pattern contains peaks that are inconsistent with a set of peaks known to be characteristic of the intended zeolite. *Id.* ¶ 12. Dr. Lobo explains that, if a person of ordinary skill in the art wanted to know whether a specific impurity is present in the synthesized material, he would consult XRD data for the impurity (a peak listing, XRD pattern, or both), identify peaks that are characteristic of the impurity, and then look for those peaks in the XRD pattern for the synthesized material. *Id.* ¶ 14.

Comparing Dr. Lobo's testimony regarding how XRD analysis is used to determine the presence or absence of impurities with his testimony regarding Valyocsik, we are not convinced that a person of ordinary skill in the art would conclude that Valyocsik Examples 10–14 are free from ZSM-50 impurity without considering XRD data for these examples. Dr. Lobo testifies that a person of ordinary skill in the art would have understood that Valyocsik used XRD analysis to determine the identity and purity of the examples. Ex. 1004 ¶ 73 (citing Ex. 1002, 13). There is no indication, however, that Valyocsik performed the steps that Dr. Lobo testifies a person

of ordinary skill in the art would perform if he wanted to know whether ZSM-50 is present as an impurity in the synthesized material. Ex. 1026 ¶ 14. Although Valyocsik mentions ZSM-5, ZSM-11, ZSM-12, and cristobalite as possible impurities in ZSM-48 (Ex. 1002, 3, 6, 12), there is no mention of ZSM-50. Nor is there any indication that Valyocsik consulted a peak listing or XRD pattern for ZSM-50. Cf. Ex. 1026 ¶ 14.

Valyocsik discloses a list of XRD peaks characteristic of ZSM-48 (Ex. 1002, 3), but does not disclose any XRD patterns for the products of Examples 10-14. According to Dr. Lobo, a person of ordinary skill in the art would have analyzed an XRD pattern for the synthesized material, not merely a peak listing, to determine whether impurities are present. Ex. 1026 ¶¶ 12–14. We find, therefore, that a person of ordinary skill in the art would not have concluded that Examples 10–14 are free from ZSM-50 impurity without considering XRD data for the examples.

Fourth, the cases relied upon by Petitioner are distinguishable. Pet. Reply 1–2 (citing *In re Baxter Travenol Labs.*, 952 F.2d 388 (Fed. Cir. 1991); *Arthrocare Corp. v. Smith & Nephew, Inc.*, 406 F.3d 1365 (Fed. Cir. 2005)). In each of these cases, the court relied upon expert testimony and/or party admissions to find that a person of ordinary skill in the art would have understood that a claim limitation not expressly disclosed in an asserted anticipatory reference was intrinsically present in the disclosed device. *Baxter*, 952 F.2d at 390; *Arthrocare*, 406 F.3d at 1373–74. Unlike this case, however, neither party in *Baxter* or *Arthrocare* relied on experimental evidence to support or contradict the expert testimony as to what a person of ordinary skill in the art would have understood from the asserted reference. Petitioner also cites *Helifix*, which held that there was insufficient evidence

that the asserted anticipatory reference was enabling. 208 F.3d at 1347. Like *Baxter* and *Arthrocare*, neither party in *Helifix* relied on experimental evidence. Accordingly, none of Petitioner's cited cases persuades us that we should decline to consider Petitioner's XRD data (Ex. 1009) or the related expert opinions.

Accordingly, we find that Petitioner has not shown by a preponderance of the evidence that Valyocsik implicitly discloses ZSM-48 that is free from ZSM-50 impurity.

ii. *Inherent Disclosure*

Petitioner argues that Mr. Cooper's experiments show that ZSM-48 produced according to Valyocsik Example 13 is necessarily free from ZSM-50. Pet. Reply 7, 15–16 (quoting Ex. 1026 ¶ 21).

After considering the arguments and evidence presented by both parties, we find that Petitioner has not shown by a preponderance of the evidence that Valyocsik Example 13 is necessarily free from ZSM-50. Our finding is supported by at least three categories of evidence: (1) Dr. Lobo's concession that Experiment 5 is an "exception" to his opinion that the XRD data for Mr. Cooper's reproductions of Example 13 show a ZSM-48 product that is free from ZSM-50 impurity (Ex. 1004 ¶ 96); (2) Dr. Higgins' analysis of the XRD data for Mr. Cooper's experiments (Ex. 1009) and conclusion that the data for Experiments 1–5 are consistent with the presence of ZSM-50 in the samples (Ex. 2013, ¶¶ 33–41); and (3) Dr. Lobo's concession on cross-examination that the XRD data for Mr. Cooper's Experiments 1, 3, and 5 include peaks consistent with the presence of ZSM-50 (Ex. 2017, 61:1–62:20, 67:22–75:2). We discuss each category of evidence below:

Dr. Lobo concedes that the XRD data for Mr. Cooper's Experiment 5 does not show a ZSM-48 product that is free from ZSM-50 impurity. Ex. 1004 ¶ 96; *see also id.* ¶ 97 ("the product of Experiment 5 . . . contained an impurity"). According to Dr. Lobo, "[a]ny of a number of experimental errors could have led to the formation of an impure product in Experiment 5." *Id.* ¶ 96. In his reply declaration, Dr. Lobo identifies three potential sources of inadvertent error that may have occurred while performing the experiments. Ex. 1026 ¶ 22.

Based on the full record developed during trial, we are not persuaded that that the XRD results for Mr. Cooper's Experiment 5 (Ex. 1009, page 8) are attributable to experimental error.

Dr. Lobo's conclusory reference to experimental error (Ex. 1004 ¶ 96) is not sufficiently supported by factual evidence. Although Dr. Lobo asserts that experimental error led to the formation of an impure product in Experiment 5 (*id.*), he does not identify anything in Mr. Cooper's procedures or analytical results that led him to that conclusion. In fact, Dr. Lobo states that, in preparing his declaration (Ex. 1004), he did not speak with Mr. Cooper. Ex. 2010, 7:21–24. We give little weight to Dr. Lobo's list of potential sources of inadvertent errors that "may have occurred" (Ex. 1026 ¶ 22) because the testimony is speculative, not supported by factual evidence relating to the actual conditions of Experiment 5, and inconsistent with Dr. Lobo's other opinions and the testimony of Mr. Cooper, as discussed below.

Based on his review of Mr. Cooper's declaration, laboratory notebook and analytical results, Dr. Lobo opines that, in Experiments 1–7, "Mr. Cooper accurately reproduced Valyocsik Example 13." Ex. 1004

¶¶ 77, 93, 94. Dr. Lobo makes no exception or qualification of this opinion with respect to Experiment 5.

Furthermore, Dr. Lobo bases his opinions on three types of analyses performed by Mr. Cooper: X-ray fluorescence chemical analysis (XRF), X-ray powder diffraction (XRD), and scanning electron microscopy (SEM). *Id.* ¶¶ 77, 94; Ex. 1006 ¶¶ 61, 64. Dr. Lobo discounts the XRD results for Experiment 5, attributing them to experimental error. Ex. 1004 ¶ 96. Yet Dr. Lobo relies on the XRF and SEM results for Experiment 5 (*id.* ¶¶ 95, 97–99, 101), even though the samples used in all three analyses (XRF, SEM, and XRD) were prepared in the same experiment (Ex. 1006 ¶¶ 61, 64), and the potential sources of error relate to sample preparation, rather than analysis of the samples (Ex. 1026 ¶ 22). Dr. Lobo provides no explanation for the disparate treatment of the analytical results for Experiment 5.

Turning to Mr. Cooper's testimony, he states that the reagents for Experiment 5 were obtained from the same sources identified in the description of Experiment 4. Ex. 1006 ¶ 56. Mr. Cooper further states that he selected the stoichiometry for Experiment 5 to match the ratios described in Valyocsik Example 13. *Id.* ¶ 58. According to his declaration, Mr. Cooper performed XRD analysis for Experiment 5 in the same manner as he performed XRD analysis for Experiments 1–4 and 6. *Compare id.* ¶ 64 (Experiment 5), *with id.* ¶¶ 22, 33, 44, 54, 74 (Experiments 1, 2, 3, 4, and 6, respectively). Accordingly, Mr. Cooper's testimony regarding Experiment 5 does not support that the XRD results are attributable to experimental error.

Viewing the record as a whole, the evidence relating to Experiment 5 is more consistent with a finding that Valyocsik Example 13 sometimes

produces ZSM-48 contaminated with ZSM-50, rather than with a finding of experimental error. As discussed below, the XRD results for Experiment 5 are consistent with those for Experiments 1 and 3, all three of which show peaks consistent with the presence of ZSM-50 in the samples.

Dr. Higgins analyzed the XRD pattern for ZSM-50 (Ex. 2001), the XRD pattern for ZSM-48 (Ex. 1001, Fig. 1), and the XRD patterns for Mr. Cooper's experiments (Ex. 1009) to determine whether the XRD patterns in Exhibit 1009 correspond to ZSM-48 that is free from ZSM-50. Ex. 2013 ¶¶ 19, 29, 33. The method used by Dr. Higgins was to look for an XRD peak at about $19^\circ 2\Theta$.² *Id.* ¶¶ 30–33. Dr. Higgins selected this peak because it is a medium intensity peak in the XRD pattern for ZSM-50 and is located where a peak for ZSM-48 is not present in Figure 1 of the '949 patent. *Id.* ¶ 31. According to Dr. Higgins, each of the XRD patterns for Mr. Cooper's Experiments 1–5 includes a peak at about $19^\circ 2\Theta$, as indicated by a red arrow in Exhibit 2003. *Id.* ¶¶ 34–41 (analyzing XRD patterns at pages 1–8 of Ex. 1009).³ Based on this analysis, Dr. Higgins concludes that

² The x-axis of the XRD patterns in Exhibits 1001 (Fig. 1), 1009, and 2001 is in units of 2Θ (two theta).

³ Exhibit 1009 includes a total of eleven XRD patterns, including two for each of Experiments 1–3 and 6 and one for each of Experiments 4, 5, and 7. Ex. 1009. Some of the XRD patterns were generated by the Analytical Services Group of Mr. Cooper's employer, PQ Corporation, and others were generated by Mr. Cooper using a different instrument. Ex. 1006 ¶¶ 1, 15, 20, 22, 29, 31, 33, 40, 42, 44, 54, 64, 74, 75, 77, 85, 87; Ex. 2017, 52:1–54:10. Exhibit 2003 corresponds to pages 1–8 of Exhibit 1009 and includes the XRD patterns for Experiments 1–5 annotated by Patent Owner to include a red arrow identifying a peak at about $19^\circ 2\Theta$ in each pattern. *See* Ex. 2013 ¶¶ 34–41; PO Resp. 34; Ex. 1025, 34:5–36:13.

each of the XRD patterns for Experiments 1–5 is “consistent with the presence of ZSM-50 in the sample.” *Id.*

Dr. Lobo was cross-examined regarding XRD patterns for the samples produced in Mr. Cooper’s Experiments 1, 3, and 5. Ex. 2017, 60:17–62:20, 67:22–75:2. Dr. Lobo was asked to compare the XRD patterns for the samples (red lines in Exhibits 1009 and 2003) with the XRD patterns for a ZSM-48 standard (blue lines in Exhibits 1009 and 2003). *Id.* at 60:17–61:9; see also Ex. 1006 ¶¶ 20, 42, 64 (in Exhibit 1009, XRD pattern for sample is plotted versus an “accepted standard” for ZSM-48).⁴ Regarding Experiment 5, Dr. Lobo testified that the XRD pattern for the sample has a “much more intense” peak at $19^{\circ} 2\theta$ compared with the standard and that the presence of this peak is consistent with the presence of ZSM-50 in the sample. *Id.* at 61:1–21. Similarly, regarding Experiments 1 and 3, Dr. Lobo testified that the red line (sample) has a “more intense” peak at $19^{\circ} 2\theta$ than the blue line (standard) and that this peak is consistent with the presence of ZSM-50 in the sample. *Id.* at 61:22–62:20. Dr. Lobo identified additional peaks in the XRD patterns for samples produced in Experiments 1, 3, and 5 that were not present in the XRD pattern for the ZSM-48 standard. Dr. Lobo testified that

⁴ We note that the blue lines in Exhibits 1009 and 2003 are XRD patterns for a standard ZSM-48 product and, like the XRD patterns for the samples, they were generated using two different instruments. Ex. 2017, 51:7–24. In other words, the blue lines in Exhibits 1009 and 2003 are not standard XRD patterns, but XRD patterns for a standard product generated using the same instruments as the XRD patterns for the samples. See *id.* (“All of the examples were measured against the same standard . . . but there are . . . two types of diffractometers. They are different and give two different [blue] lines.”).

XRD peaks at 2θ values of 7.98° , 19.11° , 20.6° , and 27.2° are consistent with the presence of ZSM-50 in each of the samples. *Id.* at 68:1–75:3.

Thus, Dr. Higgins’ analysis of the XRD data (Ex. 1009; Ex. 2013, ¶¶ 33–41) and Dr. Lobo’s admissions on cross-examination (Ex. 2017, 61:1–62:20, 67:22–75:2) support that at least three of Mr. Cooper’s experiments (Experiments 1, 3, and 5) resulted in ZSM-48 that was not necessarily free from ZSM-50.

Petitioner argues that Dr. Higgins’ analysis of Mr. Cooper’s XRD data is flawed because the peak he observed at $19^\circ 2\theta$ “may in fact be from ZSM-48, not ZSM-50.” Pet. Reply 15–17 (citing Ex. 1026 ¶ 26 and Ex. 1025 at 100:9–25, 101:14–104:6). That argument and the cited evidence are not persuasive for at least three reasons:

First, it is inconsistent with the legal standard for inherency, which requires Petitioner to show that the product of Valyocsik Example 13 is ***necessarily*** free from ZSM-50 impurity. *Robertson*, 169 F.3d at 745. Evidence that the product “may in fact be” impurity-free (Pet. Reply 16) is not sufficient. *Oelrich*, 666 F.2d at 581 (Inherency “may not be established by probabilities or possibilities.”).

Second, Petitioner’s argument is inconsistent with its position that the “proper standard” is “the standard of Figure 1 of the 949 patent.” Pet. Resp. 5. Petitioner’s argument is based on XRD patterns for ZSM-48 from various publications—not Figure 1 of the ’949 patent. Pet. Reply 15–16 (citing Ex. 1026 ¶ 21 and Exs. 1011, 1012, 1014). Furthermore, Petitioner does not direct us to any evidence suggesting that the “weak peak at about $19^\circ 2\theta$ ” in the XRD patterns from the cited publications (Pet. Reply 15–16) is not due to ZSM-50 impurity.

Third, we find that paragraph 21 of Dr. Lobo's reply declaration (Ex. 1026 ¶ 21) and the cited deposition testimony of Dr. Higgins (Ex. 1025 at 100:9–25, 101:14–104:6) are outweighed by the three categories of evidence discussed at pages 15–20 above. More specifically, we find that Dr. Lobo's concession regarding Experiment 5 (Ex. 1002 ¶ 96) and his admissions on cross-examination (Ex. 2017, 61:1–62:20) are more credible than his testimony in paragraph 21 of his reply declaration (Ex. 1026 ¶ 21). During cross-examination, Dr. Lobo was shown paragraph 21 of his reply declaration, and he read it into the record. Ex. 2017, 48:24–49:14, 58:22–59:1, 65:2–66:3. Nevertheless, when questioned about the XRD patterns for Mr. Cooper's Experiments 1, 3, and 5 (Exs. 1009, 2003, pages 1, 5 and 8), Dr. Lobo testified that a peak at $19^{\circ} 2\theta$ in is "consistent with the presence of ZSM-50," without mentioning that the peak could be attributable to ZSM-48. *Id.* at 61:1–62:20.

We also find that paragraph 21 of Dr. Lobo's reply declaration (Ex. 1026 ¶ 21) and the cited deposition testimony of Dr. Higgins (Ex. 1025 at 100:9–25, 101:14–104:6) deserve less weight than Dr. Higgins' analysis of the XRD data in his declaration (Ex. 2013, ¶¶ 33–41) and Dr. Lobo's admissions on cross-examination (Ex. 2017, 61:1–62:20, 67:22–75:2). This is because the former are based on a comparison between Mr. Cooper's XRD data (Exs. 1009, 2003) and publications unrelated to the '949 patent (Exs. 1011, 1012, 1014), and the latter are based on either: (1) a comparison with Figure 1 of the '949 patent (*see* Ex. 2013, ¶¶ 33–41), which Petitioner asserts is the "proper standard" (Pet. Resp. 5), or (2) a comparison between the red and blue lines in Mr. Cooper's XRD data (*see* Ex. 2017, 61:1–62:20, 67:22–75:2), which Dr. Lobo testified is a "conventional procedure" used by

a person of ordinary skill in the art to identify a synthesized material and assess its purity (Ex. 1026 ¶¶ 12, 13; Ex. 2017, 50:5–22).

Petitioner argues that Dr. Lobo’s testimony on cross-examination is “not relevant” because of the very different signal-to-noise ratio of Mr. Cooper’s XRD patterns as compared to Figure 1 of the ’949 patent. Pet. Resp. 5. That argument is not persuasive for the reason just stated: Dr. Lobo’s testimony establishes that the comparison he was asked to make on cross-examination (Ex. 2017, 61:1–62:20, 67:22–75:2) is a “conventional procedure” used by a person of ordinary skill in the art to identify a synthesized material and assess its purity (Ex. 1026 ¶¶ 12, 13; Ex. 2017, 50:5–22). Moreover, Petitioner cannot be heard to attack the relevance of its own evidence (Ex. 1009) that its expert relied upon to show an element of the claim (Ex. 1004 ¶ 96).

Petitioner acknowledges that Experiment 5 is an “exception” to its contention that Mr. Cooper’s reproductions of Valyocsik Example 13 show ZSM-48 that is free from ZSM-50. Pet. Reply 17. As discussed above, we are not persuaded that the results of Experiment 5 are attributable to experimental error. *See* pages 16–18, *supra*. Accordingly, Experiment 5 alone is enough to negate inherent anticipation of claim 1. *Robertson*, 169 F.3d at 745 (to establish inherency, the extrinsic evidence must show that the missing limitation is “necessarily present in the prior art in the thing described in the reference”); *Glaxo*, 52 F.3d at 1047–48 (inherent anticipation not established where experts’ reproduction of prior art example did not always yield the claimed product).

Accordingly, we find that Petitioner has not shown that Valyocsik inherently discloses ZSM-48 that is free from ZSM-50 impurity.

III. CONCLUSION

For all of the foregoing reasons, we find that Petitioner has not met its burden to show by a preponderance of the evidence that claims 1–4 of the '949 patent are unpatentable under 35 U.S.C. § 102(b) as anticipated by Valyocsik.

IV. ORDER

Accordingly, in consideration of the foregoing, it is hereby:

ORDERED that claims 1–4 of the '949 patent are held not unpatentable under 35 U.S.C. § 102(b) as anticipated by Valyocsik; and

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

IPR2016-00009
Patent 6,923,949 B1

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