

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In *Inter Partes* Reexamination of:)
BULL ET AL.) Examiner: DIAMOND, ALAN D
Reexamination Control No. 95/001,453) Group Art Unit: 3991
Patent No. 7,601,662) Confirmation No: 2755
Issued: October 13, 2009)
For: COPPER CHA)
ZEOLITE CATALYSTS)

Mail Stop Inter Partes Reexam
Central Reexamination Unit
Commissioner for Patents
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SECOND DECLARATION OF STANLEY ROTH, PH.D. UNDER 37 C.F.R. § 1.132

I, Stan Roth, do declare and say as follows:

1. I am currently the research group leader for diesel oxidation catalysts and soot filters for BASF Corporation, located in Iselin, New Jersey. I currently have 18 scientists who perform research into diesel oxidation catalysts and soot filters reporting to me. In 2005, I held the position of research group leader for NO_x control catalysts for Engelhard Corporation, which was subsequently acquired by BASF Corporation.

2. I received a doctorate degree in Inorganic Chemistry in 1982 from University of Illinois. I have been involved in the research and development of catalysts since 1986, and since 1995 I have worked in the areas of research and development of catalysts for automotive emissions, in particular diesel engines.

3. Since becoming involved in catalyst research and development in 1986, I have taken steps to stay current in the state of art of catalysts. For example, I review scientific literature submitted for publication and journals in the area of automotive catalysis. I attend scientific meetings related to general catalysis and meetings specific to catalysts for automotive emissions. I also am a named inventor in 12 United States patents and applications relating to automotive

catalysts. Since 1975, I have conducted research on zeolite catalysts, and since 1996, I have had a special interest in zeolite catalysts for hydrocarbon and ammonia selective catalytic reduction of NO_x. Since that time, I have regularly reviewed the literature regarding these catalysts.

4. This is my second declaration in the above-identified reexamination. I previously submitted a declaration dated January 20, 2011 in this matter ("First Declaration").

5. I have read United States Patent No. 7,601,662 ("the '662 patent"). Thus, as stated in my First Declaration, I am familiar with the specification and claims of the '662 patent.

6. I understand that the '662 patent is presently under reexamination in the United States Patent and Trademark Office. I further understand that all of the claims have been rejected as allegedly being obvious over various cited references.

7. The '662 patent generally describes a catalyst comprising an aluminosilicate zeolite having the CHA crystal structure, a silica to alumina ratio of about 15 and an atomic ratio of copper to aluminum exceeding about 0.25, with specific claims directed to silica to alumina ratios in the range of 15 to 40 and copper to aluminum ratios in the range of about 0.25 to 0.50. The catalysts described and claimed in the '662 patent are useful for the abatement of nitrogen oxides in lean burn engines such as diesel engines, particularly by selective catalytic reduction in excess oxygen in the presence of a reductant such as ammonia.

8. I have reviewed a number of articles and other literature related to zeolite catalysts, including an article by Dr. Gabriele Centi entitled Nature of Active Species in Copper-Based Catalysts and Their Chemistry of Transformation of Nitrogen Oxides, in *Applied Catalysis A: General*, Vol. 132, 179-259 (1995) ("the Centi Article") and a book authored by Dr. Centi, Siglinda Perathoner and Fernuccio Trifiró entitled "Sustainable Industrial Processes," published in 2009 ("the 2009 Centi Book"). I have also, once again, reviewed a series of emails related to a project I was pursuing with the Department of Energy with a university chemistry professor.

9. Based on my review of these articles, these emails and my general knowledge of the state of the art in zeolite catalysts from the early 1990s to 2007, it is my opinion that unsolved problems existed from at least 1995 to 2007 in the use of zeolite catalysts in automotive applications. It is my further opinion that from 1995 to 2007 there was skepticism about whether zeolite catalysts could ever be used in automotive applications. This skepticism was caused by concerns about the hydrothermal stability of these catalysts.

The Centi Article

10. One of the articles I reviewed in forming my opinions in this matter was authored by Dr. Gabriele Centi. The article is entitled Nature of Active Species in Copper-Based Catalysts and Their Chemistry of Transformation of Nitrogen Oxides, in Applied Catalysis A: General, Vol. 132, 179-259 (1995) ("the Centi Article"). A copy of the Centi Article is attached as Exhibit A.

11. I understand that Dr. Centi submitted a declaration in this reexamination for the Requester.

12. The Centi Article states that "several unresolved problems limit the outlook for successful use of zeolites in automotive converters." Exhibit A at 183. In my opinion, this reference in the Centi Article proves that unsolved problems existed in the use of zeolite catalysts in automotive converters in 1995.

13. The Centi Article also states that "[a] low hydrothermal stability, in particular, is the more critical weakness of copper-containing zeolites." *Id.* The Centi Article also states that "[c]opper-based catalysts are thus a reference catalyst family for the investigation of the mechanism of selective reduction of NO_x, *albeit for practical applications their low hydrothermal stability may prevent a commercial use.*" *Id.* at page 184 (emphasis added). In my opinion, these references in the Centi Article demonstrate that unsolved problems existed in the use of copper based catalysts and that the perceived problem was a low hydrothermal stability of these catalysts. Further, in my opinion, these references demonstrate skepticism about whether copper-based catalyst could ever be used in automotive applications.

14. Furthermore, the Centi Article recognizes that there was interest in Cu-zeolites at least as early as 1990-92, as Dr. Centi indicates that there was increased interest in Cu-zeolites with reference to research papers published in the 1990-92 timeframe. Exhibit A at page 183. Yet, until the invention of the '662 patent, a Cu-zeolite had not been provided for SCR of NO_x in vehicles that exhibited hydrothermal stability.

The 2009 Centi Book

15. I have also reviewed sections of a book authored by Dr. Centi, Siglinda Perathoner and Ferruccio Trifiró entitled "Sustainable Industrial Processes," published in 2009 ("the 2009 Centi Book"). Excerpts from the 2009 Centi Book are attached as Exhibit B.

16. The 2009 Centi Book (Exhibit B) at page 380, states:

Zeolite systems are very active at low temperatures but they also have disadvantages related to their hydrothermal stability and the

possibility of inhibition or poisoning by different compounds.

These drawbacks drastically limit the industrial applications of these catalysts.

In my opinion, this statement in the 2009 Centi Book confirms that experts in the field of catalysts understood that problems regarding the use of zeolite catalysts, particularly with their perceived hydrothermal stability, remained unsolved in 2009. In my further opinion, this statement also demonstrates that experts in the field of catalysts remained skeptical about the ability to use zeolite catalysts in any industrial application in 2009.

The Department of Energy

17. When I submitted my First Declaration in this matter, I attached a series of emails relating to a proposed Department of Energy ("DOE") project relating to zeolite catalysts. I have reattached this series of emails to this Declaration as Exhibit C.

18. As I stated in my First Declaration, the company I worked for at the time, Engelhard, and a university professor had proposed a project relating to copper zeolite catalysts to the DOE in the 2005 time frame.

19. This proposed DOE project was not approved by the DOE. In my opinion, the proposed DOE project was not approved because of the skepticism regarding the use of zeolite catalysts in the field in 2005. My opinion is based, in part, on the views expressed in the Centi Article and in the Centi Book.

20. My opinion is further based on the advice the DOE gave to the professor I was working with. In September 2005, the professor advised me that:

the major criticism from the reviewer (and echoed by the DOE grant monitor) is that Cu-exchanged zeolites lack the hydrothermal stability needed to be commercially viable for SCR of NO_x with ammonia for diesel engines.

Exhibit C at 6-7.

21. The professor also noted that one DOE reviewed stated: "I have heard the same negative comment about the prospects for Cu-zeolites from several other investigators who presumably are also experts in this area." Exhibit C at page 3. Thus, in addition to the DOE reviewer and his colleagues at the DOE, several other investigators were still dismissing the commercial viability of Cu-zeolites due to their lack of hydrothermal stability in the 2005 time frame.

22. One of the DOE reviewers also stated that "[i]t is interesting to note that not a single metal-zeolite for SCR was commercialized in the USA in the 1990[s]. (see John Armor, App. Cat. A V222, page 407(2001))." Exhibit C at page 4. I am familiar with the Armor article referenced by the reviewer, and the article does a full review of catalysts commercialized in the USA during the 1990s, but does not mention any Cu-zeolites. I am also familiar with Dr. Armor and aware that he has a Ph.D. in chemistry, extensive experience in heterogeneous catalysis research, and was the President of the North American Catalysis Society for eight years. This statement indicates that the DOE was still skeptical about the use of zeolite catalysts in 2005.

23. I also note the criticism offered by the project reviewer and the DOE grant monitor reiterates the standing criticism noted in the Centi Article and the 2009 Centi Book.

24. I have also reviewed a document submitted in this reexamination by the Requester on or about July 27, 2011. Pages 30 and 31 of this document is attached hereto as Exhibit D. In this document, the Requester characterized these emails as follows:

In addition, Requester notes that in his e-mail correspondence attached as Exhibit A of his Declaration to show alleged "skepticism by experts", Dr. Roth describes these "experts" as "a reviewer who does not have much experience with zeolites", "not understanding the fundamental hydrothermal stability of zeolites", and "reviewers, who are not experts in the field, [who] are taking what little they have heard [sic] about unrelated chemistry and drawing the conclusion that your proposed project is not based on a catalyst that can survive under realistic conditions."

Accordingly, Requester respectfully submits that the proffered evidence of alleged "secondary considerations" is not effective to rebut an obviousness rejection of claims 1-55 of the '669 patent.

Exhibit D.

25. In my opinion, the Requester has focused on specific words in the email while ignoring the prevailing view of experts in the field, such as the views expressed in the emails, in the Centi Article and in the 2009 Centi Book that there were problems and skepticism regarding the use of zeolite catalysts due to their hydrothermal stability problems.

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