

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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

**Mail Stop Inter Partes Reexam**  
Central Reexamination Unit  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**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. In 2005, I held the position of research group leader for NOx 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. I am familiar with United States Patent No. 7,601,662 ("the '662 patent"), which is directed to a catalyst comprising a 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. Such catalysts 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. I understand that the '662 patent presently under reexamination in the United States Patent and Trademark Office, and that all of the claims have been rejected as allegedly being obvious over various cited references.

4. In 2005, I contacted a university professor, whom Engelhard was working with to obtain Department of Energy (DOE) funding for a proposal to study Cu-zeolites for selective catalytic reduction (SCR) of nitrogen oxides (NOx). Exhibit A attached hereto is a copy of the e-mail correspondence with the university professor on the DOE proposal, with the names of the professor, DOE review personnel and other personnel redacted. As shown on page 7 of Exhibit B, the grant proposal was wait-listed because the DOE grant monitor concluded that "Cu-exchanged zeolites lack the hydrothermal stability needed to be commercially viable for SCR of NOx with ammonia for diesel engines."

5. After receiving the information that the grant proposal had been wait-listed, I wrote back to the professor asking if the proposal could be reconsidered. The professor explained that "some reviewers, and my DOE grant manager simply think Cu-exchanged zeolites are far to [sic, too] unstable to water to be commercially feasible, so they do not want to fund work in the area." (Exhibit A, at page 5).

6. After writing to the professor a second time, the professor quoted the DOE contact as stating:

"Clarifying the water-stability issue, without revealing proprietary knowledge, would be a good point to address in a new version. 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. Thus, it is imperative to argue/present evidence that dispels such belief. More that the practicality of the concept, such as prospects for large-scale commercialization, the BES reviewers will be seeking for the rationale that will lead to new/corrected mechanistic understanding underlying such stability (or lack thereof)."

(Exhibit A, page 3).

7. The professor further quoted one of the reviewers as stating:

"The structure-property-processing relationships that the PI's describe as the scientific goals are meritorious and worthy of support. The PI's weaken their position considerably, however, by emphasizing the technology of SCR in diesel vehicles when in fact propose to study de-NOx via ammonia over Cu-exchanged mordenite. Moreover, the PI's completely side step the issue of catalysts deactivation in the presence of water; I believe this to be the primary reason why metal-exchanged zeolites have limited application. It 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 A, pages 3-4).

8. The statements made by the DOE contact and reviewers represented the view of many researchers and those skilled in the art that Cu-zeolites could not be used as catalysts for the SCR of NOx because of the inability to maintain NOx conversion upon exposure to hydrothermal conditions—namely temperatures in excess of 650° C and H<sub>2</sub>O of 10%. Even the recent literature, recognizing the results achieved in the '662 patent have called the problem of NOx reduction in lean burn engines as "daunting". (Exhibit B, first page).

9. The challenge before the invention of the '662 patent was to provide a zeolite material promoted with a metal or metal ion that exhibited high NOx conversion across a wide temperature range, including temperatures in the range of 200° to 350° C and that maintained high NOx conversion after hydrothermal aging of temperatures above 650° C, and in some cases

as high as 800° C or 900° C. The researchers at Ford summarized the problem in the research paper attached as Exhibit C (first page):

Passenger and light duty diesel vehicles will require up to 90% NO<sub>x</sub> conversion over the Federal Test Procedure (FTP) to meet future Tier 2 Bin 5 standards. This accomplishment is especially challenging for low exhaust temperature applications that mostly operate in the 200° - 350° C temperature regime. Selective catalytic reduction (SCR) catalysts formulated with Cu/zeolites have shown the potential to deliver this level of performance fresh, but their performance can easily deteriorate over time as a result of high temperature thermal deactivation.

10. In 2006, I am aware that Engelhard Corporation provided samples of a Cu-zeolite having the CHA crystal structure deposited on a substrate to Ford Motor Company for SCR testing, and I understand that this is the material tested and described in Exhibit C, a research paper by Ford Motor Company entitled "Enhanced Durability of a Cu/Zeolite Based SCR Catalyst. As noted in Exhibit C (first page), it was believed that Fe-zeolites exhibited superior hydrothermal durability compared to Cu-zeolites. After testing the samples that were sent to Ford, the Ford researchers concluded that the material of the '662 patent is "remarkable". (Exhibit C, last page).

In past years, no reported Cu/zeolite SCR formulation was able to yield stable low temperature NO<sub>x</sub> performance after exposure to hydrothermal conditions consisting of 1 hour at 950° C. Within the last year, a remarkable Cu/zeolite SCR formulation was identified with high NO<sub>x</sub> conversion in the 200° C – 350° C temperature range.

11. The material claimed in the '662 patent has been sold for use as a SCR catalyst for the removal of NO<sub>x</sub> in diesel engines in several different automotive manufacturer vehicle platforms. The material of the '662 patent has met a long-standing and previously unfulfilled need - a metal zeolite that exhibits both excellent NO<sub>x</sub> conversion over a wide temperature range, including the range of 200° to 350° C, and that maintains high conversion after exposure to hydrothermal conditions. This has allowed automotive manufacturers to meet increasingly stringent NO<sub>x</sub> standards that went into effect in 2010.

12. The Research & Development Council of New Jersey has recognized the inventors of the '662 patent with the 2010 Thomas Alva Edison Patent Award in the environmental category, which recognizes the outstanding work done by New Jersey scientists and inventors by honoring the most exceptional efforts.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made herein on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above-identified patent.

Respectfully submitted,

Dated: January 20, 2011

By:   
Stanley Roth, Ph.D.

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