

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

VOLKSWAGEN GROUP OF AMERICA, INC.

Petitioner

U.S. Patent No. 6,037,307

Issue Date: March 14, 2000

Title: CATALYST/SORBER FOR TREATING
SULFUR COMPOUND CONTAINING EFFLUENT

DECLARATION OF ROBERT J. FARRAUTO, PH. D.

Case No. IPR2014-01554

**Volkswagen Ex. 1007A
Volkswagen v. EmeraChem
IPR2014-01554**

I, Robert J. Farrauto, Ph.D., hereby declare and state as follows:

I. BACKGROUND AND QUALIFICATIONS

1. I hold the title of Professor of Practice, Department of Earth and Environmental Engineering, Fu Foundation School of Engineering and Applied Science, Columbia University in the City of New York. Previously, I was Adjunct Professor at Columbia University (2005 - August 2012). I received a B.S. in Chemistry in 1964 from Manhattan College, and a Ph. D. in Chemistry in 1968 from Rensselaer Polytechnic Institute.

2. I have over forty years of professional experience in the field of catalysis and automotive emissions control. Further, I have authored or co-authored over one hundred published technical papers and two books in this field. In addition, I am a named inventor on over fifty U.S. patents relating to catalysts and catalytic processes.

3. I was a Research Scientist at Corning Glass Works from 1968 to 1974, where I worked on catalyst development for base metal emission control catalysts. From 1974 to 1976, I was a Research Scientist at Gulf Oil, focusing on catalyst development in upgrading petroleum and coal liquids to useful fuels and chemicals.

4. From 1976 until my retirement from industry in August 2012, I was employed by BASF Catalysts and its predecessor, Engelhard Corporation, which was acquired by BASF in 2006. From 1976 to 1990, I was Manager of Chemical and Environmental Catalysts at Engelhard, and managed a team of scientists and engineers in the development of advanced three-way catalysts for abating emissions from passenger car vehicles. From 1990 to 1995, I was Principal Scientist and Manager of Diesel Technology, and led a team of scientists and engineers in the development of the first monolith-diesel oxidation catalyst for heavy duty trucks, buses and light-duty vehicles, and developed new nitric oxide catalysts and hydrocarbon traps for automobile catalytic converters. From 1995 to 2008, I was Research Fellow and Manager of Hydrogen and Fuel Cell Technology and led a team developing catalysts for hydrocarbon fuel processing for the generation of hydrogen and fuel cells. From 2008 to 2012, I held the position of Research Vice President at BASF Catalysts.

5. At Columbia, I currently teach two graduate courses ("Catalysis for Emission Control" and "Industrial Catalysis") and two undergraduate courses ("Pollution Reduction and Environmental Engineering" and "Senior Design"). I currently supervise one post-doctoral student, three Ph.D. students and three master's degree students.

6. My *curriculum vitae*, which is attached hereto, discusses my qualifications, experience, awards, and publications in the field of catalysis and automotive emissions control and my work at Engelhard/BASF in more detail.

II. ASSIGNMENT AND MATERIALS REVIEWED

7. I have been retained by Volkswagen Group of America, Inc. in connection with its Petition for *inter partes* review of U.S. Patent No. 6,037,307 ("the '307 patent"). I have reviewed the '307 patent, its prosecution history, and the prior art references cited in the Petition, Joy et al. U.S. Patent No. 4,760,044 ("Joy", Ex. 1004); Tauster et al. U.S. Patent No. 4,939,113 ("Tauster" , Ex. 1005); and Rieck, J.S., et al., "Development of Non-Nickel Additives for Reducing Hydrogen Sulfide Emissions from Three-Way Catalysts", SAE Paper 892095 (1989) ("Rieck" , Ex. 1006).

8. I submit this declaration in support of the Petition for *Inter Partes* review of U.S. Patent No. 6,037,307 ("the '307 patent").

9. I am not an employee of Volkswagen Group of America, Inc. or any parent or subsidiary thereof.

10. I am being compensated for my time at a rate of \$200 per hour. My compensation is in no way dependent upon the substance of the opinions I offer below, or upon the outcome of Volkswagen's Petition for *Inter Partes* review (or the outcome of such an *inter partes* review, if a trial is initiated).

III. PERSON OF ORDINARY SKILL IN THE ART

11. I understand that a patent must be written such that it can be understood by a “person of ordinary skill” in the field of the patent.

12. I understand that this hypothetical person of ordinary skill in the art is considered to have the normal skills and knowledge of a person in a certain technical field, as of the time of the invention at issue. I understand that factors that may be considered in determining the level of ordinary skill in the art include: (1) the education level of the inventor; (2) the types of problems encountered in the art; (3) the prior art solutions to those problems; (4) rapidity with which innovations are made; (5) the sophistication of the technology; and (6) the education level of active workers in the field. I also understand that “the person of ordinary skill” is a hypothetical person who is presumed to be aware of the universe of available prior art.

13. In my opinion, as of the July 10, 1998 filing date of the ‘307 patent, a person of ordinary skill in the art of catalysis would have about 5 years of work experience regarding the chemical aspects of catalysis, and at least a Masters of Science degree in chemical engineering, mechanical engineering, or chemistry.

14. Based on my experience and education, I consider myself (both now and as of July 10, 1998) to be a person of at least ordinary skill in the art with respect to the field of technology implicated by the ‘307 patent.

IV. CLAIM CONSTRUCTION

15. In rendering the opinions set forth in this declaration, I have considered what one of ordinary skill in the art would consider to be the broadest reasonable construction of the '307 patent terms.

16. Claims 1 and 11 of the '307 patent recite a "regenerable catalyst/sorber for removing gaseous sulfur compounds from gaseous streams". As of the July 10, 1998 filing date of the '307 patent, a person of ordinary skill in the art would understand that the term "regenerable" denotes that the catalyst/sorber is capable of releasing (i.e., "desorbing") a gaseous sulfur compound that had been previously absorbed by the catalyst/sorber.

V. TEACHINGS OF THE PRIOR ART REFERENCES

17. In my opinion, one of ordinary skill in the art as of the July 10, 1998 filing date of the '307 patent would have considered Joy, Tauster and Rieck to be within the same technical field as the subject matter set forth in the '307 patent. Further, all of these references would be considered highly relevant prior art to the claims of the '307 patent.

A. The Teachings Of Joy

18. I have read Joy and fully understand its subject matter. Set forth below, I present my opinions on what a person of ordinary skill in the art would understand certain aspects of Joy to be teaching.

19. The Joy patent discloses the process of impregnating the secondary support with the oxide form of the selected metal, as follows:

Thus, if ceria is the desired secondary refractory oxide support and nickel is the desired second active component then the ceria may be impregnated with an aqueous solution of nickel nitrate. After the ceria is impregnated with the nickel salt, the mixture is dried, and then calcined in the air at a temperature of about 400° to about 700° C. for about one to about 3 hours. This results in a uniform distribution of *nickel oxide* on the cerium oxide.

Ex. 1004 at col. 6, ln. 55-63 (emphasis added).

20. If the secondary refractory oxide support would be impregnated with the aqueous solution of copper nitrate, copper chloride or copper acetate (as expressly disclosed in Joy at col. 6, ln. 49-54), and then calcined in the same manner as described in paragraph 19 above, the result would be a uniform distribution of copper oxide on the secondary support.

21. If the secondary refractory oxide support would be impregnated with the aqueous solution of lead nitrate, lead chloride or lead acetate (as expressly disclosed in Joy at col. 6, ln. 49-54) and then calcined in the same manner as described in paragraph 19 above, the result would be a uniform distribution of lead oxide on the secondary support.

22. Calcination is a process of heating a compound at high temperature in the presence of air or oxygen to bring about a thermal decomposition of that compound.

23. Joy expressly discloses that “the second active component, such as nickel or cobalt, is preferred to be present in an amount in the range of about 0.7 to about 15 g per liter of monolithic carrier volume.” Ex. 1004 at col. 7, ln. 21-24. Issued claim 15 of Joy states “the metal is copper present in a concentration of about 0.7 to about 15 g per liter of carrier volume.” Given that a liter is 61.024 in³, this stated range converts to 0.011 to about 0.246 grams per in³.

B. The Teachings of Tauster

24. I have read Tauster and fully understand its subject matter. Set forth below, I present my opinions on what a person of ordinary skill in the art would understand certain aspects of Tauster to be teaching.

25. Tauster prepared a series of exemplary three-way catalyst (“TWC”) cores referred to as TWC-II. See Ex. 1005 at col. 5, ln. 40 - col. 6, ln. 7.

26. In particular, the initial step in preparing TWC-II is described by Tauster as follows:

TWC CATALYST PREPARATION II

Unstabilized alumina is impregnated with monoethanolamine platinum hexahydroxide and ceria by combining 600 g of gamma

alumina having a surface area of about $150 \text{ m}^2/\text{g}$, and 500 g of deionized water, milling for about ten minutes, adding 11.75 g of platinum in monoethanolamine platinum hexahydroxide in 125 ml of deionized water, milling for an additional 20 minutes, adding 230 g of ceria having a surface area of about $180 \text{ m}^2/\text{g}$, diluting 74 g of acetic acid with 175 g of deionized water which is added to the mill, then milling until 90% by weight of the particles are reduced to less than about 9 microns. The resulting slurry of about 51% solids is applied to a monolith to result in a final solids weight gain of about $1.8/\text{in}^3$ after drying at 120°C . for 2 hours and calcination at 400°C . for about 20 minutes.

Ex. 1005 at col. 5, ln. 40-55. A person of ordinary skill would understand that this disclosed preparation step would result in platinum and ceria being deposited on a monolith.

27. Persons of ordinary skill in the art use the term "ceria" to refer to cerium oxide.

C. Teachings of Rieck

28. I have read Rieck and fully understand its subject matter. Set forth below, I present my opinions on what a person of ordinary skill in the art would understand certain aspects of Rieck to be teaching.

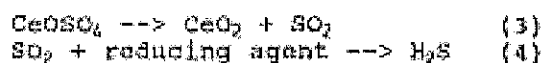
29. Rieck discloses a three-way catalyst that reduces hydrogen sulfide emissions from automotive exhaust, by including a copper ferrite (i.e., CuFe_2O_4) additive to the three-way catalyst. Ex. 1006 at p. 2.

30. A person of ordinary skill would understand CuFe_2O_4 to be a binary oxide of Cu and Fe.

31. Rieck expressly discloses that “[W]hile the engine runs under lean conditions (i.e., more oxygen than fuel), sulfur can be stored on the catalyst surface by the following mechanism.”



Ex. 1006 at p. 1. “Following a change in exhaust composition to a rich stoichiometry (i.e., more fuel than oxygen), the stored sulfur can be reduced to H_2S by the following reactions.”



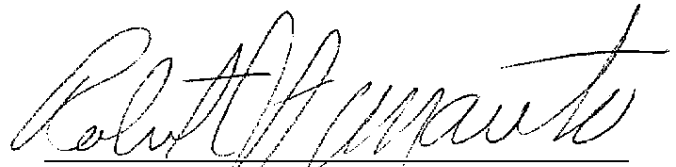
Ex. 1006 at p. 1.

32. A person of ordinary skill in the art would understand that the removal of the stored sulfur from the three-way catalyst during “rich” operations (i.e., less oxygen than is required for stoichiometric combustion of fuel) regenerates the three-way catalyst.

33. Table 1 of Rieck shows that in sample C6, which has a cordierite substrate, the loading of the CuFe_2O_4 additive is 14.4 g/liter. Ex. 1006 at p. 2. Given that a liter is 61.024 in^3 , this stated loading converts to 0.236 grams per in^3 .

I declare that all statements made herein of my own knowledge are true and that all statements made 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 §1001 of Title 18 of the United States Code.

Dated: New York, New York
September 26, 2014


Robert J. Farrauto, Ph.D.



Curriculum Vita
Robert J. Farrauto, Ph.D.
Professor of Practice
Columbia University

Personal

Birth Date: November 22, 1941 / New York City
Gender: Male
Citizenship: United States of America
Status: Married with two children/six grandchildren, two step-daughters

Education

Ph.D. Chemistry, 1968, Rensselaer Polytechnic Institute, Troy, New York
Advisor: David Aikens
B.S. Chemistry, 1964, Manhattan College, Bronx, New York

Employment Experience

September 2012- present: Professor of Practice, Columbia University, Earth and Environmental Engineering Department, Columbia University, in the City of New York

Currently I am supervising one post doc, three PhD and three master's degree students. I teach two graduate courses (1,2) and two undergraduate courses (3,4)

1) "Catalysis for Emission Control" 2) Industrial Catalysis, 3) "Pollution Reduction and Environmental Engineering" 4) "Senior Design"

2005-August 2012: Adjunct Professor, Earth and Environmental Engineering Department, Columbia University, in the City of New York

2008-retired August 2012:Research Vice President, BASF Catalysts (formerly Engelhard), 25 Middlesex-Essex Turnpike, Iselin, New Jersey 08830

1995-2008: Research Fellow and Manager of Hydrogen and Fuel Cell Technology

Currently leads a team of scientists and engineers developing catalysts for hydrocarbon fuel processing for the generation of hydrogen and fuel cells. Our team has commercialized 25 new products for world wide markets.

1990-1995: Principle Scientist of Manager of Diesel Technology

Led a team of 20 scientists and engineers in the development of the first monolith-diesel oxidation catalyst for heavy duty trucks, buses and light duty vehicles for the US, Asian and European market. Total revenues have exceeds \$200 million. Developed new nitric oxide catalysts and hydrocarbon traps for automobile catalytic converters

1976-1990: Manager of Chemical and Environmental Catalysts

Managed a team of scientists and engineers in the development of advanced three way catalysts for abating emissions from passenger car vehicles serving the US, Asian and European market. Farrauto led a team in the development of improved chemical catalysts for the production of nitric acid. Supervised a research team developing new catalysts for the hydrogenation of a wide variety of organic functional groups. Was responsible for establishing a materials characterization group serving the entire worldwide company needs for catalyst and materials product.

1974-1976 Research Scientist, Gulf Oil, Harmarville, PA

Catalyst development in upgrading petroleum and coal liquids to useful fuels and chemicals.

1968-1974 Research Scientist at Corning Glass Works, Corning New York

Catalyst development for base metal emission control catalysts

Recent Publications

1. Farrauto, R.J. "New Catalysts and Reactor Designs for the Hydrogen Economy" Chemical Engineering Journal 238 (2014) 172-177
2. Duyar, M., Farrauto, R. J., Castaldi, M. and Yegalulp, T. "Enhanced Hydrogen Generation in the Water Gas Shift Reaction" Industrial and Engineering Chemical Research, 53, (#3) 1064-1072, 2014
3. Janke, C., Duyar, M, Hoskins, M. and Farrauto, R.J. "Catalytic and adsorption studies for the Hydrogenation of CO₂ to Methane , Applied Catalysis B: Environmental" 26 June (2014) 184-191
4. Farrauto, r. J. Low temperature oxidation of methane" Science 337 (2012) 659-660

5. Simson, A. Castaldi, M. and Farrauto, R. "Kinetic and process study for ethanol reforming using a Rh/Pt washcoated monolith catalyst" *Applied Catalyst B: Environmental*, "Kinetic and process study for ethanol reforming using a Rh/Pt washcoated monolith catalyst." *Applied Catalysis B: Environmental*, 89, (1-2), 2009, 58-64
6. Simson, A. Farrauto, R. and Castaldi, M. "Catalyst deactivation and regeneration reforming ethanol/gasoline blends containing sulfur" *Applied Catalysis B Environmental* 106 (2011) 295-303
7. Gruene, P, Belsova, A., Yegalup, T. Farrauto, R. And Castaldi, M "Dispersed calcium oxide as a reversible and efficient CO₂-sorbent for intermediate temperatures. IE and C Research 50 (2011) 4042-4049
8. Zhang, Q and Farrauto, R. "Methanol steam reforming catalyst for portable power applications" *Applied Catalysis A. General* (2011) 395, 64-70
9. R. Farrauto, A. Schaefer, E. Schwab, H. Urtel "Hydrocarbon Reforming Catalysts and New Reactor Designs for Compact Hydrogen Generators" *Oil and Gas Europe* 2011.
10. Kohn, M., Castaldi, M. and Farrauto, R "ATR and dry reforming of landfill gas over a Rh/Al₂O₃ monolith catalyst" *APCAT B: Environmental* 94 (2010) 125

Total publications ~ 115, US patents 56

Awards

- 2008 Ciapetta Lectureship Award sponsored by the North /American Catalysis Society
- 2005 Catalysis and Reaction Engineering Practice Award from the American Institute of Chemical Engineers
- 2001 International Precious Metal Institute (IPMI) Award for Outstanding Contributions in the field of precious metal catalysis
- 1998 Cross Canada Lectureship Award sponsored by the Canadian Catalysis Society
- 1995 Teaching Excellence Award from the New Jersey Institute of Technology

Society Memberships

- Program Chairman for the 19th North American Catalysis Society Meeting, Philadelphia, PA 2005
- American Institute of Chemical Engineers (AIChE)
- American Chemical Society (ACS)
- International Precious Metal Institute

Plenary Lectures and from 2006-Present

- XX Reaction Engineering December 2012 "New Catalysts and reactor designs for the hydrogen economy, Luxembourg
- Mitsui Keynote lectureship Award, March 2009:"Automobile Catalysis: Past, Present and Future" . Tokyo, Japan
- Idecat, Porguellos, France "The Road to the Hydrogen Economy," May 2007

- Natural Gas Conversion Conference, Natal, “The Convergence of Fundamental and Applied Catalysis in Solving Industrial Problems.” May 2007
- Mittasch Conference, BASF Ludwigshafen, Germany, “Advances in Environmental and Petroleum Catalysis by Engelhard” November 2006
- 13th Nordic Conference on Catalysis, Goteborg, Sweden “New Catalysts for the Hydrogen Economy” October 2008
- 50th Anniversary of the Japanese Catalysis Society “From the Internal Combustion Engine to the engine of the future: The Fuel Cell” November 2008

Books authored

Heck and Farrauto “Catalytic Air Pollution Control: Commercial Technology” Third edition, Wiley and Sons, Hoboken, NJ 2009

Bartholomew and Farrauto “Fundamentals of Industrial catalytic Processes” Second edition, Wiley and Sons, Hoboken, NJ 2006

Journal Publications (1973-Present)

1. R.J. Farrauto and W.L. Haynes, Cements from glass powders, Amer. Cer. Soc. (Bull) **52**(3), 276 (1973).
2. W. Morgan and R.J. Farrauto, Active sites on a copper chromite catalyst, J. Catal. **31**, 140 (1973).
3. W. Hertl and R.J. Farrauto, Mechanism of carbon monoxide and hydrocarbon oxidation on a copper chromite catalyst, J. Catal. **29**, 352 (1973).
4. R.J. Farrauto and B. Wedding, Poisoning by SO_x of some base metal oxide auto exhaust catalysts, J. Catal. **33**, 249 (1973).
5. B. Wedding and R.J. Farrauto, Rapid evaluation of automotive exhaust oxidation catalysts with a differential scanning calorimeter, Ind. Eng. Chem. **13**, 45 (1974).
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9. R.J. Farrauto, Adsorption studies on a nickel alumina methanation catalyst, J. Catal. **41**, 482 (1976).

10. C. Bartholomew and R.J. Farrauto, Chemistry of nickel-alumina catalysts, *J. Catal.* **45**, 41 (1976).
11. J. Patzer, R.J. Farrauto, and A. Montagna, Characterization of coal liquefaction catalysts using 1-methyl naphthalene as a model compound, *Ind. Eng. Chem. Proc. Des. Dev.* **18**, 625 (1979).
12. R. St.Amand, J. Williams and R.J. Farrauto, Application of metallorganic deposition for solar energy collectors, American Optical Society (AOS) Proc., 2nd Ann. Conf. Sept. (1979).
13. R.D. Lanam and R.J. Farrauto, Solar absorber coatings, Solar Energy Research (SERI) Proc., 2nd Ann. Conf. 1981.
14. R.J. Farrauto and M.H. Hobson, Catalyst characterization, *Encyclopedia of Physical Science and Technology*, Academic Press, **2**, 563 (1987).
15. R.J. Farrauto, M.H. Hobson and N. Brungard, Relationship between catalyst characterization and performance, *Catalysis of Organic Reactions*, Marcel Dekker, **33**, 177 (1988).
16. R.J. Farrauto, R. McClung and A.B. Stotler, Precious metals in the petrochemical industry, *Proc. 12th Intern. Prec. Metals Inst.*, 1988, pp.13.
17. J.C. Bonacci, R.J. Farrauto, and R.M. Heck, Catalytic incineration of hazardous wastes, *Library for Environ. Sci.*, Gulf Publishing **1**(4), 130 (1988).
18. C.H. Lee and R.J. Farrauto, Catalyst deactivation due to transient behavior in nitric acid production, *Ind. Eng. Chem. Res.* **28**, 1 (1989).
19. R.J. Farrauto and H.C. Lee, Ammonia oxidation catalyst with enhanced activity, *Ind. Eng. Chem. Res.* **29**(7), 1125 (1990).
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22. J. Chen, R.M. Heck and R.J. Farrauto, Deactivation, regeneration and poison resistant commercial catalysts, *Catal. Today* **11**, 517-545 (1992).
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