# Patent Owner BASF Corporation's Oral Argument

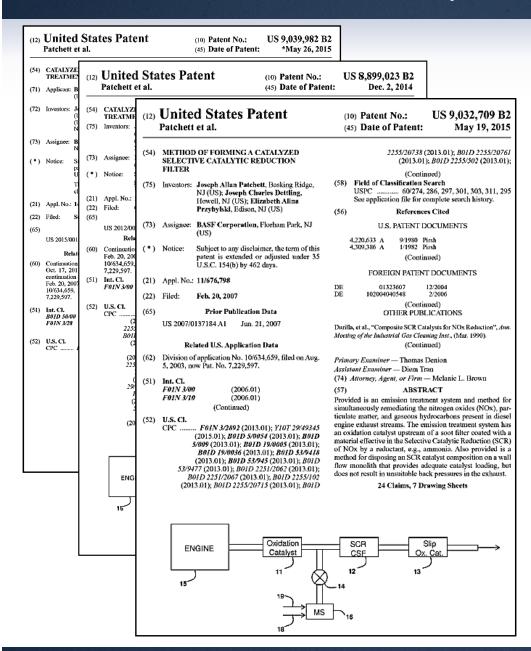
Johnson Matthey Inc. and Johnson Matthey PLC v. BASF Corporation

Case IPR2015-01265 U.S. Patent No. 8,899,023 Case IPR2015-01266 U.S. Patent No. 9,039,982

Case IPR2015-01267 U.S. Patent No. 9,032,709

August 23, 2016

#### BASF Patents: '709, '023, And '982 Patents



#### '709 Patent

- Filed: February 20, 2007
- Issued: May 19, 2015
- Priority Date: August 5, 2003

#### <u>'023 Patent</u>

- Filed: October 17, 2011
- Issued: December 2, 2014
- Priority Date: August 5, 2003

#### '982 Patent

- Filed: September 26, 2014
- Issued: May 26, 2015
- Priority Date: August 5, 2003

#### **Patent Claims**

#### '709 Patent (method)

#### Indep. Claim 1

- -Oxidation catalyst
- -Metered ammonia
- -SCR catalyzed wall flow monolith
  - -50%-60% porosity
  - -Pore size 10-25μm
  - -Zeolite & base metal catalyst
  - -Washcoat loaded up to 2.4 g/in<sup>3</sup>
  - -Integrated NOx and PM removal

#### Indep. Claim 20

-Washcoat loading of at least 1.3 g/in<sup>3</sup>

#### '023 Patent (system)

#### Indep. Claim 1

- -Oxidation catalyst
- -Injector meters ammonia
- -SCR catalyzed wall flow monolith
  - -50%-60% porosity
  - -Pore size 10-25μm
  - -Zeolite & base metal catalyst
  - -Washcoat loaded up to 2.4 g/in<sup>3</sup>
  - -Integrated NOx and PM removal

#### Indep. Claim 16

- -Washcoat loading of at least 1.3 g/in<sup>3</sup>
- Indep. Claim 22
  - -50%-55% porosity

#### '982 Patent (catalyst article)

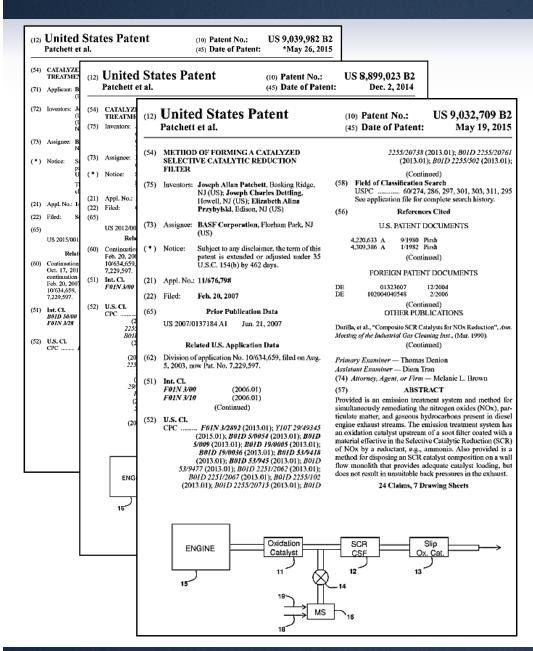
#### Indep. Claim 1

- -SCR catalyzed wall flow monolith
  - -50%-60% porosity
  - -Pore size 10-25μm
  - -Zeolite & base metal catalyst
  - -Washcoat loaded up to 2.4 g/in<sup>3</sup>
  - -Integrated NOx and PM removal

#### Indep. Claim 16

- -Washcoat loaded up to 1.3 g/in<sup>3</sup>
- Indep. Claim 22
  - -50%-55% porosity

#### **BASF Patents: Solution To Challenge Facing The Industry**



#### **Solution: Unique Catalyzed Filter**

- > Highly porous wall flow filter
  - > Porosity: 50% 60%
  - Avg. Pore Size: 10 25 microns
- SCR Catalyst composition is part of a slurry-loaded washcoat that permeates filter walls SCR
- SCR Catalyst comprised of a zeolite and base metal of either copper or iron
- Catalyst loading up to 2.4g/in³
- Simultaneous effective chemical reduction of NOx and oxidation of soot

#### Dr. Tennent: Industry Required High SCR Washcoat On Filter

By 2002, the auto and truck manufacturers believed that meeting the 26. Euro IV and Euro V emission standards, while still maintaining the compactness of a system designed for mobile applications, required putting a very high SCR washcoat loading into the walls of the filter. That is, to achieve the increase in NOx abatement, catalyst would have to be incorporated into the system without increasing the overall size of the exhaust gas treatment system. Auto and truck manufacturers responded to the proposed Euro IV and Euro V emission standards by requesting that Diesel Particulate Filter ("DPF") suppliers develop filters with a higher porosity that could accommodate a very high catalyst washcoat. Based on the requests that Corning received, the auto and truck manufacturers were, as of at least 2002, trying to develop a filter that could accommodate a catalyst washcoat loading of between 100 g/L to 125 g/L. Accordingly, as of 2002, there was a move towards developing higher porosity filters, so that they could be combined with a high catalyst loading.

Ex. 1003 (Declaration of David L. Tennent, Ph.D. ) at ¶26 (cited in Paper 20, Patent Owner's Response, p.38).

#### Dr. Tennent: Industry Required High SCR Washcoat On Filter

- Q. So your testimony is that the industry as a whole believed that you needed a high amount of SCR washcoat to be loaded onto the walls of a high-porosity filter and you believe the industry believed that was required as of the end of 2002; right?
- A. Absolutely.

#### Dr. Tennent: No Art With High SCR Washcoat On Filter

- Q. So you don't actually cite any art that discloses a high SCR washcoat onto a high porosity filter; correct?
- A. Not in that, no, I do not cite that combination.

#### Dr. Harold: No Art With SCR Washcoat On Filter

- Q. What's your best estimate of when you first became aware of a published report of a DPF washcoated with an SCR catalyst, putting aside Hüthwohl?
- A. I believe it would probably be the BASF patent.

When choosing an SCR catalyst, therefore, it would have been necessary to select

a catalyst capable of withstanding the high temperatures needed to periodically

clear the filter of soot that would otherwise begin to clog it, causing high

backpressure. See, infra, § IV.A (discussing the structure of wall flow filters and

the known means to clear a filter of trapped soot).

#### Claim 1

#### Hüthwohl + Hashimoto + Speronello, in view of Teraoka

wherein the wall flow monolith comprises a washcoat of SCR catalyst composition that permeates the walls, the SCR catalyst composition comprising a zeolite and base metal component selected from one or more of a copper and iron component,

Speronello. Teaches an "iron and/or copper-promoted zeolite catalyst," col. 3, line 60, prepared by "dipping" the substrate into a slurry of an iron or copper zeolite, col. 7, lines 23-25. Provides at least three motivations for selecting the disclosed catalysts:

- Temperature Stability "[B]y utilizing suitable zeolite catalysts in accordance with the teachings of the present invention, high temperature gaseous streams, for example, gaseous streams at temperatures up to about 600° C., may be treated without seriously adversely affecting the life or efficiency of the catalyst." Col. 6, lines 16-23. "Suitable promoted zeolite materials demonstrate sufficient thermal and hydrothermal stability to ... provide an acceptably long life and efficiency of the catalysts." Col. 6, lines 22-25.
- Sulfur Tolerance. "The tolerance of the catalyst material for such sulfurous contaminants is increased, i.e., the catalyst is rendered more resistant to sulfur poisoning, by selecting" one of the disclosed zeolites. Col. 6, lines 38-41. "The most preferred types of zeolite for resistance to sulfur poisoning are those which have a pore system in which the 7 to 8 Angstrom diameter pores are interconnected in all three crystallographic dimensions." Col. 6, lines 46-49.
- NOx Conversion Efficiency. The disclosed Examples "demonstrate the [NOx reduction] efficacy ... of the present invention." Col. 8, lines 12-13. "Tt will be appreciated that the catalysts of the present invention provide a simple and relatively inexpensive means for efficiently catalyzing the reduction of nitrogen oxides." Col. 16, lines 44-49.

Paper 1, Petition for Inter Partes Review, p. 45.

By 2002, industry had recognized that meeting the newly proposed emissions standards required applying some relatively high catalyst washcoat loading, such as those taught by Speronello, into a wall flow filter. See Tennent Decl., Ex. JM 1003, ¶¶ 18-27, see also id. at ¶ 26 ("[T]he auto and truck manufacturers were, as of at least 2002, trying to develop a filter that could accommodate a catalyst washcoat loading of between 100 g/L to 125 g/L."). This, the industry recognized, presented challenges. Id. at ¶¶ 24-30. Thus, "[i]n 2001 and 2002, the filter manufacturer industry began an intensive effort to develop a filter capable of being loaded with a catalyst washcoat while still achieving acceptable backpressure." Id. at ¶ 30.

Significantly, Hüthwohl demonstrated that its system worked successfully. See id. at 8-9 (interpreting results). Specifically, in one configuration taught by Hüthwohl (the "ECE" configuration), the system reduced particulate matter emissions by 97.58% and reduced NOx emissions by 47.72%. Id. In another configuration (the "ESC" configuration), particulate matter emissions were reduced by 94.17% and NOx emissions were reduced by 55.79%. Id. Therefore, not only would a person or ordinary skill have been motivated to load an SCR catalyst into a filter, but also would have possessed a reasonable expectation that it would successfully work to simultaneously reduce NOx and particulate matter emissions. The Hüthwohl system achieved exactly that.

# Petitioner's Reply: Ignores Real-World Operating Needs

Time and again, BASF argues that the claims are nonobvious because a person of ordinary skill would not have had a reasonable expectation of success in practicing certain unclaimed characteristics. The claims do not require any particular level of NOx or PM reduction. Cf. BASF Response, § IV.A.2. The claims do not require that the soot combustion temperature be lowered by any particular amount. Cf. id. at § IV.A.1. The claims do not require that the SCR catalyst perform up to 1000°C or even 700°C. Cf. id. at § IV.A.3. The claims do not require that the SCR catalyst be resistant to poisoning by ash and unburned hydrocarbons. Cf. id. at § IV.A.4. The claims do not require a particular mode of filter regeneration or performance of the filter during filter regeneration. Cf. id. at § IV.B. The claims do not require that the wall-flow filter exhibit a particular thermo-mechanical durability. Cf. id. at § IV.E. Yet, to make its arguments, BASF assumes that the claims require each of these limitations.

# The State of The Art

#### No Pre-Existing Answers For New EU Emissions Standards

As Hüthwohl describes, the automotive industry had no pre-existing answers for complying with newly proposed European Union emissions standards. In 1998, the EU Environment Council proposed standards to "reduc[e] the emissions limits for nitrogen oxides (NOx) and particles in 2 stages"—the first stage to go into effect in 2005 and the second in 2008. *Id.* at 1. Meeting these new standards presented "a huge challenge for the vehicle and supplier industry." Id. at 2. Starting in 1998, the industry, therefore, had to come up with "new approaches" for how to limit NOx emissions while simultaneously limiting particulate matter emissions, id. at 3, and it had to do so in time to comply with the new standards going into effect in 2005 and 2008. Id. at 1.

Paper 1, Petition for Inter Partes Review of U.S. Patent No. 9,032,709, pp. 23-24.

#### The NOx-Particulate Tradeoff

The control of particulate emissions and  $NO_x$  represent significant challenges to the diesel engine manufacturer because they are coupled inversely. When the engine operates cooler, it produces less  $NO_x$  but more particulate. At higher temperatures combustion is more complete, generating less particulate but more  $NO_x$ . This is referred to as the  $NO_x$ -particulate tradeoff; when one is high the other is low.

Ex. 1011 (Chapters 8 & 9 from Ronald M. Heck and Robert J. Farrauto with Suresh T. Gulati, Catalytic Air Pollution Control (2002)) at 191 (cited in Paper 20, Patent Owner's Response, p. 7).

#### Petitioner In 2000: Divide and Conquer

So, for example, the engine can be modified to give low NOx and high PM, and this PM can be controlled using filter technology, such as a Continuously Regenerating Diesel Particulate Filter (CR-DPF) [1]. Alternatively, the engine can be calibrated to give low PM and high NOx, and this NOx can be controlled using Selective Catalytic Reduction (SCR) technology,

Ex. 2034 (Guy R. Chandler, et al., An Integrated SCR and Continuously Regenerating Trap System to Meet Future Nox and PM Legislation, Diesel Exhaust Aftertreatment, 2000), at .003 (cited in Paper 20, Patent Owner's Response, p. 40).

#### Petitioner In 2003: Still Divide and Conquer

In principle there are two ways to meet the Stage 4 limits. EGR may be applied to reduce the NOx level, but this will lead to an increase in PM emissions, so it is expected that a filter will need to be added to control PM emissions when using this strategy. The alternative approach is to advance the injection timing, which leads to low PM emissions (within the Stage 4 limits) but high NOx emissions (outside the Stage 4 limits). Selective Catalytic Reduction (SCR) will then be applied to control the NOx (see below).

Ex. 2027 (Philip G. Blakeman, et al. Developments in Diesel Emission Aftertreatment Technology, Society of Automotive Engineers, 2003) at .007 (cited in Paper 20, Patent Owner's Response, p. 8).

# Petitioner's Expert: NO<sub>2</sub> Is Key For Soot Oxidation

- A. So definitely in the '90s the community would have known that NO<sub>2</sub> was an <u>integral</u> part of an oxidant to help in soot reduction.
- A. As we talked about earlier, NO<sub>2</sub> is a very good oxidant of soot and is <u>key</u> to bringing the light-off temperature of soot down and enabling passive regeneration to be more easily accomplished.
- A. So that the combination of a catalyst, the presence of NO<sub>2</sub>, which reduces the oxidation temperature, those are both key to the continuous regeneration to occur.

# Petitioner's Expert: NO<sub>2</sub> Is Integral For Soot Oxidation

- A. The addition of  $NO_2$  to a large surplus of  $O_2$  can serve to reduce the temperature needed to combust the soot. So that's well-known.
- Q. Okay. And that was well-known as of August 2003?
- A. I believe so, yes.
- Q. And that was an important consideration in passive filter regeneration as of August 2003?
- A. I believe so, yes.

# 2005: Ensuring Adequate NO<sub>2</sub> Supply -- A "Major Task"

Hence, a major task of catalyst development is to help ensure an adequate supply of NO<sub>2</sub> necessary for regeneration of the soot-loaded filter. This goal is even more challenging because of the expected lowering in NO<sub>X</sub> raw emissions by future engine concepts [4].

Ex. 2029 (K.V.R. Babu, et al., The Effect of No<sub>x</sub>/Soot Ratio on the Regeneration Behaviour of Catalysed Diesel Particulate Filters for Heavy Duty Applications, Society of Automotive Engineers, 2005), at .002 (cited in Paper 20, Patent Owner's Response, p. 22).

# Petitioner In 2003: Filter Upstream Of SCR To Preserve NO<sub>2</sub>

By placement of the PM device upstream of the SCR catalyst, the PM device can regenerate soot with NO<sub>2</sub>, and the SCR can subsequently remove NOx.

Ex. 2027 (Philip G. Blakeman, et al. Developments in Diesel Emission Aftertreatment Technology, Society of Automotive Engineers, 2003), at .010 (cited in Paper 20, Patent Owner's Response, p. 42).

# 2014: Filter Still Upstream Of SCR To Preserve NO<sub>2</sub>

For HDD applications, exhaust temperature is comparatively high, and oxidation of soot with NO<sub>2</sub> (i.e., passive soot oxidation) is highly desirable. This motivates placement of the SCR catalyst downstream of the DPF to avoid depletion of NO<sub>2</sub> prior to the DPF;

Ex. 2030 (Kenneth G. Rappé, Integrated Selective Catalytic Reductions—Diesel Particulate Filter Aftertreatment: Insights into Pressure Drop, NO<sub>x</sub> Conversion, and Passive Soot Oxidation Behavior, I&EC Research, 2014), at .002 (cited in Paper 20, Patent Owner's Response, p. 22).

# Petitioner In 2000: Filter Upstream of SCR

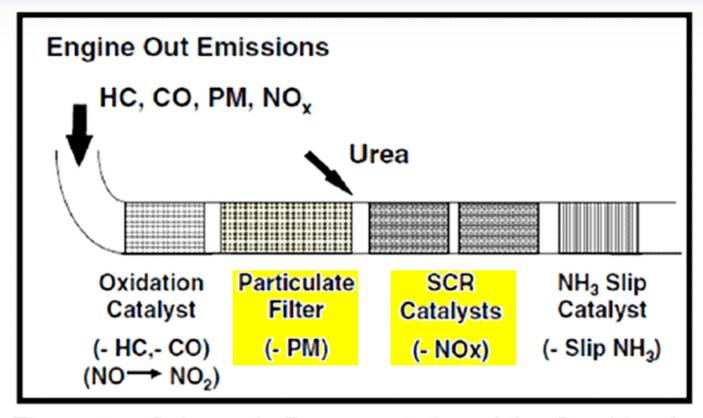
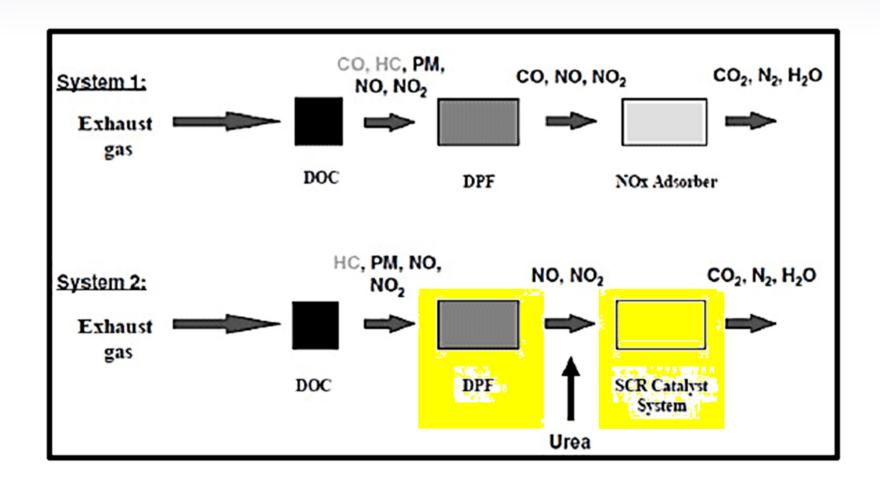


Figure 3. Schematic Representation of the Combined HC, CO, NOx and PM Control System

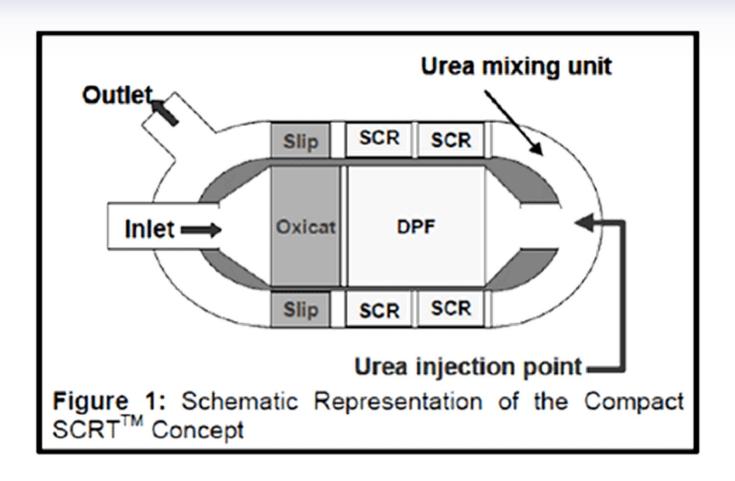
Ex. 2034 (Guy R. Chandler, et al., An Integrated SCR and Continuously Regenerating Trap System to Meet Future Nox and PM Legislation, Diesel Exhaust Aftertreatment, 2000), at .005, Fig. 3 (cited in Paper 20, Patent Owner's Response, p. 41).

# 2001: Filter Upstream Of SCR



Ex. 2035 (J. Gieshoff, et al., Regeneration of Catalytic Diesel Particulate Filters, Diesel Exhaust Emission Control: Diesel Particulate Filters, 2001), at .011, Fig. 21 (cited in Paper 20, Patent Owner's Response, p. 41).

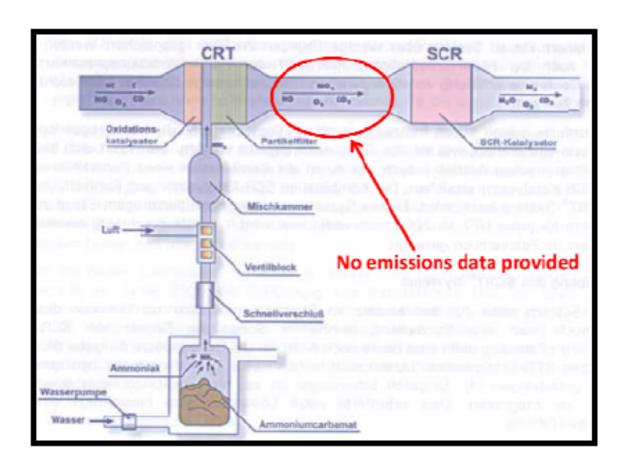
#### Petitioner in 2003: Filter Upstream Of SCR



Ex. 2019 (Andrew P. Walker, et al., The Development and Performance of the Compact SCR-Trap Systems: A 4-Way Diesel Emission Control Systems, Diesel Exhaust Emissions Control, 2003) at .004, Fig. 1 (cited in Paper 20, Patent Owner's Response, p. 44).

# Hüthwohl: No Motivation To Combine And No Reasonable Expectation Of Success

#### Hüthwohl's System



Ex. 1006 (Original German-Language Publication of Georg Hüthwohl, Bernd Maurer and Gennadi Zikoridse, The SCRT® system – a combination particle filter with SCR catalyst – enables both particle and NOx emission to be reduced simultaneously in commercial vehicle diesel engines, Proceedings of the Dresden Motor Conference, held in May 1999), at 136 (cited in Paper 20, Patent Owner's Response, p.34).

# Petitioner's Expert: NO<sub>2</sub> Is Key For Soot Oxidation

- A. So definitely in the '90s the community would have known that NO<sub>2</sub> was an <u>integral</u> part of an oxidant to help in soot reduction.
- A. As we talked about earlier, NO<sub>2</sub> is a very good oxidant of soot and is <u>key</u> to bringing the light-off temperature of soot down and enabling passive regeneration to be more easily accomplished.
- A. So that the combination of a catalyst, the presence of NO<sub>2</sub>, which reduces the oxidation temperature, those are both key to the continuous regeneration to occur.

# Petitioner's Expert: No Synergy With SCR On Filter

A. So soot traps and catalyzed soot traps were well along before it was demonstrated that you could combine or put SCR onto a diesel particulate filter. And it's kind of an obvious reason, putting an oxidation catalyst on a diesel particulate filter enhances the performance of the diesel particulate filter, whereas putting and SCR function on a diesel particulate filter brings another function into the system. So it's bringing two technologies together rather than optimizing one technology.

# **Petitioner's Expert: Avoid SCR**

A. Well, if you can avoid the SCR, then you can focus, for example, on the particulate filter by itself, and that would enable a strategy of , for example, EGR to reduce  $NO_x$  and then continue to develop the particulate filter without having to worry about yet another technology. So I guess that gets back to cost and complexity. So those are the main factors.

# Petitioner's Expert: Obvious Benefits To Avoiding SCR

A. I think also a significant cost is incurred by putting an SCR system on a vehicle. So there's obvious benefits to avoiding it as long as possible.

#### NH<sub>3</sub> Oxidation Will Take Place On The Filter

NH<sub>3</sub> oxidation is a key side reaction as it competes with the SCR reaction for the reductant ammonia and hence it is considered as an undesired side reaction. On Fe- and Cu-zeolite catalysts, most of the NH<sub>3</sub> is selectively oxidized to N<sub>2</sub> by

$$4NH_3 + 3O_2 \rightarrow 2N_2 + 6H_2O$$
,  $\Delta H = -3.12 \times 10^5 \text{ J/mol NH}_3$ 

Ex. 2031 (Pranit S. Metkar, et al., Experimental study of mass transfer limitations in Feand Cu-Zeolite-based NH3-SCR monolithic catalysts, Chemical Engineering Science, 2011), at .002

(cited in Paper 20, Patent Owner's Response, p. 24).

# Petitioner's Expert: Concerns About NH<sub>3</sub> Oxidation

- Q. Okay. What were some of the other complications that arise from having to do reduction in a net-oxidizing environment?
- A. Well, definitely the one that we've talked about, ammonia consumption by oxygen, would be a tradeoff, no doubt about that. That would be the main one.

# Petitioner's NH<sub>3</sub> Oxidation Complication Known in 2003

- Q. Okay. And this complication you mentioned about the reductant being consumed by the oxidant, that's something that was known in 2003; correct?
- A. Uh-huh.

# Petitioner's Expert: NH<sub>3</sub> Oxidation Is Main Challenge

- Q. That's what I want to know. What were the complications?
- A. Well, in a sense that's a complication because you don't know how much of that reduction will occur to supplement the reduction that is lost by ammonia consumption. So that can only be done by throwing everything together and measuring the outcome. And Hüthwohl showed that you can do it.

So I guess in answer to your question, the main challenge is ammonia oxidation. And whenever you combine two things together, you don't really know what the outcome is going to be because of all the synergies and couplings. So as Hüthwohl showed, without necessarily understanding everything, the benefits outweighed the risks.

Ex. 2025 (February 9, 2016 Harold Dep. Tr.), at 117:6-21 (cited in Paper 20, Patent Owner's Response, pp. 25-26).

# Petitioner's Expert: Put SCR On Filter To Save Space

Combining a wall flow filter with an SCR catalyst would have had 34. many well-known advantages, including a decrease in the amount of space needed to accommodate the dual functions of particulate matter removal function and NOx reduction. Performing the SCR and particulate filtration functions using separate substrates, (e.g., loading the SCR catalyst on a substrate other than the particulate filter) would likely require more space than using one substrate for both functions For example, Hüthwohl teaches about how "[c]urrent SCR catalysts cannot be accommodated in the physical volume of today's city buses," Hüthwohl at 3, and furthermore, that "[i]t is disproportionately more difficult to integrate a[n additional] particle filter" into that same system. *Id.* Space is a valuable commodity in all diesel exhaust treatment systems. Thus, a lower system volume would have many known benefits.

Ex. 1004 (Declaration of Michael P. Harold, Ph.D. ) at ¶34 (cited in Paper 20, Patent Owner's Response, p. 42).

# **Space Concerns Did Not Motivate SCR On Filter**

Finally, it should be noted that the combined demands of particulate control and NO<sub>x</sub> conversion can lead to quite complicated and large aftertreatment systems on diesel vehicles. One scenario would consist of an oxidation catalyst followed by a particulate filter, followed by a urea injection system and an SCR catalyst, followed by yet another oxidation catalyst to convert any slip of ammonia. Packaging and the ability to achieve temperatures required for the various catalytic reactions are still major question marks at the present time.

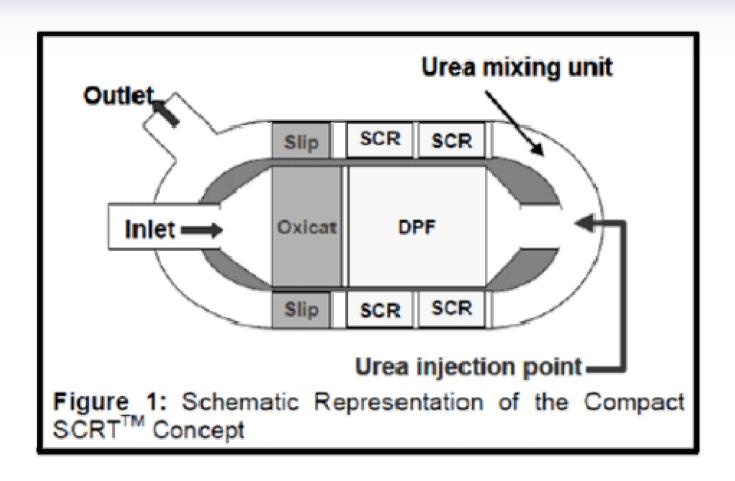
Ex. 2018 (M. Shelef & R.W. McCabe, Twenty-five years after introduction of automotive catalysts: what next?, Catalysis Today, 2000), at .012 (cited in Paper 20, Patent Owner's Response, p. 40).

# Petitioner: Space Concerns Did Not Motivate SCR On Filter

The system has been designed to be as easy to package as possible, by minimising the total volume of the system and by incorporating the SCR catalysts on annular substrates placed around the outside of the DPF-based system. This novel design gives rise to an easy-to-package emission control device capable of providing very high conversions of all four major pollutants, NOx, PM, CO and HC.

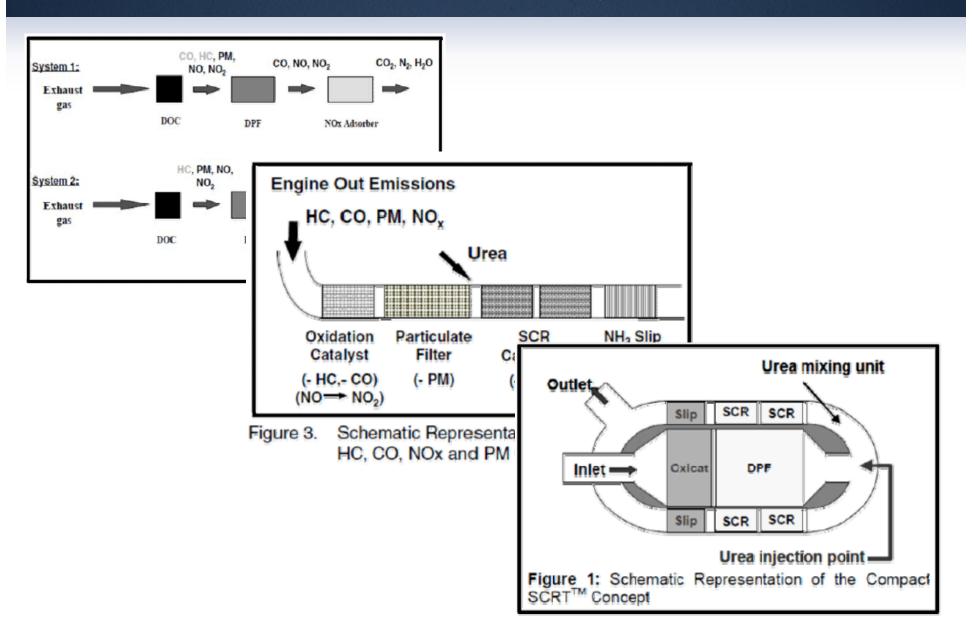
Ex. 2019 (Andrew P. Walker, et al., The Development and Performance of the Compact SCR-Trap Systems: A 4-Way Diesel Emission Control Systems, Diesel Exhaust Emissions Control, 2003) at .003.

# Petitioner in 2003: Filter Upstream Of SCR



Ex. 2019 (Andrew P. Walker, et al., The Development and Performance of the Compact SCR-Trap Systems: A 4-Way Diesel Emission Control Systems, Diesel Exhaust Emissions Control, 2003) at .004, Fig. 1 (cited in Paper 20, Patent Owner's Response, p. 44).

# The State Of The Art In 2003: Filter Upstream Of SCR



# Hüthwohl: No NO<sub>x</sub> Reduction Data

- Q. Okay. And, in fact, we talked about this earlier, we don't have any idea how much reduction of  $NO_x$  is happening in that coated particle filter, do we?
- A. We don't know.

# **Hüthwohl: No Information About The Catalyst**

- Q. You understand we talked about this earlier we don't know what kind of catalyst is being used in the Hüthwohl system; right?
- A. It doesn't really get into detail on what SCR catalyst is used.

# **Hüthwohl: No Information About Catalyst Loading**

- Q. You don't have any idea what the loading is on the particle filter, do you?
- A. No.

# **Hüthwohl: No Information About Catalyst Loading**

- Q. Okay. And it could have been that the coating on the downstream SCR catalyst is substantially higher than the coating on the upstream catalyst; right?
- A. Or it could be less.
- Q. We don't know, do we?
- A. No.

# Hüthwohl: No Information About Regeneration Mode

- Q. You don't know one way or the other whether the Hüthwohl system is doing active regeneration?
- A. I don't know.

# **Hüthwohl: No Information About Filter Stability**

- Q. He doesn't tell you anything about the stability of the system during filter regeneration; right?
- A. I don't believe so.

## Hüthwohl: Unclear About Whether SCR Is On DPF

- Q. It says: "In diesel engine operation, however, this can only" be "achieved by combining a particle filter with an SCR catalyst." Do you see that?
- A. Yes.
- Q. Okay. And so that could be referring to using an SCR catalyst downstream of uncoated DPF, right?
- A. Right.

## Hüthwohl: Unclear About Whether SCR Is On DPF

- Q. But reading [Hüthwohl] as a whole and all the statements that he's included in there, you agree with me, he's not saying that the SCR catalyst has to be coated onto the DPF; right? He's not saying that, is he?
- A. I can't draw that conclusion because I can't read his mind.

# Speronello: No Motivation To Combine And No Reasonable Expectation Of Success

# Petitioner's Motivation: Pick Catalyst To Survive Regeneration

#### It was known that filters

would often be subjected to high temperatures to "regenerate" the filter by burning off the accumulated soot. See Heck, Ex. JM 1011, at 200-01 (describing how it is "necessary to periodically regenerate the [the wall flow filter] by combustion"). When choosing an SCR catalyst, therefore, it would have been necessary to select a catalyst capable of withstanding the high temperatures needed to periodically clear the filter of soot that would otherwise begin to clog it, causing high backpressure. See, infra, § IV.A (discussing the structure of wall flow filters and the known means to clear a filter of trapped soot).

Paper 1, Petition for Inter Partes Review, pp. 27-28.

# **BASF's Patents: Regeneration Temperatures Exceed 700°C**

First, the catalyst composition must be durable so that it maintains its SCR catalytic activity even after prolonged exposure to higher temperatures that are characteristic of filter regeneration. For example, combustion of the soot fraction of the particulate matter often leads to temperatures above 700° C.

# BASF's Patents: Regeneration Temperatures Exceed 650°C

Useful SCR catalyst compositions used in the inventive system also have thermal resistance to temperatures greater than 650° C.

# Petitioner's Expert: Regeneration Temperatures Exceed 800°C

- A. If you build up enough soot in the filter, you can easily see a temperature of about 800 degrees C, which would be detrimental to a zeolite structure.
- Q. And that's even in the systems designed to primarily use passive filter regeneration?
- A. Yes. It all depends upon how the person drives.

# **Known Zeolite Catalysts Unstable At Regeneration**

Cu/ZSM-5 catalyzes NO<sub>x</sub> reduction with certain hydrocarbons but is poisoned by SO<sub>2</sub> (Feeley et al. 1995) and lacks hydrothermal stability above 600°C. Therefore, neither will be sufficient to meet emission standards for 2003.

Ex. 1011 (Chapters 8 & 9 from Ronald M. Heck and Robert J. Farrauto with Suresh T. Gulati, Catalytic Air Pollution Control (2002)), at 205 (cited in Paper 20, Patent Owner's Response, pp.29-30).

# Speronello: Zeolite Catalysts Stable Only Up To 600°C

However, by utilizing suitable zeolite catalysts in accordance with the teachings of the present invention, high temperature gaseous streams, for example, gaseous streams at temperatures up to about 600° C., may be treated without seriously adversely affecting the life or efficiency of the catalyst.

# Speronello: Zeolite Catalysts Stable Only Up To 600°C

- Q. Does the Speronello application provide any reason to believe that the zeolite catalyst disclosed in there would be hydrothermally stable at temperatures greater than 650 degrees Celsius?
- A. Probably not because Speronello was limited to 600. so if you just look at Speronello in an isolated way, basically that would confine to applications where you don't expect to exceed 600, which would be getting back to what I said earlier, CRT® application.

# Speronello: Zeolite Catalysts Stable Only Up To 600°C

- Q. Okay. So someone of skill in the art in 2003 looking at Speronello would understand this as saying the catalyst disclosed in this patent will perform adequately and hydrothermally stable up to 600 degrees Celsius; right?
- A. Yeah. The answers is the interpretation is up to 600 degrees Centigrade for these particular zeolite catalysts.

# **Speronello: Proposes Beta Zeolites**

- Q. Well, let me just ask you: Based on your memory of having studied the Speronello patent and all the work you've done in this case, what are the specific types of catalysts that you understand are recommended by Speronello? Earlier you mentioned the beta. Are those the ones that you recollect?
- A. Well, I believe that beta was one of the zeolites.
- Q. Okay. What are the other ones you understand Speronello to be recommending?
- A. That's the main one that I remember.

# **Speronello: Proposes Beta Zeolites**

- Q. So when you refer to "the Speronello catalysts" in Paragraph 42, you are referring to the copper and iron beta catalysts?
- A. Right.

# Petitioner's Expert: No Diesel Art Using Speronello

- Q. Your declaration doesn't cite any reports between 1996 and the filing of the BASF patent that involved the use of the Speronello beta catalysts for either coating a DPF or in a diesel after treatment system generally; right?
- A. Definitely not of coating a diesel particulate filter. Again, I only refer to five references in my declaration. That doesn't mean that there weren't other studies being done I know there were of copper and iron zeolite catalysts. There were a myriad of them going on in that time period.
- Q. But you don't cite any report involving the beta catalysts in the use of a diesel exhaust aftertreatment system, do you?
- A. I don't cite any, no.

Ex. 2025 (February 9, 2016 Harold Dep. Tr.), at 241:11-242:1 (cited in Paper 20, Patent Owner's Response, p.30).

# **Known Catalysts Inadequate For NO<sub>x</sub> Reduction**

#### 8.10 NO<sub>x</sub> REDUCTION TECHNOLOGIES UNDER DEVELOPMENT

One of the most important technologies that will impact the design of diesel engine exhaust treatment systems in the near future is  $NO_x$  reduction. The lean operation of the diesel engine gives rise to high fuel efficiency that, in turn, decreases  $CO_2$  emissions that contribute to the "greenhouse" effect. This advantage, however, may be offset by the inability of existing catalysts to reduce  $NO_x$  to  $N_2$  in the high- $O_2$ -content environment. The catalytic reduction of  $NO_x$  from lean-burn diesel engines has proved to be an even greater challenge relative to the stoichiometric operated gasoline engine (see Chapter 6). The modern TWC catalyst cannot reduce  $NO_x$  in the presence of excess  $O_2$ . Consequently, there is a strong driving force to develop a four-way catalytic system capable of reducing  $NO_x$  to  $N_2$  and oxidizing particulates, HC, and CO to  $CO_2$  and  $CO_2$  in the presence of excess  $CO_2$ . (Liu et al. 1996). To date no satisfactory solution has been found.

Ex. 1011 (Chapters 8 & 9 from Ronald M. Heck and Robert J. Farrauto with Suresh T. Gulati, Catalytic Air Pollution Control (2002)) at 204 (cited in Paper 20, Patent Owner's Response, p. 6).

# Petitioner's Expert: Teraoka's Zeolite Of "Dubious Use"

- Q. Now, we talked earlier about copper ZSM-5. That's the catalyst that's used in Teraoka and that's the catalyst that we know is not going to have sufficient thermal stability [in] an active filter regeneration situation; right?
- A. Well, I think it's recognized that its thermal stability is in the range of 6 to 7 hundred, so it might be of dubious use.

# Hashimoto: No Motivation To Combine And No Reasonable Expectation Of Success

# Petitioner's Expert: Stability Required Thick Walls

- Q. And so in 2005 the belief of the skilled artisan was that due to the low thermal expansion excuse me the low heat capacity of cordierite, you had to have thicker walls?
- A. Yes.
- Q. And that was the understanding in 2003 as well?
- A. Yes.

# Petitioner's Expert: Stability Required Low Porosity

- A. And when you add porosity to any filter material, you increase the tortuous path, it will decrease the effect of thermal conductance.
- Q. And that was understood not just in 2006 but in 2003 as well?
- A. Just a fundamental physical property; yes.
- Q. And that was the conventional wisdom in 2003 regarding the parameters that would impact the survivability of a filter?
- A. Absolutely.

# **Balancing Filter Porosity And Durability Was Not Routine**

With the worldwide tightening regulations, it seems that all diesel vehicles sold in these areas (Europe and Japan) will ultimately have integrated NO<sub>x</sub> and PM functions. Alternatively, selective catalytic reduction (SCR) catalysts can be used to replace NO<sub>x</sub> trap catalysts in a DPF to provide NO<sub>x</sub> reduction. Because such a system is also able to reduce CO and HC emissions through catalytic oxidation, the system is also called 4-way catalyst system. Both NO<sub>x</sub> trap and SCR 4-way systems have their own advantages and drawbacks in catalyst technology and system design. However, their NO<sub>x</sub> reduction performance (efficiency and capacity) is dependent upon the total amount of catalyst loading in DPF filter. A high porosity filter is the leading approach to achieve high catalyst storage. How to maintain the delicate balance between high porosity and thermo-mechanical durability is a challenging problem, and this paper sheds some light on the latest progress in high porosity filter product development.

Ex. 2020 (High Porosity Cordierite Filter Development For Nox/PM Reduction in Developments in Advanced Ceramics And Composites, 2005), at 2020.009 (cited in Paper 20, Patent Owner's Response, p. 45).

# Petitioner's Expert: Hashimoto Omits Durability Issues

- Q. Right. But Hashimoto didn't address the balance between high porosity and thermo-mechanical durability, did it?
- A. I don't believe so.
- Q. Okay. And that was still something that was viewed as a challenge in 2005; right?
- A. Well, according to these authors.
- Q. Do you disagree with that?
- A. No.

# **Secondary Considerations**

# Petitioner's Expert: Long Felt Need Existed

- Q. So is it fair to say that as early as 1998 the industry felt the need for a system that could meet emission standards?
- A. Yes, I think it would be fair to say.

# Petitioner's Expert: Long Felt Need Existed

A. If you came up with a solution for this problem, you would have the potential for a very large market, millions of cars, all the heavy-duty trucks.

# **Dr. Tennent: Nexus Between Claims And Emission Standards**

- Q. Okay. Since you say this was required, those things that we just referred to, the SCR coating, the high washcoating, the high porosity, since you state that those are required, the skilled artisan would agree that those were the important and critical elements for meeting the emission standard, and saving valuable space; right?
- A. So it is certainly part of the requirements. I mean, it may not be all the requirements, but it's certainly part of the requirements.
- Q. Those were important requirements; right?
- A. So those were important requirements.

# Dr. Tennent: No Art With High SCR Washcoat On Filter

- Q. So you don't actually cite any art that discloses a high SCR washcoat onto a high porosity filter; correct?
- A. Not in that, no, I do not cite that combination.

## Petitioner's Characterization Of SCR On Filter In 2008

Recently, a new technology, termed 2-way SCR/DPF by the authors, has been developed by several catalyst suppliers for diesel exhaust emission control. Unlike a conventional emission control system consisting of an SCR catalyst followed by a catalyzed DPF, a wall-flow filter is coated with SCR datalysts for controlling both NOx and PM emissions in a single catalytic converter, thus reducing the overall system volume and cost. In this work, the potential and limitations of the Cu/Zeolitebased SCR/DPF technology for meeting future emission standards were evaluated on a pick-up truck equipped with a prototype light-duty diesel engine.

Ex. 2033 (Jong H. Lee, et al., Evaluation of Cu-Based SCR/DPF Technology for Diesel Exhaust Emission Control, SAE International, 2008), at .001 (cited in Paper 20, Patent Owner's Response, p. 52).

# Dependent Claims Related To Coating Both Sides Of The Filter

# **Dependent Claims With SCR Catalyst On Both Sides Of Filter**

11. The method of claim 1, wherein the longitudinally extending walls have an inlet side and an opposing outlet side and SCR catalyst is coated on both the inlet and outlet sides of the walls.

(IPR2015-01267 Exhibit 1001, '709 patent, Claim 11).

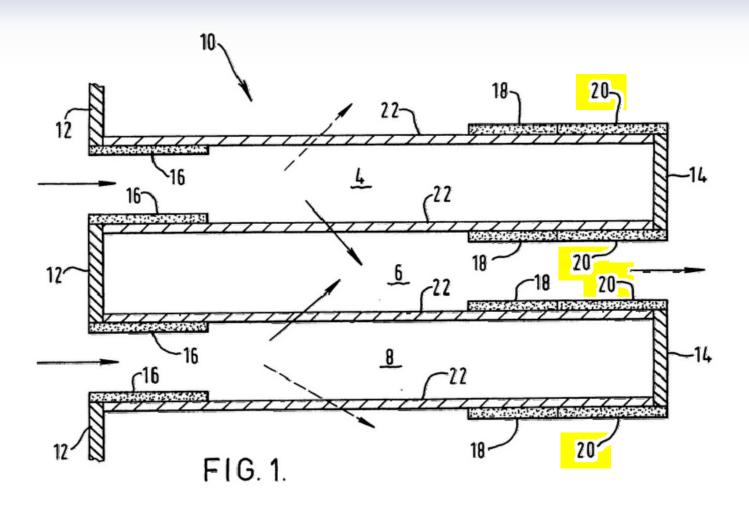
15. The catalyst article of claim 1, wherein the longitudinally extending walls have an inlet side and an opposing outlet side and SCR catalyst is coated on both the inlet and outlet sides of the walls.

(IPR2015-01266, Exhibit 1001, '982 patent, Claim 15).

11. The emission treatment system of claim 1, wherein the longitudinally extending walls have an inlet side and an opposing outlet side and SCR catalyst is coated on both the inlet and outlet sides of the walls.

(IPR2015-01265, Exhibit 1001, '023 patent, Claim 11).

# '294 Patent Only Coats The Outside Of The Filter



Ex. 1003 (Declaration of David L. Tennent, Ph.D.) at ¶44 citing Fig. 1 of U.S. Patent No. 6,753,294 (cited in Paper 20, Patent Owner's Response, p. 58).

# Petitioner's Product-By-Process Arguments Are Meritless

### **Petitioner's Arguments**

VI. Claim 11 Recites A Product-By-Process Limitation, Which Cannot Confer Patentability.

(cited in Paper 23, IPR2015-01267 Petitioner's Reply to Patent Owner's Response, p. 21).

#### The Claims

11. The method of claim 1, wherein the longitudinally extending walls have an inlet side and an opposing outlet side and SCR catalyst is coated on both the inlet and outlet sides of the walls.

(IPR2015-01267 Exhibit 1001, Claim 11).

VI. Claim 15 Recites A Product-By-Process Limitation, Which Cannot Confer Patentability.

(cited in Paper 23, IPR2015-01266 Petitioner's Reply to Patent Owner's Response, p. 21).

15. The catalyst article of claim 1, wherein the longitudinally extending walls have an inlet side and an opposing outlet side and SCR catalyst is coated on both the inlet and outlet sides of the walls.

(IPR2015-01266 Exhibit 1001, Claim 11).

VI. Claim 11 Recites A Product-By-Process Limitation, Which Cannot Confer Patentability.

(cited in Paper 23, IPR2015-01265 Petitioner's Reply to Patent Owner's Response, p. 21).

11. The emission treatment system of claim 1, wherein the longitudinally extending walls have an inlet side and an opposing outlet side and SCR catalyst is coated on both the inlet and outlet sides of the walls.

(IPR2015-01265 Exhibit 1001, Claim 11).

# **Motion to Exclude**

# **Dr. Tennent: Declaration Lacks Supporting Data**

- Q. Okay. And again, the details of that something you are not willing to provide due to confidentiality issues; right?
- A. Yes. I cannot tell you who we deployed materials to, no.
- Q. You can't even tell us who was asking for the materials; right?
- A. No, I will not tell you who was asking, other than I will tell you in general it was catalyst companies and it was the car companies and the truck companies. But I will not give you specific companies, no.

#### **CERTIFICATE OF SERVICE**

The undersigned hereby certifies that on August 18, 2016, the foregoing

#### PATENT OWNER'S DEMONSTRATIVES FOR THE 2015-01265,

2015-01266 AND 2015-01267 was served via electronic mail, upon the following

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