UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY
RESPONSE

MAY 1 4 2009

Mr. Mitchell H. Bernstein
VanNess Feldman, Attorneys at Law
1050 Thomas Jefferson Street, N.W.
Washington, DC 20007-3877

Dear Mr. Bernstein:

Thank you for your letter of April 7, 2008 to Robert Dellinger, Director of the Hazardous Waste Division, Office of Solid Waste. The letter was forwarded to me, the Director of the Office of Solid Waste for response.[1] You submitted the letter on behalf of the Alliance of Automobile Manufacturers (Alliance) for a determination that the use of the zirconium oxide coating process as part of coating operations on automotive bodies containing aluminum would not cause the downstream wastewater treatment sludge to be classified as hazardous waste F019.

In addition to the information provided in your letter, we met with representatives of the Alliance, its member companies, and outside vendors on March 19, 2008 to discuss the new zirconium oxide process. Furthermore, on November 7, 2008, you provided chemical analysis data from samples of wastewater treatment sludge from an automotive plant that has been using the zirconium oxide process. In the cover letter to this submission, you indicated that the Alliance does not believe that the analytical data is necessary for a determination that the zirconium oxide process is not chemical conversion coating within the scope of the F019 listing. Nevertheless, you noted that the data demonstrated that the wastewater treatment sludge is environmentally benign.

Based on a detailed review of the information you provided and an examination of the basis for the F019 listing, we have concluded that the zirconium oxide process as described in your various correspondence is not chemical conversion coating within the meaning and scope of the F019 listing. Therefore, wastewater treatment sludge generated from use of this process would not be covered by the F019 listing. The reasoning for this determination follows.

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[1] Note that the division and office have since been renamed to the Materials Recovery and Waste Management Division, Office of Resource Conservation and Recovery.

Chemical conversion coating is a general term that refers to processes used to protect metals from corrosion and to prepare for painting or other surface treatment. To determine whether the zirconium oxide process is included within the scope of the listing, we examined the existing record for the F019 listing. In the original listing regulations issued May 19, 1980, the listing for F006, "Wastewater treatment sludges from electroplating operations," included chemical conversion coating of aluminum as one of the covered electroplating operations. When EPA finalized the listing for F006 on November 12, 1980, EPA removed "chemical conversion coating of aluminum" from the F006 listing and added a separate listing as F019 for "wastewater treatment sludges from the chemical conversion coating of aluminum."

There is no substantive discussion on the scope of the F019 listing in the preamble to the November 12, 1980 Federal Register notice. Instead, detailed justifications for the listings are found in the specific listing background document (LBD). The LBD for F006/F019 includes the following language (emphasis added): [2, 3]

> Electroplating, as defined in this document, includes a wide range of production processes which utilize a large number of raw materials. Production processes include common and precious metals electroplating, anodizing, <u>chemical conversion coating (i.e., coloring, chromating, phosphating and immersion plating)</u>, electroless plating, chemical etching and milling and printed circuit board manufacturing.

The definition of chemical conversion coating in the LBD was more specific than the other processes listed. Thus, when EPA separated "chemical conversion coating of aluminum" from the broader F006 listing, EPA identified specific categories of metal finishing as chemical conversion coating. (In the F006/F019 listing, EPA only designated wastes from certain metal-finishing operations as hazardous, because each of the electroplating and conversion coating processes produced different types of wastes.) The LBD identified these four specific operations (coloring, chromating, phosphating and immersion plating) as being within the scope of chemical conversion coating in the F019 listing, and included a detailed discussion of each of these four processes in the LBD.

As you noted in your letter, the zirconium oxide process does not fit into any of these four categories of chemical conversion coating. The zirconium oxide process uses fluorozirconic acid as the key active ingredient in the pretreatment bath. This process is not chromating or phosphating, because it does not use chromium or phosphate. (The bath also does not contain

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[2] *Background Document, Resource Conservation and Recovery Act, Subtitle C -Identification and Listing of Hazardous Waste, §§261.31 and 261.32 -Listing of Hazardous Wastes (Finalization of May 19, 1980 Hazardous Waste List),* U.S. Environmental Protection Agency, Office of Solid Waste, November 14, 1980; Electroplating and Metal Finishing Operations.

[3] This definition of electroplating/conversion coating in the LBD is from a document EPA developed for the pretreatment standards under the Clean Water Act. In defining chemical conversion coating, this document refers principally to chromating and phosphating, but also included coloring and immersion plating processes in this category. *Development Document for Existing Source Pretreatment Standards for the Electroplating Point Source Category.* EPA No. 440/1-78/085. February, 1978.

other constituents of potential concern, e.g., cadmium, nickel, or cyanide.) The zirconium process also is not coloring, which the LBD defines as a "chemical process in which the metal surface is converted into an oxide or other insoluble metal compound." In the zirconium oxide process, the aluminum is not converted to an oxide, but rather is coated with precipitated zirconium oxide. Finally, the process is not immersion plating, which the LBD describes as a process in which "a thin metal deposit is obtained by chemical displacement" of the basis metal, such as the plating of aluminum with zinc or tin metal. As noted above, the zirconium oxide process does not deposit a layer of metal, but rather a layer of zirconium oxide. Therefore, the zirconium oxide process is not covered by any of the four processes discussed in the LBD.

In subsequent letters discussing the scope of the F019 listing, EPA has referred to the LBD to assist in determining whether various coating processes are within the scope of the F019 listing. While none of these processes were the zirconium oxide process at issue here, EPA has not determined that any other processes, other than chromating, phosphating, coloring, and immersion plating, are included in the scope of the F019 listing. Therefore, a determination that the zirconium oxide process is not covered by the F019 listing is consistent with EPA's contemporaneous (as evident by the LBD) and long-standing interpretation of the scope of the F019 listing.

We also examined the analytical data you provided for samples of waste generated from the zirconium oxide process. While these analyses do not serve as a basis for our decision that the zirconium oxide process is not included in the F019 listing, our assessment of the results indicate that the sludge generated by the process is relatively low in the potential constituents of concern (e.g., compared to constituents identified in sludge generated from the zinc phosphate process used in automotive manufacturing; see 73 FR 31756, June 4, 2008). In addition, as was noted in the information you provided, the zirconium oxide process appears to have a number of potential environmental advantages over the zinc phosphating process, in addition to the advantage of not using toxic chemicals associated with other conversion coating process. These include: the use of less energy by operating at significantly lower temperatures for less time; the production of dramatically less sludge from both the baths and the wastewater treatment system; and the significant reduction in the overall use of water.

Therefore, based on the information you provided to EPA in your letters and our meetings, the zirconium oxide process used on automotive bodies that contain aluminum is not within the scope of the F019 listing. As a result, the use of this coating process would not generate a wastewater treatment sludge classified as EPA Hazardous Waste code F019. Note, however, that while it appears unlikely based on the information you provided, the waste is question could be hazardous if it exhibits one or more of the characteristics of hazardous waste in 40 CFR 261.21 through 261.24. Finally, states are authorized to implement the hazardous waste program in lieu of the federal program, and state regulations may be more stringent than the federal regulations. Therefore, I suggest you contact the appropriate agency in the states in which this process may be used to confirm the regulatory status of this waste stream in these states.

Should you have any further questions about the applicability of the regulations, please contact Robert Dellinger, Director of the Materials Recovery and Waste Management Division, Office of Resource Conservation and Recovery at (703) 308-8252.

Sincerely,

Matt Hale

Matt Hale, Director
Office of Resource Conservation and Recovery

A PROFESSIONAL CORPORATION
1050 Thomas Jefferson Street, N.W.
Washington, D.C. 20007-3877
(202) 298-1800 Telephone
(202) 338-2416 Facsimile

VanNess
Feldman
ATTORNEYS AT LAW

Seattle, Washington
(206) 623-9372

April 7, 2008

Mitchell H. Bernstein
(202) 298-1820
mhb@vnf.com

Robert Dellinger, Director
Hazardous Waste Identification Division (MC-5304P)
Office of Solid Waste
U.S. Environmental Protection Agency
1200 Pennsylvania Avenue, NW
Washington, D.C. 20460

Re: Zirconium Oxide Pretreatment Process in the Automotive Industry

Dear Mr. Dellinger:

This letter is submitted on behalf of the Alliance of Automobile Manufacturers in support of its request for a regulatory determination that use of a zirconium oxide process as part of coating operations on automotive bodies containing aluminum will not cause downstream wastewater treatment sludge to be classified as hazardous waste F019.

Introduction

As a preliminary matter, I would like to thank you and your staff for taking the time to meet with representatives of the Alliance, two of its member companies, and two of their outside vendors, on March 19, 2008, to discuss changes taking place in the automotive industry.

Specifically, we explained that the automotive industry is in the early stages of replacing zinc phosphating on automotive bodies with a zirconium oxide process. The purpose of the meeting was to present information regarding the characteristics of the zirconium oxide process, and to highlight its many environmental and energy advantages. In addition, the two independent developers of the zirconium oxide process, Henkel and PPG, provided non-confidential information regarding their respective proprietary processes, and also discussed confidential formulas and data with CBI-authorized EPA representatives.

One of the significant features of the zirconium oxide process is that it can be used on automotive bodies that are comprised of several different metal substrates, including cold-rolled steel, galvanized steel, and aluminum. Due to the universal nature of this new process, it will not be necessary for the automotive industry to retool or to install additional pretreatment lines in order to use aluminum as one of the automotive body substrates. Increased use of aluminum means lower-weight vehicles, which in turn improves fuel efficiency and reduces emissions of greenhouse gases.

However, a significant concern has arisen regarding the possible classification of wastewater treatment sludges from the zirconium oxide process as hazardous waste F019 if there is any aluminum present on the automotive body substrate. That analysis hinges on whether the zirconium oxide process is "chemical conversion coating" within the scope of the F019 listing.

For the reasons discussed below, we believe that the zirconium oxide process is not chemical conversion coating within the intended scope of the F019 listing, and we respectfully request your office to provide us with a regulatory determination confirming that wastewater treatment sludge resulting from that process will not be F019 waste.

Benefits of the zirconium oxide process

We have attached to this letter the non-confidential descriptions of the zirconium oxide process that were prepared by Henkel and PPG, and which were distributed at our meeting. The salient features of both companies' processes can be summarized as follows.

First, the zirconium oxide process uses fluorozirconic acid as the principal active ingredient in the pretreatment bath. Unlike zinc phosphating baths that must be heated to 115° F or higher, the fluorozirconic acid bath functions at ambient temperatures at least as low as 55° F. This difference alone results in significant energy savings as compared to zinc phosphating.

Second, the zirconium oxide process also eliminates several conditioning, activation, sealing and de-ionized water rinse stages that are required in the zinc phosphating process. In addition, the water that is used in later stages of the zirconium oxide process can be "cascaded" back upstream in the process and re-used in rinses prior to the fluorozirconic acid bath (unlike zinc phosphating, where post-phosphating rinse water cannot be re-used ahead of the phosphating step). Both factors combine to significantly reduce overall water usage.

Third, the fluorozirconic acid bath does not contain cadmium, chromium, nickel or cyanide, the constituents of concern identified in the F006 and F019 listings. It also does not contain many other constituents that are used in zinc phosphating, such as manganese and phosphate. As a result, none of those constituents are found in the bath sludge, which is principally composed of non-hazardous zirconium salts that have precipitated out of solution during the reaction stage. Further, there are also significant waste minimization benefits, because the zirconium oxide process generates dramatically less bath sludge than does zinc phosphating.

Fourth, and for the same reason, the zirconium oxide process does not contribute any of those constituents of concern to, nor does it contribute significantly to the overall volume of, the facility's wastewater treatment sludge.

Fifth, the zirconium oxide process results in a deposited layer that is ultra-thin. Henkel indicates that the zirconium oxide layer is approximately 20-50 nanometers thick, and PPG indicates that it is in the range of 10-100 nanometers. Both vendors note that a zinc phosphate layer is typically in the range of 2,000-10,000 nanometers – i.e., two orders of magnitude higher. The reduced volume of deposited material means that materials handling requirements are also significantly reduced.

Sixth, the footprint of a zirconium oxide process line is approximately 30% smaller than that of a zinc phosphating line. For existing facilities, this allows changeover with relatively little disruption; for new greenfield facilities, it means smaller plants can be built.

Seventh, the zirconium oxide process meets all of the demanding quality control requirements of the automotive industry and, in fact, is actually seen as a superior process to zinc phosphating. Environmentally beneficial initiatives in the automotive industry often run into quality control issues that prevent their implementation or dilute their impact. By contrast, the zirconium oxide process has passed all quality control issues with flying colors, which will allow the automotive industry to cost-effectively realize all of its energy and environmental benefits.

The zirconium oxide process is not a "chemical conversion coating" process.

As noted above, the status of wastewater treatment sludge from the use of the zirconium oxide process on aluminum depends upon whether the zirconium oxide process comes within the scope of "chemical conversion coating" as used in the F019 listing. A careful review of the intended scope of the F019 listing, based on the discussion and response to comments in the listing background documents, as well as subsequent Agency determinations, supports the conclusion that the zirconium oxide process is outside the scope of the listing.

• History of the F019 listing

◦ May 19, 1980, regulations

The Subtitle C program was effectively established on May 19, 1980, with the promulgation of 40 C.F.R. Parts 260, 261, 262, 263, 264 and 265.[1] Some of those rules were designated as "interim final rules" on which the Agency accepted comments until July 18, 1980, including the lists of hazardous wastes set out in Subpart D of Part 261.

The May 19, 1980, list of "hazardous waste from nonspecific sources" set out in § 261.31 did not include F019. Instead, as discussed in more detail below, the listing for F006, "wastewater treatment sludges from electroplating operations," would have included chemical conversion coating of aluminum as one of the covered electroplating operations.

Due to the large number of hazardous wastes that were listed in the May 19, 1980, final rule, EPA explained that "[d]etailed justification for listing each hazardous waste. . .is

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[1] 45 Fed. Reg. 33066 (May 19, 1980). Parts 260, 262 and 263 were initially promulgated in February 1980, 45 Fed. Reg. 12722 (Feb. 26, 1980), and were amended as part of the May 19, 1980, rulemaking.

contained in specific background documents, and so will not be set forth in this preamble."[2] The Listing Background Document (LBD) is thus the reference source material for determining the Agency's intent with respect to each of the listed wastes.

The LBD for the May 19, 1980, listing of F006 provides as follows:

> Electroplating, ***as defined in this document***, includes a wide range of production processes which utilize a large number of raw materials. Production processes include common and precious metals[] electroplating, anodizing, ***chemical conversion coating (i.e., coloring, chromating, phosphating and immersion plating)***, electroless plating, chemical etching and milling and printed circuit board manufacturing.[3]

The phraseology used by the Agency is quite significant. Unlike every other process that it enumerated in the definition of electroplating operations, EPA gave a further breakdown of the operations that comprise "chemical conversion coating." Moreover, EPA used the limiting qualifier "i.e." ("that is"), rather than the more expansive "e.g." ("for example"). A fair reading of this language, in the passage where EPA is describing electroplating "as defined in this document," is that "chemical conversion coating" <u>means</u> "coloring, chromating, phosphating and immersion plating" – and no other process.[4]

- <u>November 12, 1980, regulations</u>

As noted above, the Agency accepted public comment on the "interim final" listings in the May 19, 1980, regulations. On November 12, 1980, EPA finalized the lists of hazardous waste and provided its response to comments.[5] With respect to F006, EPA took two actions to modify the listing.

First, the F006 listing was revised to exclude six specific electroplating processes that do not use the constituents of concern (chromium, cadmium, nickel and cyanides).[6] Those constituents would therefore not be expected to be present in the wastewater treatment sludge.

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[2] 45 Fed. Reg. at 33112-3 (May 19, 1980).

[3] Hazardous Waste Listing Background Document ("May LBD"), p. 83 (emphasis added, footnote omitted).

[4] The only other discussion of chemical conversion coating in the May LBD is as follows: "Chemical conversion coating most commonly involves the use of chromate or phosphate-containing baths. A number of acids can also be used (as in passivating), but are not as common as the phosphate/chromate baths." *Id.* at 84-85.

[5] 45 Fed. Reg. 74884 (Nov. 12, 1980).

[6] The six processes are: (1) sulfuric acid anodizing of aluminum; (2) tin plating on carbon steel; (3) zinc plating (segregated basis) on carbon steel; (4) aluminum or zinc-aluminum plating on carbon steel; (5)

Second, EPA added a separate listing designation, F019, for "wastewater treatment sludges from the chemical conversion coating of aluminum." The Agency's rationale was that "these sludges will not contain cadmium and nickel, two of the constituents of concern in other listed electroplating wastewater treatment sludges."[7] The F019 listing was therefore based only on the presence of chromium and cyanide.

The LBD for the November 12, 1980, regulations[8] covered both F006 and F019 in the same section. It explained the basis for the two changes just noted, but otherwise retained most of the language and discussion from the May LBD – including the definition of chemical conversion coating as "i.e., coloring, chromating, phosphating and immersion plating." In addition, the November LBD included a new section that contained EPA's responses to comments received on the May 19, 1980, listing of F006.

Of particular relevance here is EPA's response to a comment objecting "to the inclusion of wastes from chemical conversion coating operations as hazardous wastes, especially with respect to coating operations of aluminum."[9] Although EPA disagreed with the comment, based on the presence of chromium and cyanide, EPA did acknowledge that "the [May] listing background document does not provide a specific discussion on chemical conversion coating operations."[10] Consequently, although EPA retained chemical conversion coating operations in the electroplating category, it stated that "the listing background document will be revised to include a more detailed discussion of chemical conversion coating operations."[11]

That revision took the form of a detailed, two-page discussion that replaced the two sentences quoted above in footnote 4. The new discussion begins with a general description of the common characteristics of chromating, phosphating, metal coloring, and immersion plating – namely, that they "apply a coating to the previously deposited metal or basis metal for increased corrosion protection, lubricity, preparation of the surface for additional coatings or formulation of a special appearance." It then continues with a detailed discussion of each of those four

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cleaning/stripping associated with tin, zinc and aluminum plating on carbon steel; and (6) chemical etching and milling of aluminum. *See* 40 C.F.R. § 261.31 (2007) (F006 listing).

[7] 45 Fed. Reg. 74887 (Nov. 12, 1980).

[8] Background Document, Resource Conservation and Recovery Act, Subtitle C – Identification and Listing of Hazardous Waste, §§261.31 and 261.32 – Listing of Hazardous Wastes (Finalization of May 19, 1980 Hazardous Waste List), U.S. Environmental Protection Agency, Office of Solid Waste, November 14, 1980, 1941.28; Electroplating and Metal Finishing Operations ("November LBD").

[9] November LBD at 34.

[10] *Id.* at 35.

[11] *Id.* at 36.

processes – and only those four processes. There is no "catch-all" category, and no suggestion that any other process might later be categorized as chemical conversion coating.

In summary, it is very clear that EPA has always intended to define "chemical conversion coating" to include only four specific processes – chromating, phosphating, metal coloring, and immersion plating. In the next section, we show that the zirconium oxide process does not meet the November LBD's description of any of those four processes.

- **The zirconium oxide process is not chromating, phosphating, metal coloring or immersion plating.**

It is obvious that the zirconium oxide process is neither chromating nor phosphating, as it does not use any chromium, chromic acid, phosphates, or phosphoric acid.

The zirconium oxide process is also not "coloring," which is defined in the November LBD as "a chemical process in which the metal surface is converted into an oxide or other insoluble metal compound."[12] As outlined in the attachments, and explained at our meeting, the zirconium oxide process does not entail the conversion of the aluminum substrate into either an oxide or an insoluble compound. Instead, the aluminum surface reacts with the fluorozirconic acid, which causes a localized rise in pH, which causes zirconium oxide to precipitate out of solution and to be deposited on the aluminum substrate. (Also, from a more colloquial perspective, the zirconium oxide layer on the aluminum substrate has very little to no color at all, and would hardly be considered an effective "coloring" process.)

Finally, the zirconium oxide process is not "immersion plating," which is described in the November LBD as "a process in which a thin metal deposit is obtained by chemical displacement of the basi[s] metal."[13] The November LBD identifies the following metals as those that are typically deposited by this process: tin, copper, gold, nickel, and zinc.

One critical distinction between the zirconium oxide process and immersion plating is that the zirconium oxide process does not deposit a thin layer of metal, but rather deposits an ultra-thin layer of a metal oxide. Metals are conductors, while metal oxides (including zirconium oxide) are insulators of varying strength. This is important in later stages of the electrocoating process on automotive bodies, as it will affect the range and magnitude of voltage adjustments that are required in those stages.

Another critical distinction is that the "chemical displacement" of the basis metal that occurs in immersion plating is the result of oxidation and reduction reactions in the bath, where a metal salt must be reduced to the metallic state in order to be deposited. By contrast, the zirconium oxide process does not rely on any "redox" reactions for the deposition of the metal

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[12] *Id.* at 6.

[13] *Id.*

oxide; instead, it is the result of localized increases in pH that cause the metal oxide to precipitate out of solution.

Because the zirconium oxide process is not chromating, is not phosphating, is not coloring, and is not immersion plating, all as described in detail in the November LBD, it is also not chemical conversion coating within the scope of the F019 listing.

- **Prior Agency interpretations are consistent with this conclusion.**

We have exhaustively researched the available databases of RCRA interpretations, and have not found any instance in which EPA has determined that a process other than chromating, phosphating, coloring or immersion plating comes within the scope of chemical conversion coating for purposes of the F019 listing. Moreover, we have found several documents in which EPA implicitly accepts the proposition that chemical conversion coating includes only the four enumerated processes.

The most recent document is a letter dated August 30, 1996, responding to an inquiry "whether a certain 'aluminum powder preparation process' is considered chemical conversion of aluminum" because it "does not involve chromating, metal coloring, immersion plating, or phosphating as defined in the F019 listing background document."[14] The Agency responded that "[a]lthough the 'aluminum powder preparation process' does not utilize any chromate compounds, it does involve phosphating (but not zirconium phosphating) to deposit a layer of phosphate for surface preparation." The significance of this letter is that EPA did not reject the assertion that chemical conversion coating includes only chromating, metal coloring, immersion plating, and phosphating. Instead, EPA determined that the process at issue was in fact within the scope of "phosphating" as described in the November LBD.

Another supportive document is a letter dated March 7, 1991, responding to a delisting petition for wastewater treatment sludge generated at a facility that manufactured pre-sensitized aluminum plates for the lithographic industry.[15] After analyzing the petitioner's processes, the Agency stated that "none of Fuji's processes (including its silicate process) utilize any chromate compounds or involve an oxide-conversion, phosphate-conversion, or chromate-conversion coating process. Therefore, we are not convinced that Fuji's surface preparation processes (including its silicate process) fall within the scope of the chemical conversion coating processes regulated by the EPA Hazardous Waste No. F019 listing (see [November LBD])." EPA concluded that the petitioned waste was therefore not F019, and that no delisting was necessary.

Finally, in a letter dated June 24, 1987, EPA determined that use of a zirconium phosphating process to inhibit corrosion on aluminum cans was within the scope of the F019

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[14] 9444.1996(01), RO 14103.

[15] 9433.1991(01), RO11587.

listing.[16] In response to the argument that the phosphating process at issue involved amorphous coatings, while the LBD referred only to crystalline coatings, EPA stated that it intended to include both types of phosphating, and referred to two scientific texts to support its position. While we would emphatically not endorse such an approach here – i.e., looking outside the LBD to determine the Agency's intent therein – it is nevertheless instructive to note that the references cited by EPA actually support our position.

The first reference was to Chemical and Process Technology Encyclopedia (McGraw-Hill, 1974), which states that "conversion coatings are formed chemically by causing the surface of the metal to be 'converted' into a tightly adherent amorphous or crystalline coating[,] part or all of which consists of an oxidized form of the substrate metal." The second reference was to ASTM Standard B374-80, which defines conversion coating as "a process produced by chemical or electrical treatment of the metallic surface that gives a superficial layer containing a compound of the metal." As explained in detail above, the zirconium oxide process does not come within either of these broad descriptions. Nevertheless, we respectfully submit that these third party descriptions do not, and cannot, supersede the detailed regulatory definition of "chemical conversion coating" as "coloring, chromating, phosphating and immersion plating."

Analytical data

The only automotive plant in the United States where the zirconium oxide process is in use is Ford's Twin Cities plant, where operation of the new process began in late November 2007. As a consequence, there are not yet available any analytical data for the wastewater treatment sludge from the zirconium oxide process line. However, the Alliance has made arrangements for sampling and analysis of that sludge, which will be collected over a six-week period. Allowing for necessary laboratory work, preparation of reports, etc., those data will not be available until June 2008 at the earliest.

As we explained during our meeting, the reason that the Alliance is conducting the sampling is to support an alternative request for the initiation of supplemental rulemaking to amend the F019 listing, in the event that the Agency determines that the zirconium oxide process is chemical conversion coating within the scope of that listing. For all the reasons stated above, we do not believe that such a determination would be warranted, but we obviously do not presume to predict the outcome of your deliberations.

However, we do respectfully urge you to issue the regulatory determination requested in this letter without regard to the availability of that analytical data. As you noted during our meeting, the regulatory interpretation that we are seeking is a threshold determination that is independent of any analytical data: either the zirconium oxide process is chemical conversion

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[16] 9444.1987 (2), Faxback 11259. This letter was written before the F019 listing was amended in 1990 to specifically exclude "zirconium phosphating in aluminum can washing when such phosphating is an exclusive conversion coating process."

coating within the intended scope of the F019 listing, or it is not. No analytical data are required in order to make that determination.

Further, as we noted during the meeting, the wastewater treatment sludge that the Alliance is planning to sample will come from numerous other processes and operations within the Ford Twin Cities plant, and not solely from the zirconium oxide process line. Henkel and PPG have provided your office with their confidential, proprietary information regarding the chemical composition of their bath solutions. Those are the only constituents that the zirconium oxide process could possibly contribute to the facility's wastewater treatment sludge.

In summary, we respectfully submit that you need not await receipt of any analytical data from the wastewater treatment sludge at the Ford Twin Cities plant before responding to this request for a regulatory determination.

Conclusion

The automotive industry is at a cross-road as it continues its efforts to produce environmentally-friendly vehicles in an environmentally-friendly way. Increasing the use of aluminum on vehicles will improve fuel efficiency and will reduce emissions of greenhouse gases. Switching from zinc phosphating to the zirconium oxide process will save energy, use less water, minimize waste, reduce materials handling, and eliminate constituents of concern from wastewater treatment sludge. The switch will also allow smaller plants to be built, will meet all quality control requirements, and will result in cost savings to the industry.

A determination by the Agency that the zirconium oxide process is not chemical conversion coating within the scope of the F019 listing will accelerate this switch by removing an unnecessary and unintended regulatory barrier. We firmly believe that such a determination is supported by an examination of the chemistry involved in the zirconium oxide process, the language used in the relevant Listing Background Documents, the discussions in those Listing Background Documents, and prior Agency determinations. Further, we respectfully submit that no sampling of any wastewater treatment sludge is required in order to make the determination.

The Alliance is deeply appreciative of your consideration of this request, and we look forward to an expeditious response. If you have any questions, or need any further information, please do not hesitate to contact me.

Sincerely,

Mitchell H. Bernstein

Attachments

cc (w/att.): James Michael (EPA)
Robert Kayser (EPA)
Julie Becker (Alliance)

ATTACHMENTS

MEMO

Date: March 15, 2008 WEF02208

To: LaShan Haynes, US - Environmental Protection Agency

From: William Fristad

cc: ----- Non-Confidential Version -----

Subject: Zirconium Oxide Pretreatment – Henkel Bonderite® TecTalis™ Process

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Henkel and other pretreatment suppliers to the automotive industry are developing new pretreatment systems to replace the traditional tricationic zinc phosphate process, which has been the industry norm for more than 60 years. The new pretreatment systems vary in their exact chemistry from one supplier to another. The purpose of this memo is to explain the specific chemistry of Henkel's Bonderite® TecTalis product, describe some of its environmental advantages, and show how different it is compared to the traditional zinc phosphate process used during the past 50+ years. The first three pages in this memo contain generally disclosed information on the new Bonderite TecTalis product and process. The last five pages contain Confidential Business Information that must remain confidential. These are clearly marked with **CONFIDENTIAL** at the top and bottom of the relevant pages.

Background

The new zirconium oxide pretreatment technology from Henkel is called Bonderite TecTalis. It consists of a proprietary mixture of ingredients whose principal active ingredient is fluorozirconic acid. Fluorozirconic acid-based pretreatment products have become very popular since the introduction of Bonderite NT-1 approximately 5 years ago as an iron phosphate replacement. The increasing regulation and environmental concerns over Zn/Ni/Mn-phosphate is spurring the interest in replacing it as well. In the last few years, the corrosion performance of fluorozirconic acid-based pretreatments has been improved to the point that Bonderite TecTalis now meets automotive corrosion specifications to replace zinc phosphate.

Basic Chemistry

Fluorozirconic acid reacts with the metal substrate to form a zirconium oxide layer. This zirconium oxide layer is approximately 20-50 nm thick. This is much thinner than a zinc phosphate layer, which is 2-10 µm (2,000-10,000 nm), and there is much less material in the zirconium oxide layer (20-200 mg/m^2 coating weight) versus 3,000-4,000 mg/m^2 for zinc phosphate. The coating weight and thickness depends on the exact coating conditions (e.g. concentration, temperature, time). However, the largest factor controlling coating weight is the substrate, with increasing coating weights on aluminum < cold-rolled steel < galvanized steel. The atomic force micrograph below shows the coating's nodular structure (1 µm x 1 µm).

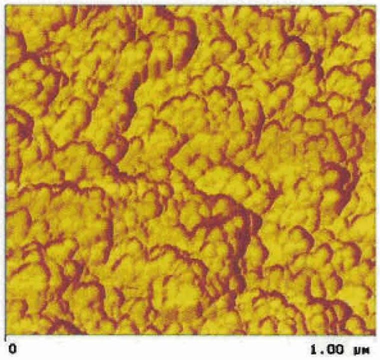

Figure 1. Atomic Force Micrograph (AFM) of a zirconium oxide coating on cold-rolled steel (CRS)

The XPS data below shows the elemental composition of a typical 20-50 nm thick zirconium oxide coating on an electrogalvanized surface. The XPS data plots the atomic percent of the various components in the coating versus the depth of the coating from the air-coating interface (zero) down into the bulk metal substrate (zinc). It is clear that the coating is composed of primarily zirconium (Zr) and oxygen (O) with a smaller amount of zinc (Zn). Underneath the coating the bulk zinc is seen in the galvanized layer. The carbon (C) detected is due to atmospheric contamination and disappears very quickly away from the air-coating interface. Based on this data from a galvanized surface, the zirconium oxide layer contains some of the substrate metal ion (Fe, Zn, or Al) within the coating. This is difficult to conclusively prove because none of these automotive substrates are perfectly flat and smooth on an atomic scale; however, this assumption is consistent with all the data

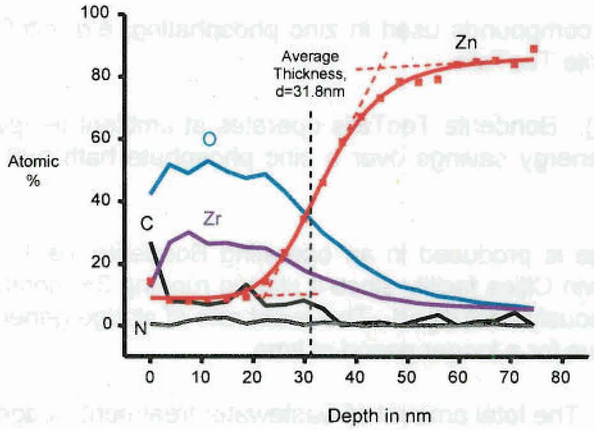

Figure 2. XPS Spectrum of a Bonderite TecTalis Coating on Electrogalvanized Steel

Bonderite® TecTalis Zirconium Oxide Pretreatment Process

The Bonderite TecTalis process is simpler than a traditional zinc phosphate process in that there are no conditioning and post rinse (final seal) stages. The only heated stage(s) are the cleaning stage(s). In the figure below, the heated stages are shown in red and ambient temperature stages shown in light blue.

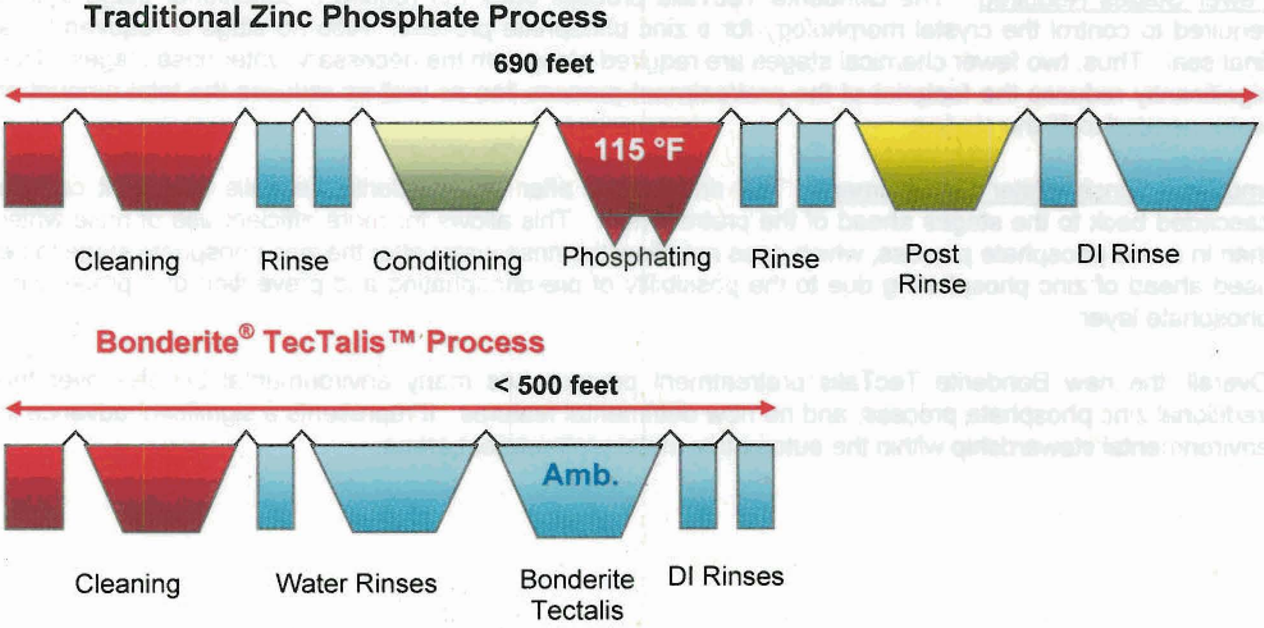

Figure 3. Comparison of Traditional Zinc Phosphate vs. Bonderite TecTalis Process

The new zirconium oxide technology can fit into an existing zinc phosphate plant by using the unnecessary stages as additional water rinses. However more importantly, a new greenfield plant can be designed with a smaller footprint that uses less water and less energy than a traditional zinc phosphate line. This has both environmental and economic value as outlined in the next section.

Environmental Advantages of the Bonderite TecTalis Pretreatment Process

Ni- and Mn-Free. Bonderite TecTalis is free of regulated nickel and its primary active ingredient is a non-regulated metal, zirconium.

Phosphate-Free. Bonderite TecTalis is totally free of phosphate. In fact phosphate is detrimental to the new process as it precipitates the active ingredient, zirconium, as zirconium phosphate.

Nitrite-Free. No typical accelerator compounds used in zinc phosphating, e.g. nitrite, hydroxylamine, or nitro-compounds, are used in Bonderite TecTalis.

Ambient Temperature (lower energy). Bonderite TecTalis operates at ambient temperature (even as low as 55 °F), so there are significant energy savings over a zinc phosphate bath that requires a constant temperature of 115 °F.

Less Bath Sludge. Very little sludge is produced in an operating Bonderite TecTalis bath. No sludge removal has occurred at the Ford-Twin Cities facility since it started running 3+ months ago. Generally, a zinc phosphate bath must be continuously desludged. The exact rate of sludge generation in the TecTalis will be determined as the product is run for a longer period of time.

Less Wastewater Treatment Sludge. The total amount of wastewater treatment sludge should be reduced. The exact amount is dependent on the production line and wastewater treatment process. An explanation for this reduced sludge generation will be more apparent in the Confidential section of this report.

Shorter Treatment Time. The new Bonderite TecTalis pretreatment builds its corrosion protective layer within 30-60 seconds, whereas a traditional zinc phosphate line requires 120-180 seconds of reaction time. This shorter time translates into a smaller tank design, lower water requirement, and a smaller footprint.

Fewer Stages Required. The Bonderite TecTalis process does not require a conditioner stage that is required to control the crystal morphology for a zinc phosphate process. Also no stage is required for a final seal. Thus, two fewer chemical stages are required along with the necessary water rinse stages. This significantly reduces the footprint of the pretreatment process line as well as reduces the total amount of water needed to fill the stages.

Improved Rinse Water Management. The rinse water after the Bonderite TecTalis treatment can be cascaded back to the stages ahead of the pretreatment. This allows for more efficient use of rinse water than in a zinc phosphate process, which does not allow the rinse water after the zinc phosphate stage to be used ahead of zinc phosphating due to the possibility of pre-phosphating and prevention of a proper zinc phosphate layer.

Overall, the new Bonderite TecTalis pretreatment process has many environmental benefits over the traditional zinc phosphate process, and no new detrimental features. It represents a significant advance in environmental stewardship within the automotive metal pretreatment arena.