

From: [Gilliam, Allen](#)
To: "little rock jeff davis"; [Little Rock Stan Suel](#)
Cc: [Fuller, Kim](#); [Blevins, Terri](#); [Pickrel.Jan@epamail.epa.gov](#); [siddiqui.ahmar@epa.gov](#); [Molina.Rudy@epamail.epa.gov](#)
Subject: AR0021806_Welspuns categorical determination for the City of Little Rock from EPA_20130114
Date: Monday, January 14, 2013 8:16:57 AM
Attachments: [1940_001.pdf](#)
[PROCESS_DESCRIPTION.PDF](#)

Jeff,

Please find the below categorical determination for Welspun from EPA HQ's Effluent Limitation Guidelines Metal Finishing "expert", Ahmar Siddiqui, and EPA's National Pretreatment Coordinator, Jan Pickrel.

It has been determined Welspun's contested phosphatizing processes and subsequently generated wastewater is subject to the Metal Finishing Standards in 40 CFR 433.17 as a core operation.

Any other ancillary operations under 40 CFR 433.10 generating and discharging wastewater to Little Rock's sewage collection system are also subject to the same limitations.

If there's further questions or guidance please feel free to contact this office.

Sincerely,

Allen Gilliam
ADEQ State Pretreatment Coordinator
501.682.0625

E/NPDES/NPDES/Pretreatment/Reports

From: Siddiqui.Ahmar@epamail.epa.gov [mailto:Siddiqui.Ahmar@epamail.epa.gov]
Sent: Friday, January 11, 2013 12:31 PM
To: Gilliam, Allen
Cc: Fuller, Kim; Pickrel.Jan@epamail.epa.gov
Subject: Re: Welspun's categorical determination request

Hi Allen,

Per the industrial process described for the Welspun facility which was attached below, EPA agrees that the Welspun phosphatizing process meets the categorizing intent of 40 CFR 433. It seems fairly clear to us that the process treats the surface aggressively enough to remove material, a basic condition of regulation by 433.

EPA also agrees that the draft guidance referred to in the request was never completed by us and should not be used in the future for conducting categorization determinations. Please let either of us know if you have any questions.

Thanks!

Ahmar Siddiqui Jan Pickrel
EAD/OST/OW WPD/OWM/OW
(202) 566-1044 (202) 564-7904

From: "Gilliam, Allen" <GILLIAM@adeq.state.ar.us>
To: Jan Pickrel/DC/USEPA/US@EPA, Ahmar Siddiqui/DC/USEPA/US@EPA
Cc: "Fuller, Kim" <FULLER@adeq.state.ar.us>
Date: 01/11/2013 12:19 PM
Subject: Welspun's categorical determination request

Jan and Ahmar,

This office is requesting a categorical determination for the above mentioned facility. Their argument and process description for this contestation is attached along with the March '95 "Draft Guidance for Distinguishing Between Phosphate Coating and Phosphate Cleaning Ops for the Purposes of Regulation under 40 CFR, Part 433 – Metal Finishing" which was not endorsed or concurred with by your Engineering and Analysis Division.

In this office's opinion their phosphatizing process is a core operation under 40 CFR 433 per the Guidance Manual for Electroplating and Metal Finishing Pretreatment Standards, page 2-2 under the paragraph titled, "4. Coatings: Phosphate coatings are formed by the immersion of steel, iron, or zinc plated steel in a dilute solution of phosphoric acid plus other reagents to condition the surfaces for cold forming operations, prolong the life of organic coatings, provide good paint bonding and improve corrosion resistance." regardless of the facility's contention that "their surface roughness necessary for coating adhesion is achieved during the abrasive blasting stage of the process".

If you have a different viewpoint and/or need additional information please advise.

Thank you for your attention to this matter.

Sincerely,

Allen Gilliam
ADEQ State Pretreatment Coordinator
501.682.0625(See attached file: 1940_001.pdf)(See attached file: PROCESS DESCRIPTION.PDF)

PROCESS DESCRIPTION

This facility manufactures steel pipes for uses such as pipelines. The pipe sizes and coatings are determined by the use intended for the product.

SPIRAL PLANT PROCESS

Steel coil is loaded on a coil car to be fed to an uncoiler and open the leading end by means of a coil opener arrangement. The opened coil is pulled and flattened before cutting and beveling the front end for joining of two strips. After the two strips are joined they are fed to the coils accumulator for continuous coil feeding. The straightened coil is inspected for any defects and further flattened before feeding it for edge beveling. The edge beveled strip is fed into the forming mill thru pinch roll and edge pre bending and continuous tack welded using metal inert gas (MIG) welding. The continuous pipe is parted off to a definite length by a plasma cutting system. The continuous tack welded pipe is then cleaned from the inside by a mechanical brush and the residue is collected by a fan that exhausts to the atmosphere through a bag filter. The formed pipe is welded on both the inside and outside using tandem submerged arc welding (SAW) welding systems. The flux and slag is removed from the pipe and collected through a bag filter. The welded pipe is inspected for any weld defects by a radiosopic system (real time radiography) and the pipe ends are faced by an end facing machine. End faced pipe is further tested in a hydro tester and weld seams (Spiral and Cross seam) are inspected for defects. Further both ends of each pipe are checked by end radioscopy and finally the pipes are beveled and checked for laminations at the beveled ends. Finally the pipes are visually inspected, weighed and marked for identification before dispatch to the yard (for coating or to the final dispatch). A process flow diagram for the Spiral Plant is shown in Figure 2.

COATING PROCESS

Internal Coating

Bare pipes go through induction preheating system where the pipes are heated up to 60°C to remove surface moisture. The preheated pipes are fed to an internal blasting machine to blast the inside surface of the pipes to generate a surface roughness of 50 to 70 microns. The blasted pipe is then sent to a blowout station where the loose dust is removed from the internal surface of the pipe. Then cutback tape is applied on the ends of the pipes to generate uncoated surfaces. Liquid epoxy paint is applied on the internal pipe surface, the pipe is then sent to a curing chamber. Hot air is used to gradually heat the internal coated pipes in a closed chamber for drying the paint. Then the pipe is fed to an end cleaning process where the masking tape is removed from the ends of the pipe. The pipe is inspected for any surface defect or pinholes and fed to the stenciling operation where an identification number is marked on the internal surface of the pipes. The internal coating process flow diagram is shown in Figure 3.

External Coating

Bare pipes go through a gas fired preheating oven where the pipes are heated up to 60°C to remove surface moisture. The preheated pipe is sent to a blasting machine to blast the outside surface to generate a surface roughness of 50 to 70 microns. The blasted pipe is sent to a blowout station where the loose dust is removed from the pipe. The pipe then enters the phosphoric acid wash cabin where phosphoric acid is applied to the blasted surface to neutralize the residual salts, followed by a high pressure water wash operation. The discharge from this activity is captured in a trench and conveyed to a pit inside the phosphate acid treatment room where the pH is adjusted prior to discharge to the city sewer system. These activities are regulated by the categorical pretreatment standards in 40 CFR 433. 17. The washed pipe is sent to an induction heating system where the pipe is heated before application of fusion bonded epoxy (FBE). The heated pipe is coated with FBE powder. The coated pipe is then sent to the

cooling tunnel and end cleaning operation where the coatings from the ends of the pipe are removed. The pipe is inspected for any surface defect or pinholes and fed to the stenciling operation where identification number is marked on the pipes. Figure 4 shows the external coating process.

There has been a question raised about the applicability of 40 CFR §433 and the requirements of the Total Toxic Organic Management Plan to the process used at both of the Welspun Little Rock facilities. This process uses a phosphoric acid wash to remove any surface contaminants prior to powder coating. It is Welspun's position that the 40 CFR 433 does not apply to our operations.

40 CFR §433 lists 6 industrial categories that have to prepare a TTOMP. They are:

- Electroplating
- Electroless Plating
- Anodizing
- Coating (Chromating, Phosphating and Coloring)
- Chemical Etching and Milling
- Printed Circuit Manufacturing

Of these categories the only one that potentially applies to our process is the Coating (Chromating, Phosphating and Coloring) category. However, these processes all involve a chemical reaction on the metal surface to change that surface. Our process is cleaning with a phosphoric acid solution to remove any residual oils not a phosphate coating (phosphating) process. The EPA memorandum dated March 23, 1995, titled "Draft Guidance for Distinguishing Between Phosphate Coating and Phosphate Cleaning Operations for the Purpose of Regulation under 40 CFR, Part 433 – Metal Finishing" demonstrates that the EPA acknowledges that cleaning with phosphoric acid is a different process than phosphate coating. This memorandum lists steps that can be used to distinguish between the processes.

Step 1 is the review of the acceptance and/or quality control specifications for specific, quantifiable measurements of physical coating properties. These include coating weight/area and coating thickness. Welspun does not track these parameters as they are not relevant to the cleaning process.

Step 2 requires a review of acceptance and/or quality control specifications for coating performance measurements. Any tests intended to measure the following coating performance parameters are associated with a phosphate coating process:

- Increased surface resistivity relative to that of the untreated base metal.
- Enhanced corrosion resistance, wear performance, or resistance to fretting.
- Any test of coating adhesion.

If acceptance and/or quality control specifications conducted on parts directly after the surface treatment in question require the types of tests described above, the process is forming a phosphate coating. Welspun does not perform these types of tests on its pipes. Pipes are cleaned then powder coated and finally visually inspected for surface imperfections prior to storage and shipment.

The following narration is an attempt to demonstrate the differences in our phosphoric acid cleaning process and a phosphate coating operation.

Phosphate coating (phosphating) is a conversion coating consisting of an insoluble crystalline metal-phosphate salt formed in a chemical reaction between the substrate metal and a phosphoric acid solution containing ions of metals (zinc, iron or magnesium).

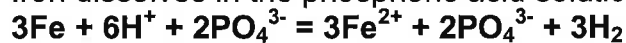
Chemistry of phosphating process

The main components of a phosphating solution are:

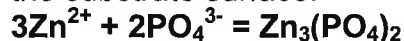
- Phosphoric acid (H_3PO_4);
- Ions (cations) of bivalent metals: Zn^{2+} , Fe^{2+} , Mn^{2+} ;
- Accelerator - an oxidizing reagent (nitrate, nitrite, peroxide) increasing the coating process rate and reducing the grain size of the deposit.

When a metal part is immersed into a phosphating solution (for example zinc phosphate) the following chemical reactions start:

Iron dissolves in the phosphoric acid solution:



Consumption of phosphoric acid for the reaction causes reduction of the acidity of the solution in the layer adjacent to the metal surface. Solubility of zinc phosphate in the neutralized solution is lowering resulting in precipitation of the salt and its deposition on the substrate surface:



A phosphating process is typically made up of the following stages:

Stages of phosphating process

- Cleaning the part is mechanically cleaned and degreased in an alkaline solution.
- Hot water rinsing at about 170°F (77°C).
- Pickling (acid cleaning). Oxide films and rust stains are dissolved in acid.
- Acid activation (if necessary).
- Water rinsing.
- Phosphating by immersion or spraying method. Typical operating temperature is about 150°F (66°C). Manganese phosphate coating is applied at 170-200°F (77-93°C). The treatment time is varying in the range 2-40 min.
- Water rinsing.
- Drying.

Our phosphoric acid cleaning does not contain any metal ions or use an accelerator. The surface roughness necessary for coating adhesion is achieved during the abrasive

blasting stage of the process. Also, our process does not follow the steps outlined above for a phosphating process. I have included the MSDS for the phosphoric acid cleaner with this document. I have also attached documents showing the particulars of the phosphate coating process. These documents were obtained through various internet sources. They show that the residence time for a phosphate coating process ranges from a 1.5 to 5 minutes. Our cleaning process has a residence time of 20 to 30 seconds.

The attached document "Surface Pretreatment by Phosphate Conversion Coatings – A Review" states:

"H₃PO₄ is an excellent time-tested **cleaning** agent which not only removes organic and inorganic solids present on the metal but also causes chemical etching of the surface by reacting with it to produce a mechanically and chemically receptive surface for **subsequent coating formation**" (emphasis added)

Also,

"Suitably cleaned surfaces are next subjected to phosphating, which causes the formation of an insoluble, corrosion resistant phosphate layer on the substrate surface. A wide variety of phosphating compositions are available. However, the right choice of the components and the operating conditions of the phosphating bath are made based on the nature of the material to be treated and its end use. **All the phosphating compositions are essentially dilute phosphoric acid based solutions containing alkali metal/heavy metal ions in them besides suitable accelerators.**" (emphasis added)

and,

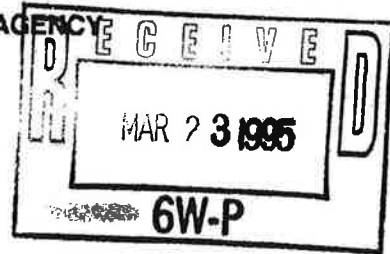
"The process of phosphating involves a consistent depletion of bath constituents and in order to obtain a satisfactory phosphate coating, the bath parameters such as: (i) the free acid value (FA) which refers to the free H⁺ ions present in the phosphating solution; (ii) total acid value TA) which represents the total phosphate content of the phosphating solution; (iii) the ratio of FA to TA, expressed as the acid coefficient; (iv) accelerator content; (v) iron content; and (vi) other metallic and nonmetallic constituents present, have to be strictly controlled within the optimum limits."

Our phosphoric acid cleaning solution does not contain metal ions or accelerator additives. We don't monitor any of the parameters listed above for our process. There is no QA/QC testing done on the pipe to determine the efficacy of the phosphoric acid wash or to determine how it affects the surface.

Based on the information outlined above, Welspun is confident that our process is an acid cleaning operation not a phosphate coating operation.



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



OFFICE OF
WATER

*For use
as a last
resort
only* 04/27/95
MAR 23 1995

MEMORANDUM

SUBJECT: Draft Guidance for Distinguishing Between Phosphate Coating and Phosphate Cleaning Operations for the Purpose of Regulation under 40 CFR, Part 433 - Metal Finishing

FROM: Mark Ingle, Project Officer, Chemicals and Metals Branch, Engineering and Analysis Division (4303) *Mark Ingle*

TO: Distribution List

I am providing the attached draft control authority guidance that describes a method for distinguishing between phosphate "coating" and "cleaning" operations for your review. This control authority guidance is intended to assist you with implementation of the Metal Finishing Point Source Subcategory regulations (i.e., 40 CFR 433). The distinction between phosphate "coating" and "cleaning" operations is important because applicability of 40 CFR 433 is determined by the use of any one of six key industrial processes at a facility. These six processes are electroplating, electroless plating, anodizing, coating (i.e., chromating, phosphating, and coloring), chemical etching and milling, and printed circuit board manufacturing. 40 CFR 433 is applicable at facilities conducting any one of these six key operations. Furthermore, if the 40 CFR 433 guidelines are applicable at a facility, effluent from any of the six processes and effluent from the other 40 industrial operations listed in 40 CFR 433 are regulated. However, if 40 CFR 433 is not applicable at a facility, effluent from the additional 40 other operations are not subject to regulation. Because the first of these additional 40 other processes is "cleaning," it is apparent that defining a phosphoric-acid-based operation as either a phosphate "coating" or a "cleaning" process can have a significant impact on a facility's regulatory status.

The process described in this memorandum will help control authority personnel distinguish between phosphate "coating" and "cleaning" processes using a hierarchical process that begins with a facility document review and ends with analytical measurement of test specimens to determine if a phosphate coating is present. This draft guidance memorandum is provided for your review and comment. Please provide your comments to me by no later than April 28, 1995. If you have any questions, or would like additional information, please feel free to contact me at (202) 260-7191 (fax: 7185).



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EXECUTIVE SUMMARY
Draft Guidance for Distinguishing Between
Phosphate Coating and Cleaning Processes for the
Purpose of Regulation under 40 CFR 433 - Metal Finishing

The draft guidance includes three steps that personnel can use to distinguish between phosphate "coating" and "cleaning" processes for the purposes of regulation under 40 CFR 433. The three steps should be conducted in the order presented to minimize costs and administrative burden. The final, Step 3, analytical measurement method involves time-consuming laboratory processes and should only be used when distinguishing between phosphate coating and cleaning operations is important.

Step 1. Review acceptance and/or quality control specifications for specific, quantifiable measurements of physical coating properties. The following are examples of typical phosphate coating physical property measurement parameters:

- A. Coating weight/area (in the United States, coating weight is frequently expressed milligrams/foot² [mg/ft²] or grams/meter² [gm/m²]).
- B. Coating thickness (typically in mils, microns, micrometers, etc.)

Specifications requiring any of these physical coating property measurements are associated with phosphate coating processes. In the absence of these physical coating property specifications, a coating performance specifications review as described in Step 2 is recommended.

Step 2. Review acceptance and/or quality control specifications for coating performance measurements. Any tests intended to measure the following coating performance parameters are associated with a phosphate coating process:

- A. Increased surface resistivity relative to that of the untreated base metal.
- B. Enhanced corrosion resistance, wear performance, or resistance to fretting.
- C. Any test of coating adhesion.

If acceptance and/or quality control specifications conducted on parts directly after the surface treatment in question require the types of tests described above, the process is forming a phosphate coating. However, if these acceptance and/or quality control tests are applied to parts only after additional treatments (e.g., painting, application of oil or wax, etc.), this Step 2 analysis cannot define if a coating was formed and analytical coating weight measurement in accordance with the Step 3 method is recommended.

Step 3. Analytically determine coating weight using the common weight/area units. For the purposes of regulation under 40 CFR 433:

- Coating weights greater than or equal to 0.2 gm/m² (or 18.6 mg/ft²) shall be considered to have been formed by a **phosphate coating process**.
- Coating weights less than 0.2 gm/m² (or 18.6 mg/ft²) shall be considered to have been formed by a **phosphate cleaning process**.

Appendix A describes the recommended weight gain/area differential measurement method. **Appendix B** describes the phosphate coating process and lists the specifications that were reviewed to arrive at the 0.2 gm/m² (or 18.6 mg/ft²) phosphate coating weight "cut-off." The following discussion presents the rationale used to develop this draft guidance, provides additional technical details, and discusses important implementation considerations.

Draft Guidance for Distinguishing Between Phosphate Coating and Cleaning Processes for the Purpose of Regulation under 40 CFR 433 - Metal Finishing

Introduction

The proposed guidance for distinguishing between phosphate coating and cleaning processes was developed to provide control authority personnel with a means of clarifying terminology used in 40 CFR 433 - Metal Finishing. The methods used to distinguish between phosphate coating and phosphate cleaning operations were developed based on analysis of current surface-finishing-industry technical reports, papers, and handbooks. Data on domestic and foreign phosphating processes were included in the review. The literature review objective was to identify those parameters that are most consistently related to phosphate coatings; and conversely, to identify those parameters associated with phosphate cleaning. The identified parameters associated with phosphate coatings were then used as the basis for the draft guidance. Although some parameters used in this guidance may seem unusual or unnecessarily complicated, unequivocally distinguishing between entire families of processes (i.e., there are at least seven distinct phosphate coating processes and hundreds of different phosphate cleaning operations) is inherently complex. Furthermore, because phosphate coatings are extremely thin (i.e., typically less than 0.001 inch) and their characteristic crystal structures can only be resolved using scanning electron microscopes, the presence of such coatings on surfaces is inherently difficult to determine.

Technical Literature Review Results

The draft guidance was developed based on a review of the technical sources presented in the reference list and an analysis of international phosphate coating specifications as presented in Appendix B. The references and specifications were reviewed to help identify those parameters associated with the phosphate coating process that could be used as the basis for process identification criteria. The literature analysis showed there are so many different phosphate coating or production operation parameters could be used to distinguish between phosphate coating and cleaning operations. Developing a distinction between phosphate coating that simultaneously clean and phosphate-coat surfaces. Because of this inherent phosphate coating process diversity, parameters related specifically to the coating formed on the metal surface were selected for use as the basis for the proposed permitting guidance. Thus, the remainder of this discussion is focused on the end-product phosphate coating physical and performance parameters that can be used to distinguishing between phosphate coating and cleaning processes.

The discussion follows the draft control authority guidance format presented in the Executive Summary. Each discussion section presents a rationale for the proposed method and provides a detailed implementation description.

Discussion of Three-Step Process Used to Distinguish Between Phosphate Coating and Cleaning Processes

- Step 1. *Review acceptance and/or quality control specifications for direct references to physical coating property measurements. The following are examples of typical phosphate coating physical property measurement parameters:*
- A. *Coating weight/area (in the United States, coating weight is frequently expressed milligrams/foot² [mg/ft²] or grams/meter² [gm/m²]).*
 - B. *Coating thickness (typically in mils, microns, micrometers, etc.)*

The end-product physical parameters most frequently mentioned in phosphate coating specifications include coating thickness and weight gain per unit area. As explained in Appendix B, phosphate coatings are formed when metal ions are released from a surface as the result of etching and then combined with available phosphorous and oxygen from the solution to form insoluble phosphate crystals at the solution/metal interface. Because phosphorous and oxygen are added to the available metal ions, a net weight gain is associated with the formation of phosphate coatings. This weight gain is a function of surface area exposed to the phosphating solution and as such units based on weight gain/area were developed. Coating weight/area is frequently measured in the United States using units of milligrams/foot² [mg/ft²] or grams/meter² [gm/m²]. As shown in Appendix B, coating weight/area units are widely used in domestic and international phosphate coating specifications.

Other physical parameters used as the basis for process specification approval include the thickness of the phosphate crystal layer and the macro-scale appearance of the coating. Although macro-scale appearance is too ambiguous a parameter to use as a basis for this regulation applicability determination, coating thickness measurements may be used. Typically, coating thicknesses are described in units of linear measurement such as mils (i.e., 0.001-inch) or microns (i.e., 1x10⁻⁶-meter). However, as shown in Appendix B, coating weight gain/area measurements are the most common physical parameters measured. Thus, any phosphate-based process at a facility that produces a coating with a measurable coating weight gain or thickness shall be considered a **phosphate coating operation**.

- Step 2. *Review acceptance and/or quality control specifications for coating performance measurements. Any tests intended to measure the following coating performance parameters are associated with a phosphate coating process:*
- A. *Increased surface resistivity relative to that of the untreated base metal.*

- B. *Enhanced corrosion resistance, wear performance, or fretting resistance.*
- C. *Any test of coating adhesion.*

Another less direct method used to identify the presence of phosphate coatings on surfaces involves measurement of performance parameters that are altered by the presence of the phosphate coating. The phosphate coating performance parameter measurements mentioned in process specifications include increased surface resistivity, enhanced corrosion control performance, increased wear (and fretting) resistance, and coating adhesion. Although it is not intuitively obvious, increases in these performance parameters reflect surface modifications that are inherently associated with coating formation and could not be caused by simple cleaning. For example, it is well known that clean, oxide-free metal surfaces conduct electricity. Thus, any performance parameter requirement for an increase in surface resistivity after a phosphate-based process is inherently caused by formation of a **phosphate coating**. Specifications calling for increased surface resistivity will frequently include units of Ohms or Ohm-square millimeter. Other units used to measure changes in performance parameters attributable to formation of phosphate coatings include:

- Corrosion Resistance: Time to first corrosion in an ASTM B117 salt fog test, weight loss/area, or time to first "red rust."
- Wear Resistance: Weight loss per number of test cycles, visual changes such as the formation of wear tracks, etc.
- Fretting Resistance: Visual changes such as the formation of pits.
- Coating Adhesion: Visual changes such as coating cracks or spalling (usually expressed relative to the diameter of the mandrel around which the specimen is bent to cause adhesion failure).

However, the use of these performance parameter measurements to identify phosphate coatings is complicated by the influence other production operations can have on the parameter measured. For example, if a part were treated with a phosphate-based solution and then sprayed with an insulating lacquer, a specification calling for an increased electrical resistivity after lacquer application could not be used to define the process as a phosphate coating operation because the lacquer alone could create any measured increase in resistivity. Similar processing steps that could confound the phosphate coating analysis include the application of corrosion control preservatives, sealants, and lubricants. Thus, analyses of these performance parameters must be conducted carefully to ensure accurate phosphate coating determinations.

★ { Unfortunately, in the "real world" of factory production, the specifications needed to make the Step 1 or Step 2 determinations discussed thus far may not exist or may not be available. Furthermore, some of the performance-parameter-based

acceptance and/or quality assurance specifications may not unambiguously distinguish between phosphate coating and cleaning operations. Thus, as a final step in the process of distinguishing between phosphate coating and cleaning processes, a method for direct measurement of coating weight/area is described in Step 3.

Step 3. Analytically determine coating weight using the common weight/area units. For the purposes of regulation under 40 CFR 433:

- *Coating weights greater than or equal to 0.2 gm/m² (or 18.6 mg/ft²) shall be considered to have been formed by a **phosphate coating process**.*
- *Coating weights less than 0.2 gm/m² (or 18.6 mg/ft²) shall be considered to have been formed by a **cleaning process**.*

The final, and most time-consuming, process for distinguishing between phosphate coating and cleaning operations involves the analytical measurement of coating weight gain/area. **Appendix A** presents the detailed laboratory instructions for performing the weight gain/area measurements (this process is based on a compendium of intentional techniques because there is no single domestic "standard). Because there is no widely-used value for the "lowest" weight gain/area that would be defined as a coating, EPA reviewed available foreign and domestic phosphate coating specifications to identify the "lowest" specified coating weight gain/area. **Appendix B**, shows the specifications included in this review. As shown in Appendix B, the lowest weight gain listed in the foreign and domestic phosphate coating specifications is 0.2 gm/m² (or 18.6 mg/ft²). EPA selected this 0.2 gm/m² (or 18.6 mg/ft²) weight gain/area as the threshold value for defining a phosphate coating. Thus, as a final, definitive step in an effort to distinguish between phosphate coating and cleaning processes, the control authority can actually measure the weight gain/area caused by a process in question and compare that measurement with the EPA standard of 0.2 gm/m² (or 18.6 mg/ft²) to classify the production process.

Although the weight gain/area measurement process is relatively straightforward, it does require careful sample handling and the use of a wet chemistry lab that has a scale capable of measuring to the nearest 0.1 milligram. Because phosphate coatings are formed by a conversion process in which metal ions are removed by etching from the substrate and then redeposited on the surface as metal-phosphorous-oxygen crystals, the weight gain/area measurements must be performed using a differential method. Simply weighing parts before and after treatment is not considered an adequate method for determining coating weight gain/area because some substrate mass is lost as part of the etching process. The differential weight gain/area measurement technique involves the following four steps:

- A: Weigh a treated part or test specimen.
- B. Chemically remove the phosphate coating.
- C. Reweigh the part.

- D. Calculate the weight change per unit area, make any process accuracy or statistical corrections required, and set the resulting value equal to the coating weight.

Appendix A includes detailed descriptions of process planning, implementation, and data reduction. The final weight gain/area values calculated using the Appendix A technique are compared with the following criteria to distinguish between phosphate coating and phosphate cleaning operations:

- Coating weights greater than or equal to 0.2 gm/m^2 (or 18.6 mg/ft^2) shall be considered to have been formed by a **phosphate coating process**.
- Coating weights less than 0.2 gm/m^2 (or 18.6 mg/ft^2) shall be considered to have been formed by a **cleaning process**.

References

1. W. Rausch, *The Phosphating of Metals*, ASM International and Finishing Publications Ltd., 1990
2. T. Cape, *Phosphate Conversion Coatings*, ASM Metals Handbook, Ninth Edition, Volume 13, "Corrosion," pages 383-388
3. *Phosphate Coatings*, ASM Handbook, Volume 5, Surface Cleaning and Finishing, 1994, pages 378-404
4. B. Gruss, *Iron Phosphating*, Metal Finishing - Organic Finishing Guidebook and Directory for 1994, Volume 92, Number 5A, May 1994, pages 21-24
5. M. Eriksson, *Zinc Phosphating*, Organic Finishing Guidebook and Directory for 1994, Volume 92, Number 5A, May 1994, pages 25-42
6. A. Ita, *Paint Pretreatments for Aluminum*, Organic Finishing Guidebook and Directory for 1994, Volume 92, Number 5A, May 1994, pages 43-51
7. E. Groshart, *Design for Finishing - Part II, Conversion Coating*, Metal Finishing, Volume 82, Number 6, June 1984, pages 69-70