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

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OFFICE OF
SOLID WASTE AND EMERGENCY
RESPONSE

Subject: Sulfuric Acid Recycling in the Semiconductor Industry

From: Elizabeth Cotsworth, Director
Office of Solid Waste

To: Jeffrey Scott, Acting Director
Waste Management Division
EPA Region IX

I have concluded, based on your memorandum of May 25, 1999 and subsequent discussions with your staff, that the spent ultra-pure sulfuric acid from the semi-conductor industry used in an industrial process to make reagent-grade sulfuric acid is not a hazardous waste under the Federal program. Under the circumstances you describe, the acid can qualify for the "use as an ingredient in an industrial process" exclusion found in the Resource Conservation and Recovery Act (RCRA) hazardous waste management regulations at 40 CFR 261.2(e)(1)(i).

You explain that the semi-conductor industry produces large quantities of this spent acid and is interested in reuse options that may be available in place of the current practice of neutralizing the acid and discharging it through wastewater treatment systems. Reuse of this material could not only benefit the industry through cost savings, but could also add to the nation's pollution prevention efforts by reducing disposal of usable chemicals and reducing the need for purchasing and producing new chemicals.

Your letter and subsequent conversations provide information about a particular process, the input materials to that process, and the products that are produced. This response addresses those facts. At any particular facility there will, of course, be additional specifics about things such as how the input materials and products are managed that should also be evaluated when making regulatory determinations.

As I understand the situation, the semiconductor industry uses ultra-pure concentrated sulfuric acid (95-97% H_2SO_4) in its manufacturing processes to clean silicon wafers before they are etched. Hydrogen peroxide (H_2O_2) is added to the acid to form the cleaning solution. The acid/hydrogen peroxide solution removes from the wafers thin mono layers of ash (formed when the wafers are heated in production) and/or photoresist (photosensitive proprietary chemicals,

typically polymethacrylate or polyethylacrylate compounds). During the cleaning processes, the hydrogen peroxide decomposes to form water, which dilutes the concentrated acid. The ultra-pure acid also picks up very small particles, such as ash and photoresist, during the cleaning process. Once enough water is formed to dilute the acid to 80% H_2SO_4, it is no longer concentrated enough to be used to clean silicon wafers.

Due to the high sensitivity of the semi-conductor production process to the presence of extraneous particles or chemicals, the quantity of contaminants contained in the used acid is so small that the acid is purer than commercially available sulfuric acid used as a raw material in other manufacturing processes. Data you provided show that the average total metals levels for three samples of used ultra-pure H_2SO_4 were well below the average total metals for three samples of technical-grade H_2SO_4. You also explained your assessment that it is unlikely that there are organics or other consitutents of concern present in the used acid. This used acid that can no longer be used in the silicon wafer cleaning process (because it is too dilute) is the material that can be used in the production of reagent-grade H_2SO_4 ($50\%H_2S0_4$).

The standard raw material manufacturing process for producing reagent-grade H_2SO_4 involves mixing highly concentrated H_2SO_4 and water to make a 50% H_2SO_4 solution, cooling this mixture by passing it through a heat exchanger, and passing the mixture through a final filter before packaging the product in drums, bottles, or tank cars. The two ingredients used in this industrial process are water and concentrated H_2SO_4, and the product is reagent-grade H_2SO_4 ($50\%H_2S0_4$). The used 80% spent sulfuric acid described above can be used as an ingredient in a virtually identical process, with filtering occurring only at the end of the process.

Section 261.2(e)(1)(i) of the Federal hazardous waste rules excludes from regulation secondary materials that are used or reused as ingredients in an industrial process to make a product. Secondary materials that are "reclaimed" are not eligible for this exclusion. While the material you describe does go through a filtration step, this particular filtration step, in our view, does not constitute "reclamation," because it occurs near the end of the process, when the acid is already in essence a completed product. As in the standard reagent-grade H_2SO_4 manufacturing process, the main purpose of the filter is to protect the mechanical integrity of product handling equipment (e.g. by ensuring that broken equipment parts do not enter into pumps) or as a final quality control step prior to packaging liquid products. The filtration removes only minute quantities of particulate matter and is designed to guarantee the physical quality of the product, not to "reclaim" the secondary material in any meaningful sense. To provide an analogy, we believe that this particular filtration step is similar to final refining of reclaimed metals (e.g., triple distilling mercury which is already 99% pure), which we have considered not to be reclamation.[1]

We also note that in this particular acid production process, water is added to the input

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[1] See for example, preamble discussion concerning refined metals at 50 FR 634 (January 4, 1985) and letter concerning 99% pure mercury that is triple distilled dated May 30, 1986, to Mr. Bruce J. Lawrence, President, Bethlehem Apparatus Company, Inc. from Matthew Straus, Chief, U.S. EPA, Waste Identification Branch, Office of Solid Waste.

acid. While this addition of water does dilute the composition of the input acid, in this particular case there is no evidence this is indicative that the input acid contains undesirable constituents that are being diluted down or that the resultant product is of marginal value. To produce this valuable product, water is a normal input in the standard production process, the water content is necessary for the final product, the dilution is not occurring to remove any hazardous waste characteristic, and the used acid input to the manufacturing process and the product produced are both similar to or lower in contaminant concentrations than the inputs and products of the standard raw material manufacturing process.

In summary, based on the specific information you provided, the used ultra-pure sulfuric acid reused to produce reagent-grade sulfuric acid would meet the "use as an ingredient in an industrial process" exclusion of 40 CFR 261.2(e)(1)(i) under the conditions you described. In addition, the facts suggest this practice is "legitimate" recycling (as defined in the guidance letter on the subject from Sylvia Lowrance, Director, EPA Office of Solid Waste to EPA Regional Hazardous Waste Management Division Directors, dated April 26, 1989).

I hope this analysis is helpful and assists in minimizing disposal of useful chemicals by removing barriers to their reuse. If you have any further questions, please have your staff call Jim O'Leary, at (703) 308-8827.

cc: EPA Regional Hazardous Waste Management Division Directors
David Jones, Region 9