

PPC 9445.1984(02)

ANALYTICAL METHODS/EP TOXICITY TEST/REFERENCE STDS.

4 APR 84

RE: WCBFR0136

MEMORANDUM

SUBJECT: Notes on RCRA Methodology and QA Activities

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TO: Addresses

This memorandum is an attempt to assist regional, state and other interested persons in keeping abreast of Agency RCRA methodology and Quality Assurance activities. I plan to send out these brief memorandums periodically. They will contain information on new test methods and guidance documents being developed, method evaluations in progress, updates on the accuracy and precision of the current RCRA methods, results of quality assurance audits (without mentioning names), as well as any other topics that you feel would be useful. The following topics will be addressed in this memo:

EP Toxicity Test

Adjustment of pH

Digestion of extracts

Testing manufactured articles

Test method evaluations in progress

New test methods under development

Waste Analysis Plans Guidance Manual

RO 12212

## Reference Standards

Before getting into these topics I just want to ask that you reflect on this memorandum and send me your comments and questions regarding the information in this memo and include suggestions for future topics.

### EP Toxicity Tests

#### Adjustment of pH

Recently, differences in the results of lead analyses between two laboratories resulted in the discovery of a problem in execution of the EP Toxicity Test. Upon examination, by the Quality Assurance Officer for Region VI, it was determined that pH adjustment was being performed using pH paper and not with a pH meter. The EP test is especially sensitive to pH adjustment and for that reason the method requires that pH measurements be made only with a pH meter. The pH strips are not accurate enough and must not be used. In addition, frequent calibration of the pH meter is important. (See SW-846 "Test Methods for Evaluation of Solid Waste," Method 1310, Step 7.13.1)

#### Extract Digestion

Please remember that all extracts must be digested prior to analysis unless it has been demonstrated, on similar samples, that digestion is not necessary. All metal test methods in SW-846 explicitly require this (see, for example, Method 7040, Step 1.0).

#### Testing Manufactured Articles

The EP toxicity test procedure requires that a representative sample of the material be prepared for extraction by crushing, cutting or grinding into pieces which can pass through a 9.5 mm sieve.

The difficulty arises with manufactured articles when the material inside is an environmental problem but is encased in a leak resistant container designed to be structurally

resistant to crushing, cutting or grinding. In rare cases where such products are an appropriate size, they may be tested without being cut-up pursuant to the Structural Integrity Procedure.

Although some batteries tend to degrade rapidly when placed in a landfill, certain batteries are manufactured in such a manner as to prevent disintegration after disposal. However, at this time the Agency has not developed standardized, EP toxicity test procedures for structurally strong articles such as batteries. EPA is considering proposing amendments to the EP Toxicity test which would allow a package designed to be structurally resistant to crushing, cutting, or grinding to be evaluated in the EP Toxicity test without being cut-up. One possibility would be to test the corrosion resistance of structurally resistant articles by submerging the article in a 1M salt (NaCl) water solution at an elevated temperature (60°-80°) for a period of one month. If no leaks occur the product can be considered corrosion resistant.

I would appreciate hearing about any other suggestions you may have with respect to this issue. We hope to be able to develop a proposed amendment package in the near future.

#### Methods Evaluation

The Agency has initiated an extensive research program to determine the accuracy and precision of methods currently in SW-846. At the present time, the following methods are being evaluated.

- # 1110 Corrosivity Toward Steel
- # 1120 Polarization Resistance Method
- # 3030 Acid Digestion of Oils, Greases, or Waxes
- # 3040 Dissolution Procedure for Oils, Greases, or Waxes
- # 3050 Acid Digestion of Sludges
- # 3010 Acid Digestion Procedure for Flame Atomic Absorption Spectroscopy
- # 3020 Acid Digestion for Furnace Atomic Absorption Spectroscopy
- # 7190 Chromium: Atomic Absorption, Direct Aspiration
- # 7191 Chromium: Atomic Absorption, Furnace Method
- # 7195 Hexavalent Chromium: Coprecipitation

- # 7196 Hexavalent Chromium: Colorimetric
- # 7197 Hexavalent Chromium: Chelation - Extraction
- # 7198 Hexavalent Chromium: Differential Pulse  
Polarography Method
- # XXXX Hexavalent Chromium: Ion Chromatography

## Method Development

Our efforts continue with respect to the development of additional methods for identifying hazardous wastes. Protocols are being developed or existing methodology is being modified to address the following areas:

### Ignitable Solids

The objective is to develop methods for use in the definition of ignitable solids. Protocols have been developed and subjected to single laboratory evaluation using actual waste samples. Test have b

### Hazards Posed by Liquids with Flash Points below 60°C

The objective is to develop a single test for identifying those liquids that should not be considered as hazardous even though they will flash at a temperature below 60°C. Many such materials will not sustain combustion nor release sufficient amounts of energy to surrounding materials to spread the fire. The evaluation report of this method is also Scheduled to be prepared and available for regional review and comment by the end of 1984.

### Reactive Gases - Cyanide and Sulfide

The objective is to develop a method for determining when a waste is a reactive waste (40 CFR 261.33) by reason of potential H<sub>2</sub>S or HCN release. A method has been evaluated using standards and actual waste samples. The test method is expected to be available for Regional review and comment by June of 1984. Work is also progressing on establishing

reactive waste definition thresholds using the method. While it looks like it works acceptably well for sulfide-bearing wastes, further method refining will be necessary before it can be adopted for cyanides.

## Waste Analysis Plans Guidance Manual

Under Section 3004 of RCRA, EPA promulgated standards applicable to owners and operators of hazardous waste management facilities. These standards govern the issuance of permits for facilities that treat, store, or dispose of hazardous waste. OSW is currently developing Permit Guidance Manuals to describe the permit application process and to provide guidance to applicants and permit writers in addressing the information requirements.

As part of the permit application, owners/operators are required to submit a Waste Analysis Plan. The requirement for a Waste Analysis Plan is to insure that owners or operators possess sufficient information on the properties of wastes so that they will be able to treat, store, or dispose of the waste in a manner which will not pose a threat to human health or the environment.

To assist permit applicants and State and EPA staff members who review applications and draw up permits, the Office of Solid Waste is in the process of preparing a Waste Analysis Plan Guidance Manual. This Manual will provide specific guidance on how to comply with the general waste analysis requirements of 40 CFR 264.13. The Manual will include a discussion of the Waste Analysis Plan requirements, model Waste Analysis Plans for each of the principal waste disposal management situations, and a checklist for reviewers to use in evaluating permit applications. We anticipate that a draft of this manual will be available for Regional review early in Spring 1984.

## Reference Standards

Since 1980, EPA's Office of Research and Development has maintained and continues to expand an inventory of standard compounds for use in analytical efforts.

Organic standards consist of either single-component solutions (for instrument calibration) or multi-component solutions, containing several chemicals. Analytical reference standards can be utilized in several ways to enhance sample analysis and quality control. Reference standards can be:

- added to media before analysis to check recoveries and thus be used as a matrix spike;
- added to a sample which has been prepared for instrumental analysis, and thus be used as an internal standard;
- used as a surrogate for a particular compound allowing for both sample analysis and recovery verification to be done in the same run, e.g., deuterated or fluorinated standards can thus be used as surrogates for compounds found in hazardous waste.
- used for instrument calibration.

A single source of standard chemicals of known purity and reference materials is necessary to assure that data of known quality are produced. The Quality Assurance Materials Bank provides reference standards to analytical laboratories to support the Agency's program for monitoring hazardous waste (RCRA/CERCLA). Pure ("neat") compounds are purchased and analyzed and low purity compounds are purified. Once verified, high purity organic and inorganic standards are prepared (standard solutions) and distributed for use by laboratories in calibration of instruments and for quality control in sample analysis. The purity, concentration, stability and applicability of each standard is evaluated by the QA Materials Bank. For information regarding the availability of specific standards contact Ed Kantor at EMSL-LV (702-798-2690; FTS545-2690), Ed Berg at EMSL-Cinn (513-684-7325; FTS-684-7325), or Florence Richardson at the Office of Solid Waste (202-382-4801; FTS-382-4801).