



U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

JUL 3 0 2014

Gregory Sutherland Ph.D. (DGSA) Shane Havoc Consulting, LLC 1905 English Ivy Ct. Mount Pleasant, SC 29464

Ref. No. 13-0227

Dear Mr. Sutherland:

This is in response to your November 22, 2013 e-mail requesting clarification of the Hazardous Materials Regulations (HMR; 49 CFR 171-180) applicable to the classification of Class 8 (corrosive material). Specifically, you request guidance on authorized methods for determining the corrosivity of a solid substance.

For purposes of the HMR, a Class 8 (corrosive material) means a liquid or solid that causes full thickness destruction of human skin at the site of contact within a specified period of time (see § 173.136). A liquid, or a solid which may become liquid during transportation, that has a severe corrosion rate on steel or aluminum based on the criteria in §173.137(c)(2) is also a corrosive material. Whenever practical, *in vitro* test methods authorized in §173.137 of the HMR should be used to determine whether a material is corrosive.

The corrosivity of a solid substance must be determined using data obtained from tests conducted in accordance with the Organization for Economic Cooperation and Development (OECD) Guideline for the Testing of Chemicals, Number 430, "In Vitro Skin Corrosion: Transcutaneous Electrical Resistance Test (TER)" or Number 431, "In Vitro Skin Corrosion: Human Skin Model Test" or Number 404, "Acute Dermal Irritation/Corrosion." For a solid substance, testing protocol varies depending on the method used but, generally, requires that a solid substance be slightly wetted once in place for testing. The OECD Guidelines can be downloaded at the following link:

http://www.oecd.org/env/ehs/testing/oecdguidelinesforthetestingofchemicals.htm

I trust this information is helpful. Please contact us if you require further assistance.

Sincerely,

T. Glenn Foster

Chief, Regulatory Review and Reinvention Branch

Standards and Rulemaking Division

# Drakeford, Carolyn (PHMSA)

Stevens §172.101 §173.137 (2)(2)

From:

Billings, Delmer (PHMSA)

Sent:

Monday, November 25, 2013 1:54 PM

To: Subject: Drakeford, Carolyn (PHMSA) FW: Corrosive Solids Class 8

Attachments:

UN 18th Class 8.pdf; 13533.pdf; 9045d.pdf

\$173.136 Classification

Please log for response and assign to a specialist.

Thanks

Del

**From:** Gregory Sutherland [mailto:grsuthe@attglobal.net]

Sent: Friday, November 22, 2013 4:15 PM

**To:** Billings, Delmer (PHMSA) **Subject:** Corrosive Solids Class 8

How are you doing, Washington appears to be a confused place at this time.

I have been looking for a test that determines if a solid is corrosive or not. This is to review if a material should be classified as such.

# The 49 CFR States

49 CFR §173.136 Class 8--Definitions.

- (a) For the purpose of this subchapter, "corrosive material" (Class 8) means a liquid or solid that causes full thickness destruction of human skin at the site of contact within a specified period of time. A liquid, or a solid which may become liquid during transportation, that has a severe corrosion rate on steel or aluminum based on the criteria in §173.137(c)(2) is also a corrosive material. Whenever practical, in vitro test methods authorized in §173.137 of this part or historical data authorized in paragraph (c) of this section should be used to determine whether a material is corrosive.
- (b) If human experience or other data indicate that the hazard of a material is greater or less than indicated by the results of the tests specified in paragraph (a) of this section, PHMSA may revise its classification or make the determination that the material is not subject to the requirements of this subchapter.

The UN Class 8 section(attached) is about the same, but mentions water.

What I get so far is that if it is a solid it doesn't not get metal coupon tests done. So do you just put the solid on the test surface (membrane)?

Or do you add water?

The only process I can find is from the EPA.

Any thoughts. (I am looking at a starch that has 25% Acetic Acid sprayed on it and is a solid when done.)

Call if you want to not write an e-mail.

Gregory Sutherland Ph.D. (DGSA) Shane Havoc Consulting, LLC 1905 English Ivy Ct. Mount Pleasant, SC 29464 (843) 849-1463 Cell: (260) 414-4335 FAX: (561) 423-3907 PPC 9443.1992(01)

# CORROSIVITY CHARACTERISTIC AS IT APPLIES TO SOLIDS

United States Environmental Protection Agency Washington, D.C. 20460 Office of Solid Waste and Emergency Response

March 9, 1992

Charles A. Licht, President CLEA, Inc., P.O. Box 315 Olympia Fields, Illinois 60461

Dear Mr. Licht:

I am writing in response to your letter of February 13, 1992 to Sylvia Lowrance concerning clarification of the corrosivity characteristic as it applies to solids.

The current characteristic defines a corrosive waste as a solid waste that: a) is aqueous and has a pH less than or equal to 2 or greater than or equal to 12.5 b) or is a liquid and corrodes steel at a rate greater than 6.35 mm per year. We have two methods in SW-846 for measuring the corrosivity of these liquids. One method is for aqueous liquids (Method 9040, pH electrometric measurement) and the other method is for non-aqueous liquids (Method 1110, corrosivity of steel).

We realize that the existing corrosivity characteristic has two problems: 1) it applies only to liquid wastes, thus corrosive solids such as lye, solid acids, or in your case, baghouse dusts, are not covered and 2) the term "aqueous" (as in aqueous liquid) has not yet been defined, thus, there is a chance that some users of our method for pH determination will incorrectly apply the method to certain non-aqueous wastes.

With respect to solids, the Office of Solid Waste has developed a method that appears to be suitable for determining their corrosivity. This method, Method 9045 - Soil and Waste pH (copy attached), mixes a waste sample with water in a 1:1 ratio and determines the pH of the solution with a pH meter. Method 9045 will be included in the proposal for the second update to the third edition of sW-846, "Test Methods for Evaluating Solid Waste,

Physical/Chemical Methods." We anticipate the proposal appearing in the Federal Register sometime this spring, with promulgation sometime in 1993.

Promulgation of Method 9045 would not by itself expand the scope of the corrosivity characteristic. This requires that the Environmental Protection Agency (EPA) draft regulatory language to include corrosive solids and to employ Method 9045 for their determination. We are not sure at this time when that will happen. In the interim, we recommend the use of Method 9045 for the determination of corrosive solids.

If you have any additional questions, please call Ollie Fordham of my staff at (202) 260-4778 or call the Methods Information Communications Exchange (MICE) at (703) 821-4789. MICE calls are recorded on an answering machine and, for the majority of questions, responses are provided within 24 hours. I hope this information has sufficiently addressed your questions.

Sincerely yours, Gail Hansen Chief, Methods Section (OS-331)

Enclosure

cc: Alec McBride, Ollie Fordham, Kim Kirkland, Rafael DeLeon (OGC)

### METHOD 9045D

## SOIL AND WASTE pH

### 1.0 SCOPE AND APPLICATION

1.1 This method is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

### 2.0 SUMMARY OF METHOD

2.1 The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

# 3.0 INTERFERENCES

- 3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.
  - 3.2 Temperature fluctuations will cause measurement errors.
- 3.3 Errors will occur when the electrodes become coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

#### 4.0 APPARATUS AND MATERIALS

- 4.1 pH meter with means for temperature compensation.
- 4.2 Glass electrode.
- 4.3 Reference electrode -- A silver-silver chloride or other reference electrode of constant potential may be used.
- NOTE: Combination electrodes incorporating both measuring and referenced functions are convenient to use and are available with solid, gel-type filling materials that require minimal maintenance.
  - 4.4 Beaker -- 50-mL.
  - 4.5 Thermometer and/or temperature sensor for automatic compensation.
  - 4.6 Analytical balance -- capable of weighing 0.1 g.

#### 5.0 REAGENTS

- 5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.
- 5.3 Primary standard buffer salts are available from the National Institute of Standards and Technology (NIST) and should be used in situations where extreme accuracy is necessary. Preparation of reference solutions from these salts requires some special precautions and handling, such as low-conductivity dilution water, drying ovens, and carbon-dioxide-free purge gas. These solutions should be replaced at least once each month.
- 5.4 Secondary standard buffers may be prepared from NIST salts or purchased as solutions from commercial vendors. These commercially available solutions, which have been validated by comparison with NIST standards, are recommended for routine use.

# 6.0 SAMPLE PRESERVATION AND HANDLING

Samples should be analyzed as soon as possible.

#### 7.0 PROCEDURE

## 7.1 Calibration

- 7.1.1 Because of the wide variety of pH meters and accessories, detailed operating procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.
- 7.1.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value. If an accurate pH reading based on the conventional pH scale [0 to 14 at 25  $\cdot$  C] is required, the analyst should control sample temperature at 25 ± 1  $\cdot$  C when sample pH approaches the alkaline end of the scale (e.g., a pH of 11 or above).
- 7.2 Sample preparation and pH measurement of soils:
- 7.2.1 To 20 g of soil in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 min. Additional dilutions are allowed if working with hygroscopic soils and salts or other problematic matrices.
- 7.2.2 Let the soil suspension stand for about 1 hr to allow most of the suspended clay to settle out from the suspension or filter or centrifuge off the aqueous phase for pH measurement.

- 7.2.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrodes into the sample solution in this manner. For combination electrodes, immerse just below the suspension.
- 7.2.4 If the sample temperature differs by more than 2 € from the buffer solution, the measured pH values must be corrected.
- 7.2.5 Report the results as "soil pH measured in water at \_\_• €" where "\_• €" is the temperature at which the test was conducted.
- 7.3 Sample preparation and pH measurement of waste materials
- 7.3.1 To 20 g of waste sample in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 min. Additional dilutions are allowed if working with hygroscopic wastes and salts or other problematic matrices.
- 7.3.2 Let the waste suspension stand for about 15 min to allow most of the suspended waste to settle out from the suspension or filter or centrifuge off aqueous phase for pH measurement.
- NOTE: If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 mL of reagent water.
- NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned (Step 3.3) if it becomes coated with an oily material.
- 7.3.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant to establish good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrode into the sample solution in this manner. For combination electrodes, immerse just below the suspension.
- 7.3.4 If the sample temperature differs by more than 2 € from the buffer solution, the measured pH values must be corrected.
- 7.3.5 Report the results as "waste pH measured in water at \_\_• €" where "\_• €" is the temperature at which the test was conducted.

# 8.0 QUALITY CONTROL

- 8.1 Refer to Chapter One for the appropriate QC protocols.
- 8.2 Electrodes must be thoroughly rinsed between samples.

### 9.0 METHOD PERFORMANCE

9.1 No data provided.

# 10.0 REFERENCES

- 1. Black, Charles Allen; Methods of Soil Analysis; American Society of Agronomy: Madison, WI, 1973.
- 2. National Bureau of Standards, Standard Reference Material Catalog, 1986-87, Special Publication 260.

### METHOD 9045D

# SOIL AND WASTE pH

