

METHOD 1050

TEST METHODS TO DETERMINE SUBSTANCES LIKELY TO SPONTANEOUSLY COMBUST

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method provides test procedures which may be used to evaluate and categorize liquid and solid wastes that are likely to spontaneously combust. The method is based on the DOT regulations for the transport of spontaneously combustible materials as provided in 49 CFR Part 173, Appendix E. These test procedures are intended to identify two types of wastes with spontaneous combustion properties:

- Wastes (including mixtures and solutions, liquid or solid) which, even in small quantities, ignite within five minutes of coming in contact with air. These wastes are the most likely to spontaneously combust and are considered to have pyrophoric properties.
- Other solid wastes which, in contact with air and without an energy supply, are susceptible to self-heating. These wastes will ignite only when in large amounts (kilograms) and after long periods of time (hours or days) and are considered to have self-heating properties.

1.2 This method is not required by the Federal regulations to determine if a waste passes or fails the characteristic of ignitability according to the RCRA regulatory definition at 40 CFR 261.21. To clarify, the test results of this method cannot be used to directly classify a waste as a D001 hazardous waste, nor can the results be used by themselves to definitively classify a waste as non-hazardous. No specific test for ignitable solids is required by the RCRA regulations, and only a waste meeting the narrative regulatory definition at 40 CFR 261.21(a)(2) is an ignitable hazardous waste because it is an ignitable solid. In addition, the test in this method for a liquid may be helpful in determining whether a liquid waste has hazardous properties; however, a liquid waste is only a D001 hazardous waste if it is an oxidizer, as defined at 40 CFR 261.21(a)(4), or based on its flash point, as defined at 40 CFR 261.21(a)(1). This test method was developed to specifically illustrate the ignitable properties of materials, including wastes, and its results provide relevant and useful information that may be applied to knowledge of a waste in determining whether a waste meets the 40 CFR 261.21 criteria.

1.3 This method provides three separate test procedures for evaluating the spontaneous combustion properties of solid and liquid wastes. Refer to Sec 3.0 for definitions of some terms.

1.3.1 Test Method A is designed to evaluate the potential of a solid waste to spontaneously combust when exposed to air and may be used to classify pyrophoric solids.

1.3.2 Test Method B is used to evaluate and classify liquids that demonstrate pyrophoric properties.

1.3.3 Test Method C is used to evaluate a solid waste for self-heating properties when exposed to a test temperature of 140 °C and to determine whether the waste will undergo spontaneous ignition under the experimental conditions. Test Method C is not suitable for liquid wastes.

1.4 Prior to employing this method, analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytical objectives.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, properly experienced and trained personnel. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Test Method A -- Pyrophoric solids

Test Method A is performed to determine if a solid ignites within five minutes of coming in contact with air at ambient room temperature. A representative sample of waste is dropped from a height of one meter onto a non-combustible surface, the waste is observed to determine whether the waste ignites while dropping or within five minutes of settling. If ignition is not observed, this test is repeated five times or until a positive result is obtained, whichever occurs first. The results of this test may be used to classify a solid waste as having pyrophoric properties.

2.2 Test Method B -- Pyrophoric liquids

Test Method B consists of a two part procedure that may be used to determine if a liquid waste ignites when exposed to air at ambient temperature. In the preliminary test, a representative liquid sample is added to an inert carrier in a porcelain cup at ambient temperature and the mixture is observed to determine if the liquid ignites within five minutes. If ignition is not observed, this test is repeated five times or until a positive result is obtained. If no ignition occurs, the liquid waste is then delivered onto a Whatman filter paper to determine whether ignition or charring occurs on the filter paper within five minutes. If ignition or charring is not observed, this part of the test is repeated two times or until a positive result is obtained, whichever occurs first. The results of this test may be used to classify liquids as having pyrophoric properties.

2.3 Test Method C -- Self-heating wastes

CAUTION: Test A must be run before Test C is conducted. If the waste is pyrophoric, then Test C must not be run on the waste. Also, this procedure is not suitable for liquid wastes.

2.3.1 Test Method C may be performed to determine whether a solid waste exhibits self-heating properties. This test procedure is limited to granular solids, pastes, and other solid wastes that can be reduced in particle size to fit into a 25-mm or 100-mm stainless steel cube. Solid wastes that show pyrophoric properties as demonstrated by Test Method A must not be evaluated by this test.

2.3.2 In a preliminary test, a 100-mm sample cube is exposed to a test temperature of 140 ± 2 °C for 24 hours and the sample is observed to determine whether it undergoes spontaneous ignition or a rise in sample temperature to over 200 °C within 24 hours. If the results of the preliminary test are positive, a second test using a 25-mm sample cube is conducted to further classify the waste. See Table 6 for DOT Classification of self-heating solid wastes.

2.4 The presence of any smoke, incandescence, or flame under the test conditions provided should be interpreted as a positive result for spontaneous combustion. A positive result for Test Method A or B is readily demonstrated by smoke, flame, charring of the paper filter, and a positive result for Test Method C is demonstrated by spontaneous ignition or a significant increase in sample temperature. The analyst should be consistent with ignition recognition and record any visible signs of combustion including smoke, heat, flame, or incandescence. Ignition in any of the trials is considered a positive result, even when the results of the other trials are negative.

3.0 DEFINITIONS

The following definitions are specific to this method and are intended solely as guidance to assist the user in properly classifying wastes that are liable to spontaneous combustion.

3.1 Pyrophoric solids -- For the purpose of this method, a pyrophoric solid is any solid that spontaneously ignites when tested in accordance with the test procedure given in Sec. 11.1 of this method. Ignition is demonstrated by the presence of observable smoke, flame, or incandescence.

3.2 Pyrophoric liquids -- For the purpose of this method, a pyrophoric liquid is any liquid that spontaneously ignites or chars filter paper when tested in accordance with the test procedures given in Secs. 11.2 and 11.3 of this method. Ignition is demonstrated by the presence of observable smoke, flame, incandescence, or the charring of the filter paper.

3.3 Self-heating substances -- For the purpose of this method, a self-heating substance is any solid that spontaneously ignites or shows an increase in temperature above 200 °C when tested in accordance with the test procedure given in Sec. 11.4 of this method.

4.0 INTERFERENCES

No specific interferences for this test have been determined. The presence of any smoke, incandescence, or flame under the test conditions provided should be interpreted as a positive result for spontaneous combustion.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment.

5.2 This test requires the combustion of potentially highly flammable materials and the possible generation of toxic fumes. All tests must be conducted in a suitable fume hood fitted with a pull-down sash to prevent the escape of toxic fumes into working areas. The analyst should wear appropriate protective clothing, including a laboratory jacket or apron, safety glasses, and protective gloves. The laboratory should have appropriate fire fighting equipment (such as a Class A fire extinguisher) readily available to extinguish small fires.

5.3 This test may need to be conducted in a specially designed facility if proper surveillance and safety precautions are not available in the laboratory. Analysts are encouraged to use a flammability screening test such as ASTM 4982 prior to this procedure to test for highly flammable wastes.

5.4 Once the experiment is started, the oven should not be opened, especially if the sample temperature is observed to rise above 140 °C. Opening the oven when the sample is at an elevated temperature could increase the potential for a fire. Therefore, the oven and sample temperature should be allowed to return to ambient temperature before the oven is opened.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 Stop watch or clock -- Any timing device.

6.2 Measuring device, one meter -- Any measuring device capable of measuring one meter.

6.3 Low-heat conducting, non-combustible, impervious ceramic tile or equivalent material of sufficient size to support a 1-2 g test sample dropped from the height of one meter.

6.4 Porcelain or metallic crucible, low, wide form, 250-mL capacity, with a 4-inch (approximately 100-mm) diameter opening, heat resistant.

6.5 Whatman No. 3 filter paper, of sufficient diameter to support 0.5 mL of a test liquid sample.

6.6 Glass beaker with sufficient diameter to support the Whatman No. 3 filter paper across its opening.

6.7 Silica gel, fine granules.

6.8 Syringe, glass dropper, or disposable glass pipet -- To deliver a 5-mL test liquid sample.

6.9 Circulating type of oven with an inner volume of more than 9 L and capable of controlling the internal temperature at 140 ± 2 °C.

6.10 Wire mesh cubic sample containers, 25 mm x 25 mm and 100 mm x 100 mm sizes, made of stainless steel net with a mesh opening of 0.05 mm, with open tops.

6.11 Wire mesh outer container, mesh size 0.595 mm, 150 mm x 150 mm x 250 mm (L x W x H). Used to house the 25-mm and 100-mm wire mesh cube containers.

6.12 Two Chromel-Alumel thermocouples (Omega Engineering part number TJ36-CAIN-18U-18), or equivalent device, for monitoring oven and sample temperatures.

6.13 Two single-point electronic thermometers (Omega Engineering part Number HH-81), or equivalent device, for continuous temperature monitoring and recording.

7.0 REAGENTS AND STANDARDS

This procedure does not require any standards or reagents.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Sample collection

Pyrophoric solids and liquids must be collected, transported and stored in a manner to exclude all contact with air. Due to the obvious safety concerns associated with potentially pyrophoric wastes, the sample size should be minimized to only that amount required to perform the appropriate test. For the pyrophoric solids or liquids test, a sample the size of 10 g or 35 mL is sufficient. For the self-heating test, a sample size of 50 - 100 g may be sufficient.

8.2 Sample preservation

No chemical sample preservation is required for this test.

8.3 Sample storage

The sample must be collected, transported, and stored in a manner to exclude all contact with air. Materials known or suspected to have self-heating properties should be stored in a cool place away from heat sources.

8.4 Holding time

Due to the lack of an established holding time for this parameter, all samples should be analyzed as soon as possible.

9.0 QUALITY CONTROL

9.1 Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Test Methods A and B are conducted using multiple trials, unless a positive result is obtained in one of the trials, no further testing is required. A positive test is indicated by smoke, fire, incandescence, or other observable signs of spontaneous combustion. Ignition in any of the trials is considered a positive result, even when the results of the other trials are negative. These tests are operationally defined and the results of the tests depend on the observations and interpretations made by the analyst. No reference standards are currently available for these tests. No other quality control procedures are required for this procedure.

10.0 CALIBRATION AND STANDARDIZATION

This procedure does not require any external calibration or standardization techniques. Currently, there is no reference material available for this test. EPA does not believe that the use of such a material would enhance the performance of the method because a positive result for Test Methods A or B is readily demonstrated by smoke, flame, charring of the paper filter, and for Test Method C is readily demonstrated by spontaneous ignition or a significant increase in sample temperature. The analyst should be consistent with ignition recognition and record any visible signs of combustion including smoke, heat, flame, or incandescence.

11.0 PROCEDURE

WARNING: This test may need to be conducted in a specially designed facility if proper surveillance and safety precautions are not available in the laboratory. Analysts are encouraged to use a flammability screening test such as ASTM 4982 prior to this procedure to test for highly flammable wastes.

11.1 Test Method A -- Pyrophoric solids

Test Method A is performed to determine if a solid ignites within five minutes of coming in contact with air at ambient room temperature. All tests must be conducted in a fume hood to prevent the escape of toxic fumes or flames into working areas.

11.1.1 The waste should be tested in its "as received" form and should not be dried. If the waste was stored at ≤ 6 °C, it should be allowed to equilibrate to ambient temperature. Under no circumstances should the sample be opened and allowed to come into contact with air prior to conducting the test.

11.1.2 Position a ceramic tile (Sec. 6.3) in a fume hood about 20 cm (approximately 8 in) from the front of the hood in an area of laminar airflow. Airflow across the test area should be minimal, but sufficient to prevent the escape of fumes or smoke into working areas.

11.1.3 From the surface of the ceramic tile, measure a vertical distance equal to one meter above the tile surface. Attach a reference mark on the side of the hood or use some other means to identify this height.

11.1.4 Using a metal spatula or other suitable device, aliquot a 1 to 2 g sample of solid and quickly pour the sample from the height of one meter onto the surface of the ceramic tile.

11.1.5 Observe whether the test sample ignites during dropping or within 5 min of settling. Begin timing as soon as the test sample has settled onto the ceramic tile surface.

11.1.6 Ignition is demonstrated by the presence of observable smoke, flame, or incandescence. If spontaneous combustion is observed within the 5-min test period, no further testing is required. If no observable signs of combustion are initially observed, repeat the test five additional times with a fresh sample each time or until a positive result is observed, whichever occurs first.

11.2 Test Method B -- Pyrophoric liquids, porcelain cup test

11.2.1 All tests must be conducted in a fume hood. Place a porcelain or metallic crucible (Sec. 6.4) into the center of a fume hood. Pour enough silica gel (Sec. 6.7) into the crucible to cover the bottom of the crucible to a depth of approximately 5 mm.

11.2.2 Using a pipet or other suitable measuring device (Sec. 6.8), quickly transfer approximately 5 mL of the liquid waste into the bottom of the crucible containing the bed of silica gel.

11.2.3 Begin timing and observe whether the test sample ignites within 5 min.

11.2.4 Ignition is demonstrated by the presence of observable smoke, flame, or incandescence. If spontaneous combustion is observed within the 5 min test period, no further testing is required. If no observable signs of combustion are observed, repeat the test five additional times with a fresh sample aliquot and silica gel for each test, or until a positive result is observed, whichever occurs first. If the results of all six trials are negative, proceed to the filter paper test for pyrophoric liquids in Sec. 11.3.

11.3 Test Method B -- Pyrophoric liquids, filter paper test

11.3.1 Position a glass beaker (Sec. 6.6) in a fume hood about 20 cm (approximately 8 inches) from the front of the hood in an area of laminar airflow. The glass beaker should be of sufficient diameter to support a circular Whatman No. 3 filter paper, such that both sides of the filter paper are exposed to air. To assist in the placement of the filter paper over the beaker opening, tape or other mechanical devices may be used as long as the tape or mechanical device does not come into contact with the test sample. Airflow across the test area should be minimal, but sufficient to prevent the escape of fumes or smoke into working areas.

11.3.2 Using a pipet or other suitable measuring device (Sec. 6.8), quickly aliquot approximately 0.5 mL of the liquid waste onto the center of the filter paper. Begin timing and observe whether ignition or charring occurs on the filter paper within a 5-min test period. Ignition is demonstrated by the presence of observable smoke, flame, or incandescence. If spontaneous combustion is observed within the 5-min test period, no further testing is required. If ignition or charring is not observed, repeat the test two additional times using a fresh filter paper and sample aliquot, unless a positive test result is obtained prior to the third and final trial.

11.4 Test Method C -- Self-heating substances

CAUTION: Test A must be run before Test C is conducted. If the waste is pyrophoric, then Test C must not be run. Also, this procedure is not suitable for liquid wastes.

11.4.1 Test Method C is performed to determine whether a solid waste exhibits self-heating properties when exposed to a temperature of 140 °C for a period of 24 hrs. This test is based on the Bowes-Cameron cage self-heating test method for carbon. The criterion for self-heating is based on the self-ignition temperature of charcoal, which is 50 °C for a sample cube of 27 m³ and 140 °C for a one-liter sample. If self-heating occurs using the 100-mm sample cube, then the waste is tested using a 25-mm sample cube to determine the waste classification.

11.4.2 Load the solid sample, in its "as received" form, into the 100-mm stainless steel cage. Particle size reduction may be employed to facilitate the loading of the cage when the waste is presented in large pieces. Fill the cage to the brim with the sample and drop the filled cage three times from a height of 1 to 2 inches. If the waste settles, add additional waste until the cage is filled to the brim.

11.4.3 Insert the loaded cage into the outer steel cage and suspend the assembly in the center of the oven by means of a wire or similar device. No part of the assembly should contact the outer walls or bottom. Insert one of the Chromel-Alumel thermocouples (Sec. 6.12) through the outer cage and through the open top of the inner cage. This thermocouple should be positioned within the center of the waste to be tested (approximately 50 mm into the waste). Position another Chromel-Alumel thermocouple between the sample and the oven wall (the thermocouple should be suspended in mid-air and should not contact the oven surface or the loaded cage).

11.4.4 Turn on the oven and adjust the temperature controller to achieve an oven temperature of 140 ± 2 °C. Maintain this temperature for 24 hrs. During this period, both the oven temperature and the temperature of the sample must be monitored closely. Preferably, both temperatures should be continuously recorded. Electronic thermometers are commercially available that can continuously monitor and record the maximum oven and sample temperatures (refer to Sec. 6.13).

11.4.5 Observe whether the sample temperature exceeds 200 °C during the 24 ± 2 hr test period. Continuous temperature monitoring is required because the sample temperature may exceed 200 °C at any time during the 24-hour test period.

NOTE: If a positive result is obtained (i.e., the sample temperature exceeds 200 °C) prior to the conclusion of the 24 ± 2 hr test period, the experiment may be terminated by turning off the oven. The sample temperature may continue to increase even after the oven is turned off.

WARNING: Once the experiment is started, the oven should not be opened, especially if the sample temperature is observed to rise above 140 °C. Opening the oven when the sample is at an elevated temperature could increase the potential for a fire. Therefore, the oven and sample temperature should be allowed to return to ambient temperature before the oven is opened.

11.4.6 If, at the end of the 24-hr test period, the temperature of the test sample did not exceed 200 °C, then no additional testing is required. The results of the test are negative for self-heating. If the temperature of the test sample exceeds 200 °C within the 24-hour test period, then test a second sample aliquot using a 25-mm stainless steel cube to determine the self-heating classification of the waste.

11.4.7 Load the waste in the 25-mm cage as described in Sec. 11.4.2. Suspend the loaded sample into the oven and heat to 140 ± 2 °C for 24 hrs, as described in Secs. 11.4.2 through 11.4.4.

11.4.8 Observe whether the sample temperature exceeds 200 °C during the 24-hr test period. Continuous temperature monitoring is required because the sample temperature may exceed 200 °C at any time during the 24-hr test period. If the sample temperature exceeds 200 °C prior to the end of the 24-hr test period, then the experiment may be terminated by turning the oven off.

11.4.9 If at the end of the 24-hr test period the temperature of the test sample did not exceed 200 °C, then no additional testing is required. If the temperature of the test sample exceeds 200 °C within the 24-hr test period, then the waste has self-heating properties. Refer to Sec. 12.0 for appropriate waste classifications.

NOTE: If the waste is suspected to have strong self-heating properties, the 25-mm cube test may be performed in lieu of the 100-mm cube test. If the results of this test are positive, then the test using the 100-mm cage need not be performed because the waste has self-heating properties. If the results of the 25-mm cube test are negative, then the waste must be evaluated by the 100-mm cube test in order to make a proper self-heating determination.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Method of assessing results for pyrophoric solids

If the sample ignites during any of the tests, then the substance should be considered pyrophoric and classified as a waste with pyrophoric properties.

12.2 Method of assessing results for pyrophoric liquids

If the liquid ignites when mixed with an inert carrier, or if it ignites or chars the filter paper, then it should be classified as a liquid waste having pyrophoric properties.

12.3 Method of assessing results for self-heating wastes

12.3.1 A waste should be classified under DOT Packing Group II (refer to Sec. 1.2) if the sample tests positive for both the 100-mm and 25-mm cubes (or tests positive when tested using the 25-mm cube alone).

12.3.2 A waste should be classified under DOT Packing Group III (refer to Sec. 1.2) if the sample tests positive for the 100-mm cube, but negative for the 25-mm cube.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 Pyrophoric solids

Test Method A was applied to various mixtures of manganese ethylene bis/zinc salt complexes. Table 1 shows the results, which were obtained from Ref. 3. These data are provided for guidance purposes only.

13.3 Pyrophoric liquids

Test Method B was applied to various diethyl aluminum chloride/isopentane mixtures and to various triethyl aluminum/heptane mixtures. Table 2 shows the results, which were obtained from Ref. 3. These data are provided for guidance purposes only.

13.4 Self-heating wastes

13.4.1 Test Method C was applied to granular cobalt/molybdenum, nickel catalysts, nickel/vanadium catalysts, nickel/molybdenum catalysts, and manganese ethylene bis/zinc mixtures. Table 3 shows the results, which were obtained from Ref. 3. These data are provided for guidance purposes only.

13.4.2 In a study conducted by the EPA National Enforcement Investigations Center (NEIC), three samples of used Raney[®] nickel catalyst were evaluated for self-heating using Test Method C. Raney[®] nickel is widely used as a hydrogenation catalyst and also as a scavenger for undesirable chemical species otherwise left in chemical products. Table 4 shows the results of this study, which were obtained from Ref. 4. These data are provided for guidance purposes only.

13.4.3 In a separate study conducted by EPA, the performance of Test Method C was evaluated by testing simulated wastes consisting of mixtures of charcoal, fish meal, and cotton with linseed oil and tung oil. Fish meal, charcoal, and linseed oil are identified in the 17th Edition of the *National Fire Protection Association Handbook (NFPA)* as materials that have a high tendency of spontaneous heating. Tung oil is classified as having moderate self-heating tendencies. Cotton is not listed in the NFPA handbook and has a low self-heating tendency. A real-world waste consisting of white phosphorus-contaminated soil was also evaluated. Table 5 summarizes the results of this investigation, which were obtained from Ref. 5. These data are provided for guidance purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, <http://www.acs.org>.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. 49 CFR Part 173, Appendix E, "Guidelines for the Classification and Packaging Group Assignment of Class 4 Materials."
2. United Nations Classification Scheme for the Classification of Substances Liable to Spontaneous Combustion, Division 4.2, Sec. 32.3, ST/SG/AC.10/C.3/R.370.
3. United Nations Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, ST/SG/AC.10/11/Rev2, 1995, Classification Procedures, Test Methods, and Criteria Related to Class 4, Substances Liable to Spontaneous Combustion, Division 4.2, Sec. 33.3.
4. U. S. EPA, National Enforcement Investigations Center, "Analytical Results Raney® Nickel Wastes," April 13, 1998.
5. Science Applications International Corporation, "Evaluation of Method 1050," submitted to the U.S. EPA, Office of Solid Waste, June 1998.
6. 62 FR 24690, May 17, 1997, Department of Transportation, Hazardous Materials: Harmonization With the United Nations Recommendations, International Maritime Dangerous Goods Code, and International Civil Aviation Organization's Technical Instructions; Final Rule.

17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1
EXAMPLE RESULTS FOR PYROPHORIC SOLIDS

Substance	Time to Ignition	Test Result
Manganese ethylene bis(dithiocarbamate) complex with zinc salt 88% (Mancozeb)	No ignition within 5 minutes	Not Pyrophoric
Manganese ethylene bis(dithiocarbamate) complex with zinc salt 80% (Mancozeb)	No ignition within 5 minutes	Not Pyrophoric
Manganese ethylene bis(dithiocarbamate) complex with zinc salt 75% (Mancozeb)	No ignition within 5 minutes	Not Pyrophoric

Data taken from Ref. 3.

These data are provided for guidance purposes only.

TABLE 2
EXAMPLE TEST RESULTS FOR PYROPHORIC LIQUIDS

Substance	Effect on Exposure to Air	Effect on Filter Paper	Test Result
Diethyl aluminum chloride/isopentane (10/90)	No ignition	No charring	Not pyrophoric
Diethyl aluminum chloride/isopentane (15/85)	No ignition	Charring	Pyrophoric
Diethyl aluminum chloride/isopentane (95/5)	No ignition	Charring	Pyrophoric
Triethyl aluminum/heptane (10/90)	No ignition	No Charring	Not Pyrophoric
Triethyl aluminum/heptane (15/85)	No ignition	Charring	Pyrophoric
Triethyl aluminum/heptane (95/5)	No ignition	Charring	Pyrophoric

These data are provided for guidance purposes only.
Data taken from Ref. 3.

TABLE 3
EXAMPLE TEST RESULTS FOR SELF-HEATING SOLIDS

Substance	Cube Size (mm)	Maximum Sample Temperature (°C)	Result
Cobalt/molybdenum catalyst granules	100	> 200	Positive
	25	181	Negative
Manganese ethylene bis-80% (Maneb)	25	> 200	Positive
Manganese ethylene bis complex with zinc salt (75%) (Mancozeb)	25	> 200	Positive
Nickel catalyst granules with 70% hydrogenated oil	100	140	Negative
Nickel catalyst granules with 50% white oil	100	> 200	Positive
	25	140	Negative
Nickel/molybdenum catalyst granules (spent)	100	> 200	Positive
	25	150	Negative
Nickel/molybdenum catalyst granules (passivated)	100	161	Negative
Nickel/vanadium catalyst granules	25	> 200	Positive

These data are provided for guidance purposes only.
Data taken from Ref. 3.

TABLE 4
EXAMPLE SELF-HEATING TEST RESULTS FOR RANEY® NICKEL WASTES

Sample	Cube Size (mm)	Maximum Sample Temperature (°C)	Results
Sample A (from hydrogenation process)	100	670	Positive
	25	691	Positive
	25	677	Positive
Sample B (from hydrogenation process)	100	630	Positive
	25	620	Positive
Sample C (from tetrahop process)	100	144	Negative

These data are provided for guidance purposes only.
Date taken from Ref. 4.

TABLE 5

EXAMPLE RESULTS FOR SELF-HEATING TESTS OF SIMULATED WASTES

Simulated Waste	Maximum Observed Temperature (°C)		Results (100-mm/25-mm)
	100-mm Cube	25-mm Cube	
Charcoal (alone)	143	NA	negative
9:1 Charcoal:tung oil	144	NA	negative
3:1 Charcoal:tung oil	152	NA	negative
9:1 Charcoal:linseed oil ²	359	168	positive/negative
3:1 Charcoal:linseed oil ²	339	227	positive/positive
Pellet Fish Meal (alone) ²	278	141	positive/negative
9:1 Pellet Fish Meal:tung oil ²	241	139	positive/negative
3:1 Powdered Fish Meal:tung oil	166	NA	negative
9:1 Powdered Fish Meal:linseed oil	180	NA	negative
3:1 Powdered Fish Meal:linseed oil ²	215	148	positive/negative
9:1 Cotton:tung oil	140	NA	negative
3:1 Cotton:tung oil	143	NA	negative
9:1 Cotton:linseed oil	180	NA	negative
3:1 Cotton:linseed oil ²	212	137	positive/negative
White phosphorus-contaminated sediment	146	151	negative

¹Experiments with the 25-mm cube were conducted only if the maximum temperature observed for the 100-mm cube exceeded 200 °C (except for white phosphorus).

²100-mm experiments terminated prior to the conclusion of the 24-hour test period because the sample temperature exceeded 200 °C.

These data are provided for guidance purposes only.
Data taken from Ref. 5.

TABLE 6

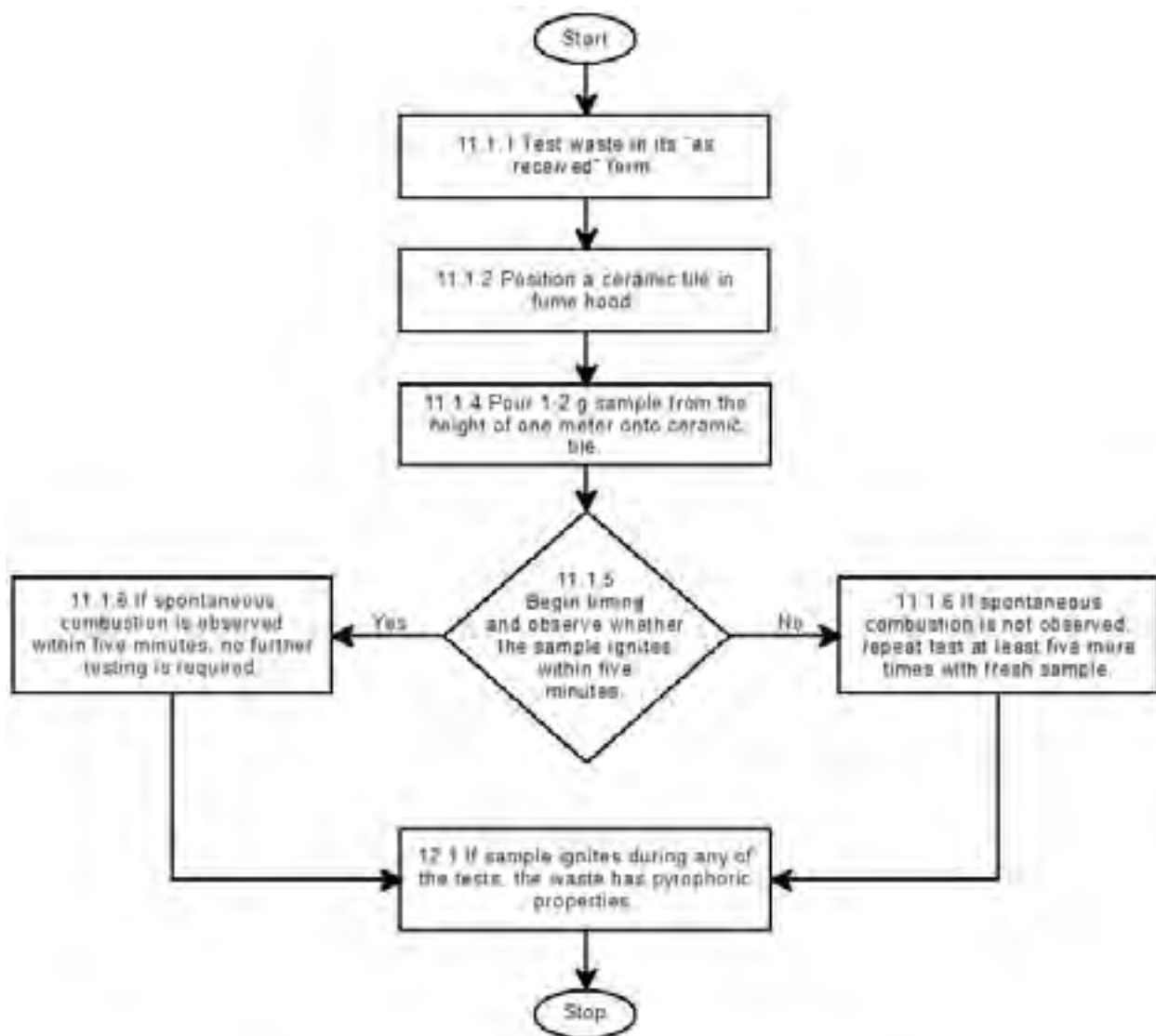
DOT CLASSIFICATION OF SELF-HEATING SOLID WASTES

Results of Self-Heating Test	DOT Packing Group
Negative for the 100-mm cube test	Not a Self-Heating Waste
Positive for the 100-mm, but negative for the 25-mm cube test	III
Positive for both the 100-mm and 25-mm cube tests or positive for the 25-mm cube test, if tested alone	II

METHOD 1050

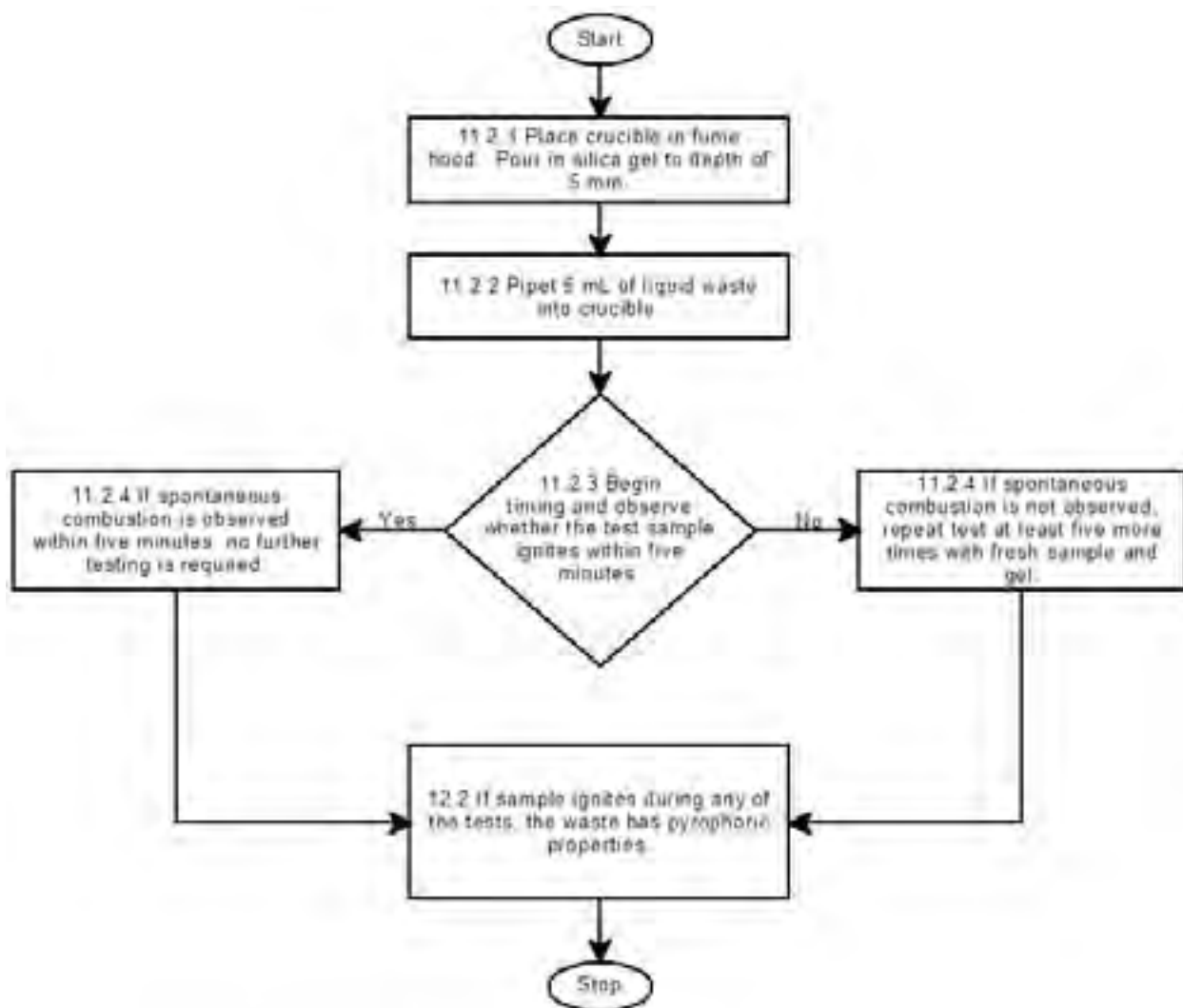
TEST METHODS TO DETERMINE SUBSTANCES LIKELY
TO SPONTANEOUSLY COMBUST

TEST METHOD A -- PYROPHORIC SOLIDS



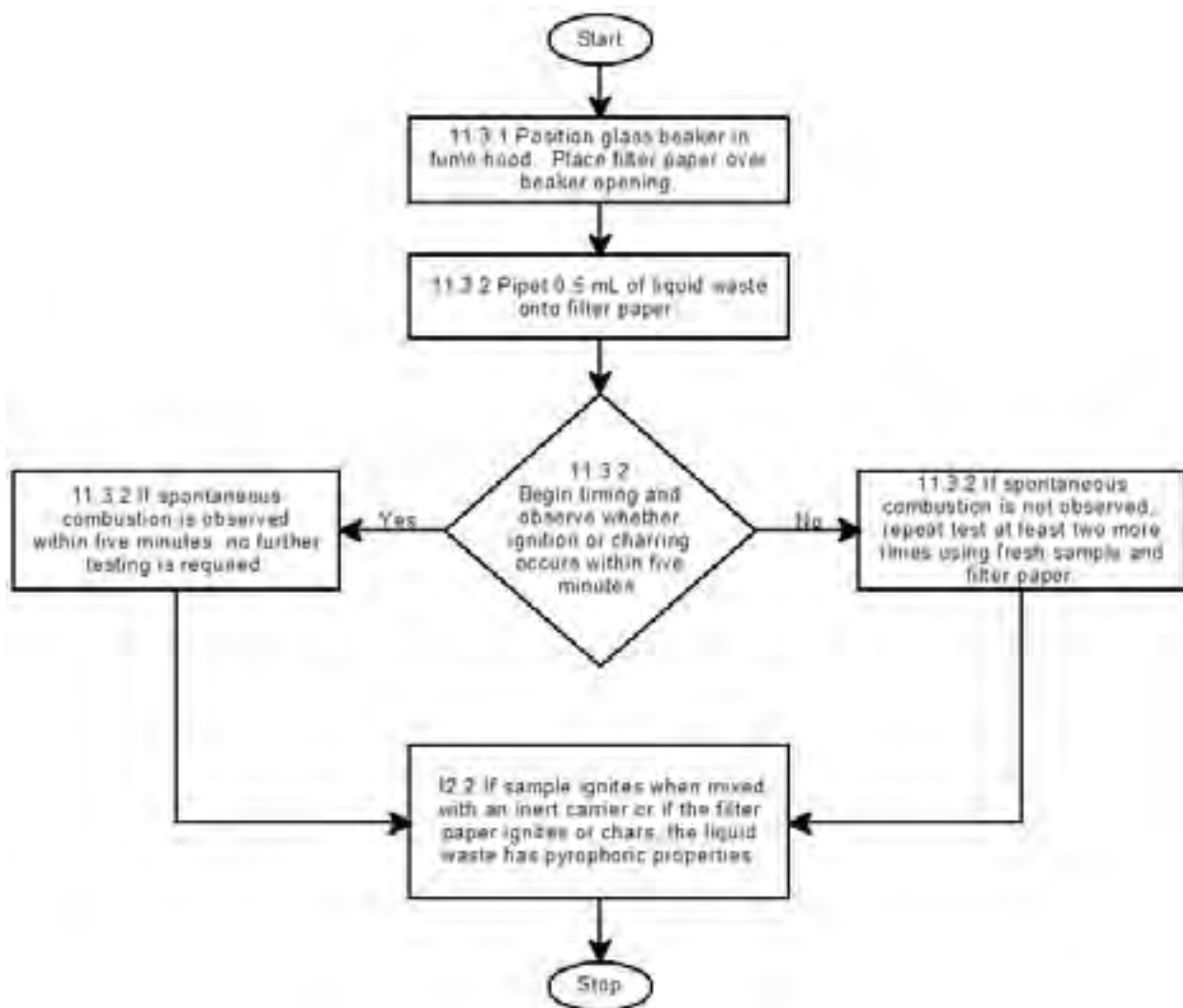
METHOD 1050
(Continued)

TEST METHOD B -- PYROPHORIC LIQUIDS, PORCELAIN CUP TEST



METHOD 1050
(Continued)

TEST METHOD B -- PYROPHORIC LIQUIDS, FILTER PAPER TEST



METHOD 1050
(Continued)

TEST METHOD C -- SELF-HEATING SUBSTANCES
(Not suitable for liquid wastes)

