# CHAPTER 7045
# MINNESOTA POLLUTION CONTROL AGENCY
# SOLID AND HAZARDOUS WASTE DIVISION
# HAZARDOUS WASTE RULES

**CLASSIFICATION, EVALUATION, AND CERTIFICATION OF WASTE**

**GENERATION OF HAZARDOUS WASTE**

**LOCATION, OPERATION, AND CLOSURE OF A HAZARDOUS WASTE FACILITY**

**TRANSPORTATION OF HAZARDOUS WASTE**

**HAZARDOUS WASTE FACILITY PERMIT PROGRAM**

**FACILITY PERMIT APPLICATIONS**

**HAZARDOUS WASTE SHIPPING PAPERS**

**COUNTY REGULATION OF HAZARDOUS WASTE MANAGEMENT**

**SPILLS AND LEAKS**

## 7045.0010 SCOPE.

The provisions of this chapter govern the identification, classification, storage, labeling, transportation, treatment, processing, and disposal of hazardous waste by any person and the issuance of permits for the construction, operation, and closure of a hazardous waste facility for the protection of the environment.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0020 DEFINITIONS.

Subpart 1. **Scope.** As used in these hazardous waste rules the following words shall have the meanings defined herein.

Subp. 2. **Agency.** "Agency" means the Minnesota Pollution Control Agency.

Subp. 3. **Chemical composition.** "Chemical composition" means any of the following:

A. a standard chemical nomenclature such as those adopted by the International Union of Pure and Applied Chemistry or the Chemical Abstracts' Service;

B. common chemical name when it is documented to the director that the number of isomers, related compounds of similar chemical structure and property, etc., make chemical analysis or delineation impractical;

C. common chemical name of a mixture of components with similar properties, but not including a trade name.

Subp. 4. **Components of the waste.** "Components of the waste" means chemical elements, chemical compounds, and ions that constitute the waste, and those that may form during the management of the waste from chemical reactions among the components or as biological products of microbial action.

Subp. 5. **Container.** "Container" means any packaging or containment unit, excluding portable tanks and storage tanks.

Subp. 6. **Corrosive material.** "Corrosive material" means a material that has any one of the following properties:

A. a pH that is greater than 12 or less than three for an aqueous material;

B. the ability to cause a visible destruction or irreversible alteration of skin tissues at the site of contact following an exposure period of four hours or less when tested by the technique described in Code of Federal Regulations, title 16, section 1500.41 (1977);

C. a corrosion rate of 0.250 inch per year or more on Society of Automotive Engineers' 1020 Steel when tested in accordance with the minimum requirements described in the National Association of Corrosive Engineers' Standard TM-01-69, at a test temperature of 130 degrees Fahrenheit (54.4 degrees Centigrade).

Subp. 7. **Demolition debris.** "Demolition debris" means concrete, blacktop, bricks, stone facing, concrete block, stucco, glass, structural metal, and wood from demolished structures.

Subp. 8. **Director.** "Director" means the executive director of the Minnesota Pollution Control Agency.

Subp. 9. **Explosive material.** "Explosive material" means a material that has the property either to evolve large volumes of gas that are dissipated in a shock wave or to heat the surrounding air so as to cause a high pressure gas that is dissipated in a shock wave. Explosive materials include, but are not limited to, explosives as defined in Code of Federal Regulations, title 49, section 173.50 (1976) and compressed gases as defined in Code of Federal Regulations, title 49, section 173.300 (1976).

Subp. 10. **Facility operator.** "Facility operator" means any person who owns, leases, operates, controls, supervises, closes, or abandons a hazardous waste facility.

Subp. 11. **Flammable material.** "Flammable material" means any material that:

A. has a flash point below 200 degrees Fahrenheit (93.3 degrees Centigrade), except the following:

(1) a material comprised of miscible components having one or more components with a flash point of 200 degrees Fahrenheit (93.3 degrees Centigrade), or higher, that make up at least 99 percent of the total volume of the mixture;

(2) a material that has a flash point greater than 100 degrees Fahrenheit (37.8 degrees Centigrade) and that when heated to 200 degrees Fahrenheit (93.3 degrees Centigrade) will not support combustion beyond the flash;

(3) an explosive material; or

B. may ignite without application of a flame or spark including, but not limited to, nitro cellulose, certain metal hydrides, alkali metals, some oily fabrics, some processed meals, and acidic anhydrides;

C. is capable of spontaneously producing temperatures in excess of 200 degrees Fahrenheit (93.3 degrees Centigrade).

Subp. 12. **Flash point.** "Flash point" means the minimum temperature at which a material gives off vapor within a test vessel in sufficient concentration to form an ignitable mixture with air near the surface of the material.

Subp. 13. **Floodplain.** "Floodplain" has the meaning as defined in Minnesota Statutes, section 104.02, subdivision 3.

Subp. 14. **Garbage.** "Garbage" means discarded material resulting from the handling, processing, storage, preparation, serving, and consumption of food.

Subp. 15. **Generator.** "Generator" means a person who produces a hazardous waste within the state of Minnesota or a person who produces a hazardous waste outside the state of Minnesota that is transported to a hazardous waste facility within the state of Minnesota.

Subp. 16. **Groundwater.** "Groundwater" means the water contained below the surface of the earth in the saturated zone including, without limitation, all waters whether under confined, unconfined, or perched conditions, in near surface unconsolidated sediment or regolith, or in rock formations deeper underground. The term groundwater shall be synonymous with underground water.

Subp. 17. **Hazardous property.** "Hazardous property" means any property of a waste that requires the waste to be classified as a hazardous waste.

Subp. 18. **Hazardous waste.** "Hazardous waste" has the meaning as defined in Minnesota Statutes, section 116.06, subdivision 13.

Subp. 19. **Hazardous waste facility.** "Hazardous waste facility" means real or personal property that is used or is constructed to be used for the management of hazardous waste including, but not limited to the following:

A. hazardous waste containerized storage facility: a hazardous waste facility that is designed or operated for the on-site storage of hazardous waste that is in containers, portable tanks, or storage tanks;

B. hazardous waste noncontainerized storage facility: a hazardous waste facility that is designed or operated for the storage of hazardous waste in lagoons, basins, ponds, vaults, or similar bulk storage other than containers or tanks;

C. hazardous waste transfer station: a hazardous waste facility that receives wastes from one or more generators and is designed or operated for the purpose of intermediate storage of wastes prior to transportation of the waste to another hazardous waste facility;

D. hazardous waste processing facility: a hazardous waste facility that is designed and operated to modify the chemical composition or chemical, physical, or biological properties of a hazardous waste by means such as incineration, reclamation, distillation, precipitation, or other similar processes;

E. hazardous waste land disposal facility: a hazardous waste facility that is designed or operated for the purpose of disposing of, or storing for a period greater than one year, hazardous waste in the subsurface of the land;

F. hazardous waste land treatment facility: a hazardous waste facility that is designed or operated for the purpose of utilizing the surface of the land as the medium by which biological, physical, or chemical processes can provide treatment of hazardous waste.

Subp. 20. **Hazardous waste management.** "Hazardous waste management" means the total system for the identification, storage, collection, and removal of hazardous waste from public or private property, the transportation of the waste to a hazardous waste facility, and the ultimate processing or disposal of the waste by approved methods in accordance with these regulations. Any reference to hazardous waste being managed shall refer to the foregoing.

Subp. 21. **Incompatible wastes.** "Incompatible wastes" means wastes that when in contact with each other pose a threat to human health and safety that does not exist when they are separate, including, but not limited to, wastes that pursuant to Code of Federal Regulations, title 49, section 177.848 (1976) cannot be stored or transported together.

Subp. 22. **Irritative material.** "Irritative material" means a noncorrosive material which has the property to cause a local reversible injury to a biological membrane at the site of contact as determined by either of the following:

A. practical experience with the waste where short term exposures have caused first degree burns and where long term exposure may cause second degree burns;

B. skin irritation of an empirical score of five or more as determined pursuant to Code of Federal Regulations, title 16, section 1500.41 (1977).

Subp. 23. **Leachate.** "Leachate" means a liquid that is released from, or percolated through, a waste as a result of conditions that arise during storage, land disposal, or land treatment.

Subp. 24. **Manifest.** "Manifest" means the shipping papers used in transporting hazardous waste.

Subp. 25. **Median lethal concentration ($LC_{50}$).** "Median lethal concentration ($LC_{50}$)" means the calculated concentration at which a material kills 50 percent of a group of test animals within a specified time:

A. aquatic $LC_{50}$: the $LC_{50}$ determined by a test in which the specified time is 96 hours, the test animals are at least ten fathead minnows, and the route of administration follows accepted static or flow through bioassay techniques;

B. inhalation $LC_{50}$: the $LC_{50}$ determined by a test in which the specified time is 14 days, the group of the test animals is at least ten white laboratory rats of 200 to 300 grams each, half of which are male and half of which are female, and the route of administration is continuous respiratory exposure for a period of one hour.

Subp. 26. **Median lethal dose ($LD_{50}$).** "Median lethal dose ($LD_{50}$)" means the calculated dose at which a material kills 50 percent of a group of test animals within a specified time:

A. oral $LD_{50}$: the $LD_{50}$ determined by a test in which the specified time is 14 days, the group of test animals is at least ten white laboratory rats of

200 to 300 grams each, half of which are male and half of which are female, and the route of administration is a single oral dose;

B. dermal $LD_{50}$: the $LD_{50}$ determined by a test in which the specified time is 14 days, the group of test animals is ten or more white rabbits, half of which are male and half of which are female, and the route of administration is a 24 hour exposure with continuous contact on bare skin.

Subp. 27. **On-site management.** "On-site management" means the handling of a hazardous waste after generation without transporting such hazardous waste by public thoroughfare.

Subp. 28. **Open burning.** "Open burning" means the burning of any matter whereby the resultant combustion products are emitted directly to the atmosphere without passing through an adequate stack, duct, or chimney.

Subp. 29. **Oxidative material.** "Oxidative material" means any material with the property to readily supply oxygen to a reaction in the absence of air. Oxidative materials include, but are not limited to, oxides, organic and inorganic peroxides, permanganates, perrhenates, chlorates, perchlorates, persulfates, nitricacid, organic and inorganic nitrates, iodates, periodates, bromates, perselenates, perbromates, chromates, dichromates, ozone, and perborates. Bromine, chlorine, fluorine, and iodine react similarly to oxygen under some conditions and are therefore also oxidative materials.

Subp. 30. **Person.** "Person" has the meaning as defined in Minnesota Statutes, section 116.06, subdivision 8.

Subp. 31. **Pesticide.** "Pesticide" means any substance or mixture of substances intended for preventing, destroying, repelling, or mitigating any pest, and any substance or mixture of substances intended for use as a plant regulator, defoliant, or desicant.

Subp. 32. **Petroleum waste.** "Petroleum waste" means an oily waste generated by petroleum storage, petroleum refining, and petroleum refinery products storage.

Subp. 33. **Resource recovery.** "Resource recovery" has the meaning as defined in Minnesota Statutes, section 473.121, subdivision 31c.

Subp. 34. **Routine waste management.** "Routine waste management" means the total system for the handling of a waste by one of the following methods:

A. storage, collection, and removal of waste from public or private property, its transportation to intermediate or final disposal facilities, and its ultimate disposal at a sanitary landfill permitted by the agency;

B. discharge into a sewer system and subsequent treatment at a wastewater treatment works operated pursuant to a National Pollutant Discharge Elimination System Permit or State Disposal Permit;

C. discharge into the atmosphere as an air contaminant or emission emitted pursuant to an Emission Facility Operating Permit;

D. a wastewater discharge pursuant to a National Pollutant Discharge Elimination System Permit or a State Disposal System Permit.

Subp. 35. **Rubbish.** "Rubbish" means discarded paper, cardboard, scrap metal, yard clippings, crop residues, brush, wood, glass, bedding, crockery, or litter.

Subp. 36. **Sanitary landfill.** "Sanitary landfill" means a land disposal site employing an engineered method of disposing of solid waste on land in a manner that minimizes environmental hazards by spreading the solid waste in thin layers, compacting the solid waste to the smallest practical volume, and applying cover material at the end of each operating day, or at intervals as may be required by the agency.

Subp. 37. **Saturated zone.** "Saturated zone" means that part of the earth's crust in which all the voids, large and small, are ideally filled with water under pressure greater than atmospheric.

Subp. 38. **Sewage.** "Sewage" has the meaning as defined in Minnesota Statutes, section 115.01, subdivision 2.

Subp. 39. **Sewer system.** "Sewer system" has the meaning as defined in Minnesota Statutes, section 115.01, subdivision 6.

Subp. 40. **Shoreland.** "Shoreland" has the meaning as defined in Minnesota Statutes, section 105.485, subdivision 2.

Subp. 41. **Tank.** "Tank" means any packaging or containment unit having a capacity of 100 gallons or greater that is used to confine and hold a material. Tanks that are anchored, fixed, or attached to one location are storage tanks, and those that are not are portable tanks.

Subp. 42. **Toxic material.** "Toxic material" means a material with any one of the following properties:

A. an oral $LD_{50}$ less than 500 milligrams of material per kilogram of body weight of test animal;

B. a dermal $LD_{50}$ less than 1000 milligrams of material per kilogram of body weight of test animal;

C. an inhalation $LC_{50}$ (when the material or a component is in a form that may be inhaled) less than:

(1) 2,000 milligrams of material as dust or mist per cubic meter of air; or

(2) 1,000 parts per million of material as gas or vapor;

D. an aquatic $LC_{50}$ less than 100 milligrams of material per liter of water.

Subp. 43. **Waste.** "Waste" means any discarded material including, but not limited to, solids, semisolids, sludges, liquids, gases, and their vapors, mists, or dusts.

Subp. 44. **Waters of the state.** "Waters of the state" has the meaning as defined in Minnesota Statutes, section 115.01, subdivision 9.

Subp. 45. **Water table.** "Water table" means the surface of the ground water at which the pressure is atmospheric. Generally this is the top of the saturated zone.

Subp. 46. **Wetland.** "Wetland" means a natural marsh where water stands near, at or above the soil surface during a significant portion of most years, and which is eligible for classification as an inland fresh water wetland type 3, 4, or 5 under United States Department of Interior classifications.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0030 ABBREVIATIONS.

The abbreviations used in these hazardous waste rules have the following meanings:

A. ASTM: American Society for Testing and Materials;

B. C.F.R.: Code of Federal Regulations;

C. EPA: United States Environmental Protection Agency;

D. $LC_{50}$: median lethal concentration;

E. $LD_{50}$: median lethal dose;

F. NPDES: National Pollutant Discharge Elimination System.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0040 INCORPORATIONS BY REFERENCE.

The following are contained in the indicated appendices at the end of this rule and are hereby incorporated and made a part of this chapter and shall apply as indicated within this chapter:

A. ASTM D3243-76 (appendix A) in part 7045.1140;

B. ASTM D56-70 (appendix B) in part 7045.1150;

C. ASTM D3278-73 (appendix C) in part 7045.1160;

D. ASTM D93-73 (appendix D) in part 7045.1170;

E. ASTM D2487-69 (appendix E) in part 7045.1180;

F. ASTM D2488-69 (appendix F) in part 7045.1190;

G. National Association of Corrosion Engineers' Standard TM-01-69 (appendix G) in part 7045.1200;

H. Code of Federal Regulations, title 10, section 20.301 (1977) (appendix H) in part 7045.1210;

I. 16 Code of Federal Regulations, title 16, section 1500.41 (1977) (appendix I) in part 7045.1220;

J. Code of Federal Regulations, title 49, sections 173.50, 173.300, 177.824, and 177.848 (1976) (appendix J) in part 7045.1230;

K. United States Department of Interior inland fresh water wetland types 3, 4, and 5 (appendix K) in part 7045.1240;

L. EP Toxicity Text (appendix L) in part 7045.1250;

M. Code of Federal Regulations, title 49, sections 172.202 and 172.203 (1979) (appendix M) in part 7045.1260.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0050 SEVERABILITY.

If any provision of these rules or the application thereof to any person or circumstance is held to be invalid, such invalidity shall not affect other provisions of these rules that can be given effect without the invalid provision or application. To this end, the provision of all rules and the various applications thereof are declared to be severable.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0060 VARIANCES.

Any person may apply for a variance from any requirement of these hazardous waste rules. Such variance shall be applied for and acted upon by the agency in accordance with Minnesota Statutes, section 116.07, subdivision 5 and other applicable statutes and rules.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0070 OTHER STANDARDS.

Nothing in these hazardous waste rules shall relieve any person from any obligations or duties imposed by any other laws, statutes, rules, standards, or ordinances of the federal, state, or local governments or any agency thereof now in effect or which become effective in the future. In the event these hazardous waste rules conflict with any such laws, statutes, rules, standards, or ordinances, the more stringent shall apply. Nothing in this chapter shall be construed to require any person to comply with any portion of this chapter if that portion should at any time be preempted by federal law.

**Statutory Authority:** *MS s 116.07 subd 4*

## CLASSIFICATION, EVALUATION, AND CERTIFICATION OF WASTE

### 7045.0100 SCOPE.

Parts 7045.0100 to 7045.0170 establishes the criteria for determining whether a waste is a hazardous waste.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0110 HAZARDOUS WASTES.

Subpart 1. **List 1.** A waste that contains a component specified in list 1 is a hazardous waste if the concentration of that component in the waste exceeds the concentration listed.

| Component | Concentration (ppm) |
|---|---|
| 2-Acetylaminofluorene (2-AAF) | 1,000 |
| 4-Aminodiphenyl (4-ADP) | 100 |
| Arsenic and its Compounds | 500 |
| Benzene | 100 |
| Benzidine | 100 |
| Beryllium and its Compounds | 20 |
| Cadmium and its Compounds | 500 |
| Carbon Tetrachloride | 100 |
| Chloroform | 100 |
| bis-(Chloromethyl) ether (BCME) | 100 |
| Chloromethyl methyl ether (CMME) | 100 |
| Chromium and its Compounds (VI) | 1,000 |
| 3,3-Dichlorobenzidine (DCB) | 1,000 |
| 4-Dimethylaminoazobenzene (DAB) | 1,000 |
| Ethyleneimine (EI) | 1,000 |
| Lead and its Compounds | 600 |
| 4,4-Methylene-bis-2-Chloroaniline (MOCA) | 100 |
| α-Naphthylamine (1-NA) | 1,000 |
| β-Naphthylamine (2-NA | 100 |
| Nickel and its Compounds | 10,000 |
| 4-Nitrobiphenyl (4-NBP) | 100 |
| n-Nitrosodimethylamine (DMN) | 1,000 |
| Polychlorinated biphenyl (PCB) | 500 |
| β-Propiolactone (BPL) | 1,000 |
| Vinyl Chloride (VCM) | 100 |

Subp. 2. **List 2.** A waste which contains a component specified in list 2 is a hazardous waste unless leachate from that waste does not contain that component at a concentration in excess of that specified in list 2.

| Component | Concentration (ppm) |
|---|---|
| Aldrin | 0.03 |
| Arsenic and its Compounds | 5.0 |
| Cadmium and its Compounds | 1.0 |
| Chlordane | 0.1 |
| Chromium and its Compounds | 5.0 |
| DDT | 0.01 |
| Endrin | 0.02 |
| Heptachlor | 0.01 |
| Lead and its Compounds | 3.0 |
| Mercury and its Compounds | 0.2 |
| Methoxychlor | 0.3 |
| Mirex | 0.01 |
| Polychlorinated biphenyl (PCB) | 0.01 |
| Toxaphene | 0.05 |

Subp. 3. **Other wastes.** Any of the following wastes:

A. an explosive material;

B. a flammable material;

C. an irritative material;

D. a corrosive material;

E. an oxidative material;

F. a toxic material;

G. used crankcase oil; and

H. petroleum waste.

Subp. 4. **Mixtures.** A waste that is a mixture of small amounts of unrelated chemicals such that the description of any sample or set of samples is not representative of the total waste. Examples are discarded chemicals from a chemistry laboratory, wastes from pilot plant chemical reactions, and discarded prescription drugs.

Subp. 5. **Waste not subject to routine waste management.** Any other waste that is not a hazardous waste under any provision in subparts 1 to 4, but that the agency determines pursuant to part 7045.0270, subpart 2 cannot be handled by routine waste management techniques because it poses a substantial present or potential hazard to human health or other living organisms.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0120 EXEMPT WASTES.

The following wastes may be stored, labeled, transported, treated, processed, and disposed of without complying with the requirements of this chapter:

A. Normal refuse from households.

B. Sewage.

C. Garbage, rubbish, and demolition debris from nonhousehold sources.

D. Asbestos in taconite wastes.

E. Septic tank sludge from households.

F. An air contaminant or emission emitted pursuant to an emission facility operating permit.

G. Any composite wastewater that is formed in a sewer system by the combination of two or more individual wastes that have been discharged into the sewer system. This exemption does not include any of the individual wastes which form the composite wastewater.

H. Wastes discharged pursuant to an NPDES permit or a state disposal system permit.

I. Municipal sewage sludge.

J. Radioactive waste that is produced pursuant to a permit issued under Code of Federal Regulations, title 10, parts 30, 40, and 70 (1976) and that is disposed of in compliance with Code of Federal Regulations, title 10, section 20.301 (1976).

K. A waste pesticide that is not in list 1 or list 2 or an unrinsed pesticide container that contained a pesticide that is not in list 1 or list 2.

L. The director shall exempt wastes resulting from spills from all or any provision of this chapter if the exemption is necessary to expedite the proper management of the spilled material and to prevent, abate, or control pollution.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0130 EVALUATION OF WASTES.

Any person who produces any waste within the state of Minnesota or any person who produces a waste outside the state of Minnesota that is managed within the state of Minnesota, and which waste is not an exempt waste under part 7045.0120, shall evaluate the waste to determine if it is hazardous. The person evaluating the waste shall compare the properties of the waste with the criteria for a hazardous waste in part 7045.0110 and determine whether the waste is hazardous, in accordance with the procedures set forth in these parts.

The person shall reevaluate the waste whenever the person has reason to believe that the composition of the waste is altered so that the results of the previous evaluation are no longer representative of the waste.

This evaluation shall be of the individual waste prior to any mingling or combining with other wastes. If wastes are subsequently mingled or combined, except for wastes that are mingled or combined in a sewer system, the generator shall also evaluate the waste resulting from the mingling or combining.

A person who produces two or more wastes that are similar or are from similar processes such that one waste is representative of the other wastes may use one evaluation for all such wastes.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0140 COMPARISON OF PROPERTIES.

Subpart 1. **General.** Any person evaluating a waste shall obtain such data as are necessary to determine whether the waste has any hazardous properties at any time during its management. The data may be obtained from the literature, from experience with the waste or from other sources, but if data are not available, then actual tests of a sample of the waste shall be conducted.

Subp. 2. **List 1 and list 2 components.** Whenever the person evaluating a waste knows or suspects that any of the components in list 1 or list 2 is in the waste, the person shall conduct a quantitative analysis to determine the concentration of each component in list 1 that is known or suspected to be in the waste and the EP Toxicity Test for each component in list 2 that is known or suspected to be in the waste with the following exceptions:

A. a waste known to contain a component of list 1 in excess of the concentration listed in list 1 need not be analyzed for that component;

B. a waste which is a hazardous waste because it contains a component of list 1 in excess of the concentration listed in list 1 need not be tested for that component in a list 2 test.

Subp. 3. **Other hazardous properties.** A person evaluating a waste shall determine whether the waste has any of the properties of an explosive material, a flammable material, an irritative material, a corrosive material, an oxidative material, and a toxic material. However, once the person determines that a waste has one of the properties of one of the classes of hazardous wastes described above, the person need not determine whether it has any of the other properties of the same class. For example, if a waste is a hazardous waste because of its oral $LD_{50}$, the dermal $LD_{50}$ need not be determined, but the waste must be evaluated to determine whether it is also explosive, flammable, irritative, corrosive, or oxidative.

Generators of wastes comprised of small amounts of unrelated chemicals such that a description of any sample or set of samples is not representative of the total waste, generators of petroleum waste and generators of used crankcase oil need not evaluate such wastes to determine whether they have any of the properties of an explosive material, a flammable material, an irritative material, a corrosive material, an oxidative material, and a toxic material. Such generators are also not required for such wastes to conduct a quantitative analysis to determine the concentration of each component in list 1 that is known or suspected to be in the waste nor a leachate test for each component in list 2 that is known or suspected to be in the waste.

Subp. 4. **Testing for flammable properties.** Whenever the flash point of a waste is to be determined, one of the following test procedures shall be used. The test chosen shall be appropriate for the characteristics of the waste that is tested.

A. Standard Method of Test for Flash Point by Tag Closed Tester (ASTM D56-70);

B. Standard Method of Test for Flash Point of Aviation Turbine Fuels by Setaflash Closed Tester (ASTM D3243-76);

C. Standard Methods of Test for Flash Point of Liquids by Setaflash Closed Tester (ASTM D3278-73);

D. Standard Method of Test for Flash Point by Pensky-Martens Closed Tester (ASTM D93-73) or alternate tests authorized in this standard.

For any waste containing components with different volatilities and flash points and having a flash point higher than 200 degrees Fahrenheit (93.3 degrees Centigrade) according to the test procedure employed, a second test shall be conducted on a sample of the liquid portion of the material that remains after evaporation in an open beaker (or similar container), under ambient pressure and temperature (20 to 25 degrees Centigrade) conditions, to 90 percent of original volume or for a period of four hours, whichever occurs first, with the lower flash point of the two tests being the flash point of the material.

Subp. 5. **Testing for toxic properties.** Any person who is determining whether a waste is a toxic material may elect to use the following modification to the $LC_{50}$ and $LD_{50}$ test procedures if the actual $LC_{50}$ or $LD_{50}$ is unknown:

A single dosage or exposure level equivalent to the maximum dosage or exposure level in which establishes that a material is a toxic material shall be administered to a test population of ten animals. The animals shall be the kind specified in the $LC_{50}$ and $LD_{50}$ test procedures. The animals are then observed for a period of 14 days or 96 hours whichever is applicable. If five or more of the test animals die, the waste shall be classified as a toxic material. If three or four of the test animals die, then either the waste shall be classified as a toxic material or additional dosage or exposure levels shall be tested and the actual $LD_{50}$ or $LC_{50}$ determined. If less than three of the ten test animals die, then the waste shall not be classified as a toxic material.

Subp. 6. **Testing for corrosive properties.** Any person testing for corrosive properties may elect to use the following procedure for a nonaqueous waste: the person may prepare an aqueous solution that contains equal parts of the waste and water and test it for pH. If the pH of the solution is greater than 12 or less than three, the person may classify the waste as a corrosive waste in lieu of evaluating the waste for the criteria indicated by part 7045.0020, subpart 6. If the person elects not to classify the nonaqueous waste as a corrosive waste or if the pH of the solution is not greater than 12 or less than three, additional evaluation to determine corrosivity must be performed.

Subp. 7. **Sample collection.** In the event the person evaluating the waste must conduct tests to determine the properties of the waste, the person shall collect a representative sample of the waste. In an attempt to collect a sample at the time when the properties being measured pose the greatest hazard, the person shall consider the following variations in the waste composition and their causes in collecting a sample for evaluation:

A. variations in the process by which the waste is produced;

B. variations in chemical composition and physical state;

C. any other variations indicated by past experience with the waste or similar wastes.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0150 RESULTS OF EVALUATION.

If the person evaluating the waste determines that the waste has any properties of a hazardous waste, the person shall file a disclosure with the agency and manage the waste in accordance with the requirements of this chapter.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0160 SUBMISSION OF EVALUATION RESULTS.

Subpart 1. **Information.** The director may request at any time that a person producing a waste submit the results of the evaluation of the waste to the agency. Upon such request by the director, setting forth the reasons therefore, the person shall submit the following information:

A. The type of waste and the source or process from which it was produced.

B. The chemical composition of the waste and the anticipated fluctuations in its chemical composition.

C. The concentration of each component in list 1 that is known or suspected to be in the waste and the concentration in the leachate of each component in list 2 that is known or suspected to be in the waste. If the component is not detected in the waste or in the leachate, the level of detectability of the testing method used shall be reported.

D. The results of the evaluation to determine whether the waste has any of the properties of an explosive material, a flammable material, an irritative material, a corrosive material, an oxidative material, and a toxic material and the source of the data or information relied upon.

E. In the event any tests were conducted to evaluate the waste, the person shall submit the following:

(1) the sampling procedure and the reasons for determining that the sample is representative of the waste;

(2) the results of all tests conducted;

(3) a discussion of the accuracy and precision of any tests conducted.

Subp. 2. **Failure to provide information.** If the person who is requested by the director to submit the results of an evaluation of a waste fails to submit the required information within 30 days after the request, the waste shall be managed as a hazardous waste and the person who produces the waste shall be considered a generator until the agency has determined whether the waste is hazardous or not.

Subp. 3. **Additional evaluation.** If the director determines that the results of the evaluation are not adequate to determine whether or not the waste is hazardous, the director may require the person to conduct an additional evaluation. The director shall notify the person in writing of such determination, the reasons therefor, and the additional tests that must be run or additional data that must be obtained. If the results of the additional evaluation are not reported to the agency within 30 days of the request, the waste shall be managed as a hazardous waste and the person who produces the waste shall be considered a generator until the agency has determined whether the waste is hazardous or not. The director may grant up to an additional 90 days where the person demonstrates such extension to be necessary.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0170 AGENCY DETERMINATION THAT A WASTE IS HAZARDOUS.

Subpart 1. **Right to enter.** The agency or any member, employee, or agent thereof, when authorized by it, may enter upon the property of the person who produces any waste to take samples of the waste and may conduct tests, analyses, and evaluations to determine whether the waste is a hazardous waste. The results of the tests, analyses, and evaluations shall be made available, upon request, to the person.

Subp. 2. **Recommendation and notice by director.** The director may recommend to the agency that a waste be classified as a hazardous waste because it has one or more of the properties of a hazardous waste or because the waste cannot be handled by routine waste management techniques because it poses a substantial or potential hazard to human health or other living organisms. The director shall notify the person producing the waste in writing of the recommendation and the person shall have at least 30 days to submit any additional material or written comments to the agency before the agency makes a determination. The agency shall notify the person in writing of its decision. The agency shall hold a contested case hearing pursuant to Minnesota Statutes, sections 14.57 et seq. upon request of the person producing the waste.

Subp. 3. **Management as hazardous waste.** In the event the director recommends that a waste be classified as a hazardous waste, the waste shall be managed as a hazardous waste and the person who produces the waste shall be considered a generator until the agency has determined whether the waste is hazardous or until six months after the date of the director's recommendation, whichever occurs first; provided, however, that the person shall not be required to obtain a hazardous waste facility permit for storage of the waste on-site during this time. Any such recommendation by the director shall be considered by the agency on an expeditious basis.

**Statutory Authority:** *MS s 116.07 subd 4*

## GENERATION OF HAZARDOUS WASTE

## 7045.0200 SCOPE.

Parts 7045.0200 to 7045.0290 prescribe the duties of a generator.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0210 PRODUCTION OF A HAZARDOUS WASTE.

No person shall produce a hazardous waste within the state of Minnesota or produce a hazardous waste outside the state of Minnesota that is transported to a hazardous waste facility within the state of Minnesota unless that person has adequate financial resources to insure that the hazardous waste is disposed of, treated, or processed at a hazardous waste facility permitted to manage such waste. Nothing in this provision is intended to restrict or enlarge or affect in any way, any liability the generator may have to correct the mismanagement of the hazardous waste or pay for damages or alleviate any pollution caused by the mismanagement of the hazardous waste.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0220 PREPARATION OF A DISCLOSURE.

Each generator shall prepare a disclosure for each hazardous waste that he produces or transports, except used crankcase oil that is collected by a transporter registered pursuant to part 7045.0560.

**Statutory Authority:** *MS s 116.07 subd 4*

**7045.0230 CONTENTS OF A DISCLOSURE.**

Subpart 1. **Information.** Each generator in his disclosure shall include the following information:

A. The type of waste and the source or process from which it is generated.

B. The chemical composition of the waste and the anticipated fluctuations in the chemical composition that will occur during normal operations.

C. The concentration of each component in list 1 that is known or suspected to be in the waste and the concentration in the leachate of each component in list 2 that is known or suspected to be in the waste. If the component is not detected in the waste or in the leachate, the level of detectability of the testing method used shall be reported.

D. The hazardous properties of the waste and the source of the data or information used to identify the hazardous properties.

E. In the event any tests were conducted to evaluate the waste, the following information shall be included in the disclosure:

(1) the sampling procedure and the reasons for determining that the sample is representative of the waste;

(2) the results of all tests conducted;

(3) a discussion of the accuracy and precision of any tests conducted.

F. A list of special handling procedures, labels, and safety equipment necessary for safe handling and storage of the hazardous waste.

G. The name, address, telephone numbers, and title of the individual at the generator's facility responsible for arranging for the management of the hazardous waste.

H. A copy of procedures for personnel to follow in the case of spills of the hazardous waste.

I. A summary of the following relating to the management of the hazardous waste for the year preceding the filing of the disclosure or for the period since the last disclosure was filed if that filing was more than one year ago:

(1) the amount of the hazardous waste produced;

(2) the names and identification numbers of the transporters utilized;

(3) the names of the hazardous waste facilities utilized, and, as applicable:

(a) the numbers of the hazardous waste facility permits issued by the agency for those facilities located in the state of Minnesota;

(b) the addresses of those facilities located outside the state of Minnesota;

(c) the name of the waste water treatment works to which a sewered hazardous waste was discharged;

(d) the NPDES or State Disposal Permit number for discharges to sewers other than a municipal sewer system;

(4) a summary taken from the shipping papers and other records of the generator of the amounts spilled, amounts recovered, and any resultant environmental or health damages from spills of the hazardous waste;

J. A prediction of the following relating to the management of the hazardous waste for the year immediately following the filing of the disclosure:

(1) the estimated amounts to be produced;

(2) the names and identification numbers of the transporters to be used;

(3) the frequency with which the hazardous waste is expected to be transported or discharged;

(4) the names of the hazardous waste facilities to be involved in the management of the hazardous waste and, as applicable: the numbers of the hazardous waste facility permits issued by the agency for those facilities located in the state of Minnesota and the addresses of those facilities located outside the state of Minnesota.

K. A list of all nonexempt wastes of the generator that have been determined by the generator to be nonhazardous wastes. The list shall include the type of waste and the sources or process from which the waste was produced. Examples of the information required are salt solution from water softening, and wash water from potato processing.

L. Any other information that the generator deems important.

Subp. 2. **Unrelated chemicals.** Generators of wastes that are comprised of small amounts of unrelated chemicals such that a description of any sample or set of samples is not representative of the total waste, generators of petroleum waste, and generators of used crankcase oil are not required to include in the disclosures for those wastes the items listed in subpart 1, items B, C, D, and E, but these generators shall identify those components in list 1 or list 2 in part 7045.0110 that the generator knows or suspects are in the waste.

Subp. 3. **False statements prohibited.** No person shall make a false statement in a disclosure. The disclosure shall be submitted under oath.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0240 SUBMISSION OF A DISCLOSURE TO THE AGENCY.

Subpart 1. **Existing hazardous waste.** Each generator who is producing a hazardous waste in the state of Minnesota or who is producing a hazardous waste outside the state of Minnesota that is being transported to a hazardous waste facility within the state of Minnesota on the day these hazardous waste rules take effect shall submit a disclosure to the agency within one year after the effective date of these rules. A generator who has produced a hazardous waste in the past and who anticipates producing that hazardous waste in the future may elect to file a disclosure on that hazardous waste under this provision. In such event, the generator shall not be required to file a disclosure under subpart 2 for that waste.

Subp. 2. **New hazardous wastes.** New hazardous wastes.

A. Any generator who produces a hazardous waste in the state of Minnesota that is not being produced on the day these hazardous waste rules take effect shall submit a disclosure to the agency within 90 days after first producing the hazardous waste. The hazardous waste shall not be disposed of or change possession until at least 30 days after the disclosure is filed with the agency.

B. Any generator who produces a hazardous waste outside the state of Minnesota that is not being transported to a hazardous waste facility within the state of Minnesota on the day these rules take effect shall file a disclosure with the agency before the hazardous waste is transported to a hazardous waste facility within the state of Minnesota. The hazardous waste shall not be transported to a hazardous waste facility within the state of Minnesota until at least 30 days after the disclosure is filed with the agency.

Subp. 3. **Annual resubmission of a disclosure.** After submitting its first disclosure, each generator who is required to submit a disclosure pursuant to subpart 1 shall submit a subsequent disclosure according to the following schedule if any hazardous waste has been produced or managed since the first disclosure:

| FIRST LETTER IN NAME OF GENERATOR | MONTH OF SUBMISSION (The disclosure shall be made the first time the indicated month occurs after the regulations have been in effect for a period of two years.) |
|---|---|
| L-N | January |
| A-C | March |
| D-G | May |
| T-Z | July |
| O-S | September |
| H-K | November |

Each such generator shall submit a new disclosure within ten days of the anniversary date of the second disclosure if any hazardous waste has been produced or managed in the preceding year.

Any generator who is required to file a disclosure pursuant to subpart 2, shall submit a new disclosure within ten days of the anniversary date of the first disclosure.

In submitting a new disclosure for a hazardous waste, the generator need not repeat any information required in a disclosure that has not changed from the previous disclosure, but may merely indicate that the information is the same.

Any generator who does not submit a disclosure because the hazardous waste was not produced or transported during the preceding period or year shall inform the agency of such fact and shall comply with the requirements for submitting a disclosure for a new hazardous waste in the event a hazardous waste is again produced or transported.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0250 IDENTIFICATION NUMBER.

Prior to transportation or disposal of any hazardous waste a generator shall obtain a generator identification number from EPA.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0260 PREPARATION OF HAZARDOUS WASTE SHIPPING PAPERS.

Each generator shall prepare hazardous waste shipping papers for each hazardous waste in accordance with parts 7045.0850 to 7045.0930.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0270 PREPARATION OF HAZARDOUS WASTE LABELS.

Subpart 1. **Requirement.** Each generator shall attach a hazardous waste label to each container and portable tank containing hazardous waste in accordance with the applicable United States Department of Transportation regulations on hazardous materials under Code of Federal Regulations, title 49, part 172 (1979). In addition, the following words and information shall be displayed:

A. HAZARDOUS WASTE - Federal Law Prohibits Improper Disposal. If found, contact the nearest police or public safety authority or the United States Environmental Protection Agency.

B. Generator Name and Address ____________________.

C. Manifest Document Number ____________________.

D. EPA Identification Number ____________________.

E. Accumulation start date ____________________.

Subp. 2. **Labeling for interstate commerce.** The container or portable tank shall be labeled and marked in a manner that is suitable for interstate commerce.

Subp. 3. **Storage tank label.** Any generator or other person who maintains a storage tank containing hazardous waste shall display the words "Hazardous Waste" on the storage tank in a legible and conspicuous manner. The words "Hazardous Waste" shall be plainly visible and legible to any person who may operate any outlet valve.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0280 CONTAINERS AND TANKS.

Each generator shall put hazardous waste only into containers or tanks that comply with the requirements of part 7045.0350 to 7045.0430 for storage of hazardous waste in containers and tanks at hazardous waste facilities.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0290 PROPER HAZARDOUS WASTE MANAGEMENT.

No generator shall relinquish control of a hazardous waste when the generator has reason to believe that the hazardous waste is not being properly managed. Nothing in these parts is intended to restrict or enlarge or affect in any way, any liability the generator may have to correct the mismanagement of the hazardous waste or pay for damages or alleviate any pollution caused by the mismanagement of the hazardous waste.

**Statutory Authority:** *MS s 116.07 subd 4*

## LOCATION, OPERATION, AND CLOSURE OF A HAZARDOUS WASTE FACILITY

## 7045.0350 SCOPE.

Parts 7045.0350 to 7045.0430 establish criteria for the location, operation, and closure of a hazardous waste facility. These parts, however, do not apply to a waste water treatment works that is operated pursuant to an NPDES Permit or State Disposal Permit.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0360 HAZARDOUS WASTE FACILITY LOCATION.

Subpart 1. **Prohibited areas.** No person shall establish, construct, or operate a hazardous waste facility in a wetland, in a floodplain, or within shoreland.

Subp. 2. **Unsuitable areas.** No person shall establish, construct, or operate a hazardous waste facility in a location where the topography, geology, hydrology, or soil is unsuitable for the protection of the groundwater and the surface water.

Subp. 3. **Air pollution.** No person shall establish, construct, or operate a hazardous waste facility in a location where such activity would result in emissions of air contaminants causing the violation of the ambient air quality standards established in parts 7005.0010 to 7005.0080.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0370 HAZARDOUS WASTE FACILITY OPERATION.

Subpart 1. **In general.** No person shall operate a hazardous waste facility except in conformance with the following requirements:

A. The facility operator shall prepare procedures for personnel to follow in the case of spills of hazardous waste and in the case of fire and other emergencies. The facility operator shall post these procedures in a conspicuous place at the facility site.

B. The facility operator shall have safety equipment available at the facility site for use during spills, fires, and other emergencies.

C. The facility operator shall have available at all times written procedures for handling spills, fires, and other emergencies. The facility operator shall train and instruct all personnel at the facility site in these procedures. The facility operator shall maintain records of the training and instruction programs that are held.

D. The facility operator shall construct and begin operating a site monitoring system that is approved by the agency as adequate to determine the effect of the facility on the soil, groundwater, and air before accepting or storing any hazardous waste at the facility.

E. The facility operator shall control access to the facility by the use of fences, gates, locks, and other similar methods and allow access only to persons who are knowledgeable in the safety and emergency procedures needed for handling the hazardous waste. The facility operator shall provide security against unauthorized entry onto the site.

F. The facility operator shall have communication equipment available at the site for summoning aid in an emergency.

G. The facility operator shall maintain lighting at the facility in a manner sufficient to ensure safety and proper operation if the facility is operated during hours of darkness.

H. The facility operator shall not allow scavenging at the facility.

I. The facility operator shall prevent the discharge of hazardous waste from the facility to the surface waters or groundwaters of the state. The facility operator shall prevent hazardous waste from entering drains, sewer inlets, storm sewers, sanitary sewers, doorways, vents, tunnels, pipes, windows, or areas with permeable earth or soil floors.

J. The facility operator shall handle shipping papers as provided in parts 7045.0850 to 7045.0930.

Subp. 2. **Acceptance of hazardous waste.** Acceptance of hazardous waste:

A. The facility operator shall notify the agency by telephone immediately upon delivery and prior to acceptance of a shipment of hazardous waste if any of the following discrepancies exist:

(1) incomplete shipping papers;

(2) a container or portable tank containing hazardous waste is not properly labeled;

(3) the shipping papers and the labels are inconsistent;

(4) the shipping papers and the hazardous waste shipment are inconsistent with regard to quantity, type, or number of containers.

Within ten working days, a follow-up report which fully describes any discrepancy, its resolution, and the management of the hazardous waste shall be mailed to the agency.

B. In the event a shipment of hazardous waste without any shipping papers is delivered to a hazardous waste facility, the facility operator shall immediately notify the agency by telephone of:

(1) the transporter's name and vehicle license plate;

(2) the transporter's address and EPA identification number, if available;

(3) the generator's name, address, and EPA identification number, if available;

(4) a description of the unmanifested waste;

(5) a brief explanation of why the waste was unmanifested, if known.

Within ten working days, a follow-up report which fully describes any discrepancy, its resolution, and the management of the hazardous waste shall be mailed to the agency.

C. No facility operator shall accept a shipment of hazardous waste that the facility operator is not allowed to manage under the hazardous waste facility permit unless written approval is obtained from the director. The director shall approve the acceptance of the waste if the director determines that the hazardous waste is a waste that can be properly managed at the facility, and the generator has filed a disclosure with the agency.

The director shall act on the request as expeditiously as possible.

D. The facility operator shall schedule the arrival of hazardous waste in a manner that minimizes the potential problem of incompatible wastes coming in contact.

Subp. 3. **Storage of hazardous waste in containers and tanks.** Storage:

A. The facility operator shall segregate incompatible wastes stored in containers and tanks to minimize the potential problem of incompatible wastes coming in contact during storage.

B. The facility operator shall regularly inspect all containers and tanks to determine if any leaks have occurred and in the event a leak has occurred, take necessary action pursuant to subpart 1, item C.

C. The facility operator shall store hazardous waste in containers and tanks in a manner such that the facility operator can locate any shipment of hazardous waste and any hazardous waste from any particular generator stored on the site.

D. The facility operator shall store hazardous waste in containers and tanks that are located out-of-doors only within a liner and dike system which meets the following requirements:

(1) The liner and dike system shall have a permeability rate no greater than $10^{-7}$ centimeters per second when being subjected to a head of one foot of water and shall be of a composition that will not increase in permeability as a result of contact with the hazardous waste.

(2) The liner and dike system shall be constructed so as to hold a volume equal to the volume of the largest storage tank plus the total capacity of all containers and portable tanks plus one foot of freeboard.

(3) The interface between the dike and underlying liner shall be constructed so as to provide a seal against movement of hazardous waste or solutions thereof.

(4) The dike shall be constructed in a manner that provides necessary ramps for vehicles needing access to the storage areas.

E. The facility operator shall store hazardous waste in containers that are located out-of-doors in a manner that complies with the following requirements:

(1) The facility operator shall stack containers with a capacity of less than 45 gallons in rows no more than 30 feet in length, five feet in width, and six feet in height, unless otherwise stated in the hazardous waste facility permit.

(2) The facility operator shall store containers with a capacity of 45 gallons or more in rows no more than 30 feet in length and two containers in width and shall not stack the containers, unless otherwise stated in the hazardous waste facility permit.

(3) The facility operator shall maintain a minimum of five feet between rows of containers of hazardous waste.

(4) If exposure of the containers to moisture or direct sunlight will create a hazardous condition or adversely affect the containers' ability to contain the hazardous waste, the facility operator shall store containers in an area with overhead roofing or other covering that does not obstruct the visibility of the labels.

F. No facility operator shall store hazardous waste in containers and tanks unless the containers and tanks meet the following requirements:

(1) Containers and tanks shall be of sturdy, leak-proof construction. Containers shall be of adequate wall thickness, of adequate weld, hinge, and

seam strength and of sufficient material strength to withstand side and bottom shock, while filled, without impairment of the ability of the container or tank to fully contain the hazardous waste.

(2) Except during filling or emptying, the container or tank shall be securely closed. In the event that state or federal law requires a tank to be vented, the tank shall be equipped with a vapor recovery system; provided that persons who store used crankcase oil in tanks with a capacity of less than 5,000 gallons (18,927 liters) shall not be required to equip such tanks with a vapor recovery system.

(3) Lids, caps, hinges, or other closure devices shall be of sufficient strength and construction so that when closed they will withstand dropping, overturning, or other shock without impairment of the container's or tank's ability to fully contain the hazardous waste. Gasketed closures shall be fitted with gaskets of material that is sufficient to prevent leakage and that will not be deteriorated by the contents.

(4) Containers, tanks, and their closures shall be constructed of materials or protected by a liner that will not undergo chemical reaction with the contained waste or with other substances that the container may foreseeably contact if such a reaction may impair the container's or tank's ability to contain the waste.

(5) Corroded or damaged containers or tanks shall not be used to contain hazardous wastes.

(6) Containers and portable tanks of hazardous waste shall be suitable for interstate transportation.

G. Hazardous waste shall not be stored in containers or tanks for more than one year.

Subp. 4. **Reuse of hazardous waste containers.** The facility operator shall handle containers and tanks that have contained hazardous waste in one of the following manners:

A. rinse, clean, and drain all hazardous waste from the containers and tanks prior to leaving the hazardous waste facility;

B. manage the containers and tanks as a hazardous waste; or

C. reuse the containers or tanks without rinsing, cleaning, and draining if all of the following conditions are met:

(1) the containers and tanks be used to store or transport one type of hazardous waste exclusively;

(2) the containers and tanks are closed until reuse; and

(3) the containers and tanks be suitable for use in accordance with parts 7045.0350 to 7045.0430.

Subp. 5. **Disposal of hazardous waste.** The facility operator shall not discharge hazardous waste directly into the saturated zone by such means as injection wells or other devices used for the purposes of injecting materials.

The facility operator shall not dispose of hazardous waste by open burning.

The facility operator shall not engage in activities that would result in emissions of air contaminants causing the violation of the ambient air quality standards established in parts 7005.0010 to 7005.0080.

The facility operator shall not dispose of hazardous waste in a manner that contaminates the soil unless such disposal is authorized in a hazardous waste facility permit.

Subp. 6. **Records and reports.** Records and reports:

A. The facility operator shall submit the site monitoring results to the agency on a quarterly basis.

B. The facility operator shall file a monthly summary with the director that identifies the amount of hazardous waste managed, the names of the generators of the hazardous waste, and the identity of the types of hazardous waste managed; provided, however, that a facility operator who is also a generator and manages only its own waste at an on-site facility shall file this summary on a quarterly basis.

C. The facility operator shall maintain a log at the facility site that indicates the date that each shipment arrived, the shipment number, the name of the generator of the shipment, the name of the transporter who delivered the shipment, the location of the shipment at the facility, and the date that the hazardous waste was processed, disposed of, or transported from the facility. The facility operator shall submit the log to the agency upon the request of the director.

D. The facility operator shall submit to the agency the records of personnel training and instruction in the procedures to follow in handling spills, fires, and other emergencies upon the request of the director.

E. For hazardous waste land disposal facilities, the facility operator shall maintain a ledger for each cell containing the names of the generators of the hazardous wastes, the dates of acceptance of each shipment of hazardous waste, the amount in gallons or tons of each shipment, the shipment numbers, and the chemical composition of each shipment of hazardous waste. The facility operator shall submit the ledger to the agency at the closure of each cell or upon the request of the director.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0380 HAZARDOUS WASTE FACILITY CLOSURE OTHER THAN HAZARDOUS WASTE LAND DISPOSAL FACILITIES.

Subpart 1. **Notice.** The facility operator shall give the agency a minimum of 90 days written notice prior to the closing of the facility. The written notice shall include anticipated last day of operation, the existing inventory count and the inventory reduction schedule, and a discussion of how conditions of the hazardous waste facility permit will be met.

Subp. 2. **Removal of waste.** The facility operator shall remove, before the facility is closed, all hazardous waste from the facility unless otherwise authorized by the hazardous waste facility permit.

Subp. 3. **Compliance with permit.** The facility operator shall meet the conditions of the facility's hazardous waste facility permit for closing the facility. This provision shall apply even if the permit has expired or has been suspended or revoked.

Subp. 4. **Engineer certification.** The facility operator shall submit certification to the agency by a registered professional engineer that the facility has been closed in accordance with the requirements of these parts and the hazardous waste facility permit.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0390 HAZARDOUS WASTE LAND DISPOSAL FACILITY CLOSURE.

Subpart 1. **Notice.** The facility operator shall give the agency a minimum of 180 days written notice prior to closing a hazardous waste land disposal facility. The written notice shall include anticipated last day of operation, a discussion of how the requirements of these parts shall be met, and a discussion of how conditions of the hazardous waste facility permit shall be met.

Subp. 2. **Closure procedures.** The facility operator shall close the hazardous waste land disposal facility in accordance with the following requirements:

A. The facility operator shall close access to the facility and prevent additional waste disposal.

B. The facility operator shall provide, construct, and maintain measures to protect groundwater and surface water and to control air emissions from the facility.

C. The facility operator shall cover the hazardous waste with an adequate amount of cover material to eliminate blowing of the hazardous waste and to minimize leachate production by the hazardous waste.

D. On all areas that have been covered with soil, the facility operator shall cover the area with adequate topsoil and provide vegetation that is sufficient to prevent erosion.

E. The facility operator shall establish and maintain a final grade that promotes surface water runoff without excessive erosion and shall divert surface water drainage around and away from the disposal area.

F. The facility operator shall construct a groundwater monitoring system, a surface water monitoring system and, if necessary, a gas monitoring system if such systems are not already installed.

G. The facility operator shall record a detailed description, including a plat, with the county recorder. The description shall include a statement that the site has been used for the disposal of hazardous wastes, the general types and location of wastes, depth of fill, and other information of interest to potential land owners.

H. The facility operator shall file with the agency and with the appropriate county office a final plot plan and cross sections that delineate the location of each major type of waste disposed of at the facility.

Subp. 3. **Compliance with permit.** The facility operator to whom a hazardous waste facility permit has been issued shall close the facility as required by the permit. Such a facility operator shall submit certification to the agency by a registered professional engineer that the hazardous waste land disposal facility has been closed in accordance with the requirements of these parts and the hazardous waste facility permit. This provision shall apply even if the permit has expired or been suspended or revoked.

Subp. 4. **Financial arrangements.** A facility operator who closes a hazardous waste land disposal facility shall establish and continue in effect financial arrangements that are adequate to finance the long-term maintenance, monitoring, and surveillance required by these parts.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0400 LONG TERM MAINTENANCE, MONITORING, AND SURVEILLANCE OF HAZARDOUS WASTE FACILITIES OTHER THAN HAZARDOUS WASTE LAND DISPOSAL FACILITIES.

Subpart 1. **Prevention of pollution.** A facility operator who closes a hazardous waste facility other than a hazardous waste land disposal facility shall conduct such long term maintenance, monitoring, and surveillance of the facility as is necessary to prevent pollution of the air, land, and water resources of the state.

Subp. 2. **Duration of requirements.** The requirements of these parts shall continue for as long as the hazardous waste poses a threat to the environment, unless the state of Minnesota or the United States agrees to assume responsibility for the long term maintenance, monitoring, and surveillance requirements described herein.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0410 HAZARDOUS WASTE LAND DISPOSAL FACILITY LONG TERM MAINTENANCE AFTER CLOSURE.

Subpart 1. **Requirements.** A facility operator who closes a hazardous waste land disposal facility shall perform the following long term maintenance, monitoring, and surveillance of the facility:

A. maintain the impervious liner and final cover;

B. maintain surface water drainage in a manner that minimizes erosion;

C. treat contaminated surface water runoff;

D. collect and treat leachate;

E. maintain a groundwater monitoring system and a surface water monitoring system and, if necessary, a gas monitoring system;

F. remove hazardous waste from the facility or otherwise alleviate the threat in the event the hazardous waste is a threat to air, land, or water resources of the state, or public health or safety.

The facility operator shall remove the waste or otherwise alleviate the threat regardless of the cause of the threat.

Subp. 2. **Duration of requirements.** The requirements of these parts shall continue for as long as the hazardous waste poses a threat to the environment, unless the state of Minnesota or the United States agrees to assume responsibility for the long term maintenance, monitoring, and surveillance requirements described herein.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0420 CLOSURE OF UNPERMITTED HAZARDOUS WASTE FACILITIES.

The facility operator of a hazardous waste facility that is in operation on the effective date of these parts who does not apply for a hazardous waste facility permit pursuant to parts 7045.0650 to 7045.0730 shall close the facility in accordance with the requirements of these parts.

A facility operator who closed or abandoned a hazardous waste land disposal facility prior to the effective date of these parts shall comply with the requirements of these parts.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0430 SMALL HAZARDOUS WASTE CONTAINERIZED STORAGE FACILITIES.

The facility operator of a hazardous waste containerized storage facility with a capacity of less than 5,000 gallons (18,927 liters) of hazardous waste in containers and tanks shall not be required to comply with the requirements of parts 7045.0360, subpart 1; 7045.0370, subpart 1, items D and F; 7045.0370, subpart 2, item C; 7045.0370, subpart 6, items A, B, C, and D; 7045.0380, subparts 1, 3, and 4, provided no other hazardous waste facility is located at the same site.

**Statutory Authority:** *MS s 116.07 subd 4*

# TRANSPORTATION OF HAZARDOUS WASTE

## 7045.0500 SCOPE.

Parts 7045.0500 to 7045.0570 establish criteria for the loading and transportation of hazardous waste by any person to insure that hazardous wastes are loaded and transported in a manner which minimizes risks to human health and the environment.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0510 LOADING OF HAZARDOUS WASTES.

No person shall load or unload hazardous waste onto or from any motor vehicle, railroad car, barge, airplane, or other vehicle except in accordance with the following requirements:

A. All containers of hazardous waste shall be loaded so that they are reasonably secured against movement within the vehicle by which the hazardous waste is being transported.

B. Tank vehicles shall not be left unattended during the loading or unloading of a hazardous waste.

C. No tools or equipment likely to damage the effectiveness of the closure of any container or adversely affect the ability of a container to contain a hazardous waste shall be used for loading or unloading hazardous waste.

D. Hazardous waste and food or fiber intended for human or animal consumption or use shall not be loaded in the same vehicle.

E. Hazardous waste shall not be loaded in the same vehicle with incompatible wastes or other materials with which it is incompatible.

F. Broken or leaking containers of hazardous waste or containers with an outside surface that is contaminated with hazardous waste shall not be loaded or offered for transportation.

G. No container or tank containing hazardous waste shall be loaded on a vehicle unless the container or tank is properly labeled as required by parts 7045.0200 to 7045.0290.

H. No cargo tank or cargo tank compartment shall be loaded with hazardous waste unless it has been tested, inspected and maintained to insure that there is no unintentional release or leakage of waste during transportation and unless it fulfills the applicable requirements set forth in Code of Federal Regulations, title 49, section 177.824 (1976). The person loading the tank or compartment shall be considered to have complied with this provision if the person owning or leasing the cargo tank certifies that all requirements of this provision have been met.

I. No hazardous waste of a type or volume that is beyond the capability of the cargo tank shall be loaded in the cargo tank.

J. No cargo tank shall be loaded unless it is properly labeled as required by parts 7045.0200 to 7045.0290.

K. The hazardous wastes of two or more generators shall not be commingled unless such commingling is indicated on the shipping papers of all of the generators.

L. The hazardous waste shall be given to a transporter as soon as possible after loading.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0520 TRANSPORTATION OF HAZARDOUS WASTE.

Subpart 1. **Shipping papers.** The operator of a vehicle transporting hazardous waste shall maintain possession of the hazardous waste shipping papers during transportation as follows:

A. When the vehicle is a motor vehicle and the driver is at the vehicle's controls the shipping papers shall be either within his immediate reach while he is restrained by the lap belt, or readily visible to a person entering the driver's compartment or in a folder that is mounted to the inside of the door on the driver's side of the vehicle.

B. When the vehicle is a motor vehicle and the driver is not at the vehicle's controls, the shipping papers shall be displayed as follows: in a holder that is mounted to the inside of the door on the driver's side of the vehicle, or on the driver's seat in the vehicle.

C. When the vehicle is a train, a member of the crew shall maintain the shipping papers in the caboose.

D. When the vehicle is other than a motor vehicle or train, the operator of the vehicle shall maintain the shipping papers in an accessible location determined by the operator.

E. If, pursuant to the provisions of part 7045.0910, a vehicle operator does not have a copy of the shipping papers in his possession, as required by this section, then the transporter shall maintain the spill information given to him by the generator pursuant to part 7045.0910 in such a manner that the information will be available to the vehicle operator as soon as he requests it, or in such other manner that is approved in writing by the agency.

Subp. 2. **Compliance with other rules.** The transporter shall comply with all applicable requirements of parts 7045.0850 to 7045.0930 relating to shipping papers.

Subp. 3. **Labels.** The transporter shall replace any labels required by parts 7045.0200 to 7045.0290 if they are destroyed, lost, or detached.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0530 TIME IN TRANSIT.

Any person who transports hazardous waste shall deliver the hazardous waste to its final destination as soon as possible after loading of the hazardous waste.

In the event that a shipment of hazardous waste is not accepted by the facility operator within 48 hours after arrival at the destination or in the event the facility operator does not sign the hazardous waste shipping papers, the transporter shall immediately return the shipment of hazardous waste to the generator and the generator shall accept it and pay for the return transportation. If the wastes of two or more generators have been commingled as provided in part 7045.0510, item K, each generator shall accept a portion of the hazardous waste equal to the generator's contribution to the total volume of waste.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0540 SPILLS IN TRANSIT.

Subpart 1. **Scope.** This part shall apply to all spills of hazardous wastes while in transit within the state of Minnesota.

Subp. 2. **Compliance with other rules.** Any transporter who has a spill or leak of hazardous waste during transit shall comply with parts 7045.1110 to 7045.1130.

Subp. 3. **Documentation.** In the case of a spill or leakage of hazardous waste during transit, the amount spilled, the amount recovered, the location of the spill site, and the disposition of the spilled wastes and any contaminated material shall be noted on or attached to the hazardous waste shipping papers by the transporter.

Subp. 4. **Notice to generator.** The transporter shall notify the generator as soon as possible of any spill or leak during transit.

Subp. 5. **Written summary maintained for five years.** The generator shall maintain a written summary of all spills and leaks that occur during transit for a period of five years.

Subp. 6. **Procedures.** If during the course of transportation, a container is discovered to be broken or leaking, the transporter shall remove the container to the nearest safe location and isolate it pending proper disposition in the safest and most expeditious manner possible. The generator shall render all reasonable assistance to the transporter in repackaging, packing, and cleaning up the waste so that the trip may be resumed. "All reasonable assistance" means providing the transporter with all necessary information about the waste and about procedures for repackaging, packing, and cleaning up the waste and in addition providing any physical assistance that the generator is uniquely suited to provide and that the transporter is willing to bear the costs of. Nothing in this provision is, however, intended to restrict or enlarge or affect in any way any liability the generator may have to repackage, pack, and clean up the waste.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0550 DELIVERY OF HAZARDOUS WASTE.

Renumbered

No person shall deliver hazardous waste to a hazardous waste facility or give hazardous waste to a transporter for shipment to a hazardous waste facility located in the state of Minnesota, if the facility operator has not obtained a hazardous waste facility permit from the agency. Nothing in this provision is intended to require the transporter to undertake any evaluation of a waste to determine whether it is hazardous.

**Statutory Authority:** *MS s 116.07 subd 4*

R

## 7045.0560 REGISTRATION OF HAZARDOUS WASTE TRANSPORTERS.

Any person who transports hazardous waste that originates or terminates in Minnesota shall obtain an identification number from EPA prior to transporting the hazardous waste.

**Statutory Authority:** *MS s 116.07 subd 4*

R

## 7045.0570 TRANSPORTATION OF USED CRANKCASE OIL.

A transporter of used crankcase oil shall maintain a log that shows the source and disposition of all used crankcase oil. Upon the written request of the director, the transporter shall submit any information from the log that the director requests. The transporter shall retain all information for a period of two years.

**Statutory Authority:** *MS s 116.07 subd 4*

## HAZARDOUS WASTE FACILITY PERMIT PROGRAM

## 7045.0650 SCOPE AND CONSTRUCTION OF RULES.

R

Parts 7045.0650 to 7045.0730 govern the application procedures, the issuance, and the conditions of a hazardous waste facility permit. The provisions in these parts and chapter 7000 shall be construed to complement each other.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0660 OTHER PERMITS.

R

Obtaining a hazardous waste facility permit pursuant to these parts shall not exempt a person from any requirement to obtain any other applicable federal, state, and local permits.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0670 PERMIT REQUIRED.

R

No person shall do any of the following without obtaining a hazardous waste facility permit from the agency:

A. establish, construct, operate, close, or abandon a hazardous waste facility;

B. make any change in, addition to, or extension of a permitted hazardous waste facility or part thereof;

C. make any expansion, production increase, or process modification that results in new or increased capabilities of a permitted hazardous waste facility; or

D. operate such a permitted hazardous waste facility, or part thereof, that has been changed, added to, or extended, or that has new or increased capabilities.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0680 SUBMISSION OF HAZARDOUS WASTE FACILITY PERMIT APPLICATION.

Subpart 1. **Preliminary application.** Any person who is operating a hazardous waste facility on the day these parts take effect shall submit to the agency a preliminary application for a hazardous waste facility permit within 180 days of the effective date of these parts; except any person who, on the effective date of these parts, is operating a hazardous waste containerized storage facility that is not in the same location as any other type of hazardous waste facility shall submit a preliminary application for a hazardous waste facility permit for such facility according to the following schedule:

| The first Letter in the Name of Applicant | Application Due Date |
|---|---|
| L-N | 10 months after effective date |
| A-C | 12 months after effective date |
| D-G | 14 months after effective date |
| T-Z | 16 months after effective date |
| O-S | 18 months after effective date |
| H-K | 20 months after effective date |

Subp. 2. **Final application.** Any person who has submitted a preliminary application for a hazardous waste facility permit shall submit a final application after the person has received the director's comments on the preliminary application.

Subp. 3. **Separate applications.** In the event that a person operates hazardous waste facilities at more than one location, a separate application shall be filed by the person for each facility.

Subp. 4. **Different types of facilities.** In the event that a person operates more than one type of hazardous waste facility at one location, then the person shall file a single application containing all the required information for each type of hazardous waste facility that will be at that location.

Subp. 5. **Waivers.** When the application is for a change, addition to, or extension of a permitted hazardous waste facility or part thereof or when the application is for new or increased capabilities at a permitted hazardous waste facility, the agency may waive in writing the submission of plans and specifications or any parts thereof.

Subp. 6. **Complete applications required.** The agency need not accept a permit application unless the application contains all the information required by these parts. If a permit application is incomplete or deficient, the director shall advise the applicant of such incompleteness or deficiency. Further processing of the application may be suspended until the applicant has supplied the necessary information or otherwise corrected the deficiency.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.0690 GRANTING AND REISSUANCE OF PERMITS.

Subpart 1. **Compliance with statutes and rules.** The agency shall not grant or reissue a hazardous waste facility permit unless the agency determines that the hazardous waste facility and its operation will comply with the requirements of applicable pollution control statutes and rules.

Subp. 2. **Financial arrangements.** The agency shall not grant or reissue a hazardous waste facility permit unless the applicant has established financial arrangements that are adequate to provide for:

A. the proper removal, transportation, and disposal of the total amount of hazardous waste that the facility operator will be permitted to store;

B. the closure of the facility in accordance with these parts and the conditions of the permit;

C. the long term maintenance, monitoring, and surveillance requirements provided for in these parts and the conditions of the permit for a hazardous waste land disposal facility for a period of 30 years after closure, unless the agency determines that a shorter period of time is adequate to determine the long term effect of the facility on the soil, groundwater, and air. Nothing in this rule, however, shall limit the responsibility of the facility operator to provide maintenance, monitoring, and surveillance for a longer period of time in accordance with part 7045.0400.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0700 REVIEW OF PERMITS.

Subpart 1. **Written request.** Any person who wishes to continue to operate a hazardous waste facility shall, at least 180 days before his hazardous waste facility permit expires, submit a written request to the agency for reissuance of the permit.

Subp. 2. **Factors to consider.** The agency shall review the request for reissuance. In reviewing the request, the agency shall consider:

A. whether the permittee is in compliance with or has complied with terms, conditions, requirements, and schedules of compliance of the expiring permit, and with applicable pollution control statutes and rules, including any additions, revisions, or modifications thereto;

B. whether there have been changes in the state of the art during the term of the permit;

C. whether the agency has up-to-date information on the nature of the facility, production levels, the operational practices and monitoring data;

D. whether any modifications to the permit are necessary. In conducting the review, the agency may require additional information to be submitted to aid the review.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0710 HAZARDOUS WASTE FACILITY PERMIT; GENERAL CONDITIONS.

Subpart 1. **Requirements.** All hazardous waste facility permits shall have the following general conditions:

A. The permittee shall establish, construct, operate, and close the facility in accordance with:

(1) the plans, specifications, and reports identified in the permit;

(2) the agency's hazardous waste rules; and

(3) the conditions of the permit issued by the agency.

B. The permittee shall allow any authorized agency employee or agent to enter upon any property, public or private, to have access to and copy any applicable records, to inspect the hazardous waste facility and its operations, to sample any waste, and otherwise to obtain necessary information pertaining to the construction, operation, and closure of the hazardous waste facility and the hazardous waste managed there.

C. The permittee shall, upon the request of any authorized agency employee or agent, disclose the times at which any operation occurs.

D. The permittee shall not store volumes of hazardous wastes in excess of the volumes approved by the agency in the permit.

E. The permittee shall manage only those types of hazardous waste that are approved by the permit.

F. The permit shall have a term not to exceed five years.

Subp. 2. **Additional data.** Hazardous waste facility permits for facilities that existed prior to the effective date of these parts may in addition provide interim dates for achievement of compliance with applicable rules, other relevant laws, and conditions of the permit.

Subp. 3. **New facilities.** Hazardous waste facility permits for new facilities shall, in addition, have the following general conditions:

A. The permittee shall not begin operation of the hazardous waste facility until the permittee has submitted a certification by a registered professional engineer that the hazardous waste facility has been constructed according to the engineering plans and specifications as approved, with any modifications, by the permit.

B. The permittee shall construct and commence operation of the facility within the time schedule specified in the permit.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0720 HAZARDOUS WASTE FACILITY PERMIT; SPECIAL CONDITIONS.

The agency shall include special permit conditions to restrict the establishment, construction, operation, or closure of a hazardous waste facility whenever the agency deems such special conditions necessary in order to perform its responsibilities and duties under its rules and other relevant laws.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0730 EXCEPTIONS.

Subpart 1. **Resource recovery facility.** A generator who establishes, constructs, operates, or closes an on-site hazardous waste resource recovery facility that is owned by the generator and is operated solely for the purpose of recycling hazardous waste produced by that generator shall not be required to obtain a hazardous waste facility permit for that facility.

Subp. 2. **Containerized storage facility.** The facility operator of a hazardous waste containerized storage facility with a capacity of less than 5,000 gallons (18,927 liters) that is not in the same location as any other types of hazardous waste facility shall not be required to obtain a hazardous waste facility permit for that facility.

Subp. 3. **Waste water treatment works.** The facility operator of a waste water treatment works operated pursuant to a national pollutant discharge elimination system permit or state disposal permit shall not be required to obtain a hazardous waste facility permit for that facility.

Subp. 4. **Crankcase oil recipient.** No person shall be required to obtain a hazardous waste facility permit for receiving used crankcase oil from other persons for the purpose of supplying that oil to a recycler if the capacity of the facility is less than 5,000 gallons (18,927 liters) and if the used crankcase oil is not mixed with other wastes.

**Statutory Authority:** *MS s 116.07 subd 4*

## FACILITY PERMIT APPLICATIONS

### 7045.0750 PURPOSE.

Parts 7045.0750 to 7045.0780 establish the information that must be submitted in an application for a hazardous waste facility permit.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0760 PRELIMINARY APPLICATION.

Subpart 1. **Information.** All hazardous waste facilities. Any person who submits a preliminary application to the agency for a hazardous waste facility permit, regardless of the kind of facility, shall provide the following information in the preliminary application:

A. all information required by part 7000.0500;

B. an area plan having a scale and vertical contour intervals sufficient to show existing surrounding features to within one mile radius, and delineating the following:

(1) county, township, and municipal boundaries;

(2) a north arrow, town, range, and section number;

(3) surface waters, floodplains, and wetlands;

(4) boundaries of parks and wildlife refuges;

(5) highways, roads, and rights-of-way for railroads, including a designation of the main access to the facility;

(6) approximate daily utilization of each access route by vehicles transporting hazardous waste;

(7) surface water drainage patterns and drainage divides with the direction of the drainage denoted by arrows;

(8) land use patterns and zoning;

(9) buildings within one-fourth mile of the proposed facility and their apparent uses;

(10) quarries and gravel pits (active and abandoned);

(11) major rock outcroppings and fault zones;

(12) sanitary landfills or dumps (active and abandoned);

(13) the location and surface elevations of all active and abandoned wells within one-fourth mile of the facility;

(14) any other applicable area features necessary to determine the suitability of the area for the hazardous waste facility;

C. a site plot plan of existing conditions at the location of the proposed facility, with the site plot plan having a scale and vertical contour interval acceptable to the director, including all land within 1,000 feet of the property lines of the proposed facility and the following:

(1) county, township, and municipal boundaries;

(2) a north arrow, town, range, and section number;

(3) zoning and land use patterns;

(4) surface waters, floodplains, and wetlands;

(5) highways, roads, and railroads (including rights-of-way of railroads), including a designation of those that will be utilized as main accesses to the facility;

(6) a conceptual layout of the facility;

(7) existing and proposed drainage patterns of surface water runoff denoted by arrows;

(8) sanitary and storm sewers, sewer connections, electric power lines, and underground gas lines serving the facility;

(9) the location and surface elevations of surrounding wells (active and abandoned), on-site soil borings, well installations, and piezometers, all of which shall be tied into a benchmark;

(10) all buildings and their uses;

(11) existing ground cover vegetation;

(12) rock outcroppings, sink holes, and faults;

(13) the boundary lines and ownership of all property bordering the proposed site of the facility;

(14) any other site features necessary to determine the suitability of the site for the hazardous waste facility;

D. an estimate of the cost for:

(1) the proper removal, transportation, and disposal of the total amount of hazardous waste that the applicant has requested to store;

(2) the closure of the facility in accordance with these parts.

Subp. 2. **Containerized storage transfer station and processing facilities.** In addition to the information required by subpart 1, any person who submits a preliminary application for a hazardous waste facility permit for a hazardous waste containerized storage facility, a hazardous waste transfer station or a hazardous waste processing facility shall submit the following additional information:

A. a report that summarizes the available information on the subsurface conditions at the proposed site for the facility and reviews dominant soil types, underlying bedrock, groundwater quality, and the location and depths of all wells within one thousand feet;

B. when required by the director, a report that summarizes the subsurface field investigations conducted by the applicant to determine the feasibility of the proposed location;

C. an engineering report that conceptually assesses the construction of the facility and any existing construction proposed to be used at the facility;

D. a report that conceptually addresses the operation of the facility including when applicable:

(1) a general description of the waste types proposed to be brought to the facility describing the approximate chemical composition, the hazardous properties, and the estimated quantities that will be handled on a yearly basis;

(2) a discussion of the inventory control procedures to be utilized in managing each waste type at the facility;

(3) a description of any processing including, but not limited to, chemical precipitation, incineration, chemical fixation, blending, or repackaging that is proposed to occur at the facility;

(4) a delineation of the actual or proposed management of the hazardous waste that is brought to the facility and that is subsequently removed from the facility for management elsewhere;

(5) a description of the anticipated air emissions, wastewater effluents, hazardous wastes, and solid wastes that will be produced by the facility.

Subp. 3. **Noncontainerized storage land treatment and land disposal facilities.** In addition to the information required by subpart 1, any person who submits a preliminary application for a hazardous waste facility permit for a hazardous waste noncontainerized storage facility, hazardous waste land treatment facility, or hazardous waste land disposal facility shall submit the following additional information:

A. A report on the subsurface conditions at the proposed facility based on a field investigation that includes a sufficient number of soil borings, groundwater monitoring wells, and piezometers to accurately investigate subsurface conditions. The location, placement, and construction of the soil borings, monitoring wells, and piezometers shall be done in a manner that facilitates the preparation of plot plans and cross sections. The report shall include, unless otherwise specified by the director:

(1) Logs of borings classified according to ASTM D 2487-69 and ASTM D 2488-69.

(2) A plot plan that delineates the surface of the underlying groundwater, the direction of groundwater flow, perched water tables, recharge, and discharge areas; the location of soil borings, groundwater monitoring wells,

and piezometers; and the dates of inspection and water levels recorded in establishing the groundwater information listed by each well and piezometer.

(3) The placement and construction of monitoring wells and piezometers.

(4) Cross sections prepared from the field investigation that illustrate soil profile, groundwater aquifers, vertical and horizontal direction of groundwater flow, and other significant geological features, and, should the field investigation indicate the need for an investigation of the underlying bedrock, core samples or cuttings taken from borings and rock types adequately defined as to petrology and stratigraphy.

(5) A comparison of the findings of the field investigations with previous research and literature on the subsurface conditions at the site and an explanation of any discrepancies in the findings of the field investigation and previous research.

(6) An estimated water balance for the location of the proposed facility that considers precipitation, drainage, infiltration, exfiltration, percolation, evaporation, and runoff.

(7) A section that addresses the porosity and permeability of major soil types that were encountered in the field investigation, including a description of the procedures used in the testing of the major soil types. The section shall discuss:

(a) the ability of the soil to attenuate the hazardous waste and the leachate thereof through ion exchange, absorption, adsorption, precipitation, and other such mechanisms;

(b) a review of the anticipated products from such mechanisms including both final and intermediate biochemical metabolites and chemical degradation products;

(c) an assessment of how effective the soil attenuation processes will be providing treatment to the hazardous waste and leachate thereof.

(8) A section that addresses the seasonal fluctuation in groundwater levels, an approximation of the historic high groundwater levels expected based on field investigations, and influences on the groundwater levels by local wells, irrigation, or drainage ditches.

(9) A section on groundwater quality that delineates the natural quality, assesses the potential impact of the hazardous waste to be accepted at the facility and the leachate thereof on groundwater quality, and appraises whether this facility would preclude beneficial present and future uses of the groundwater.

B. An engineering report that conceptually addresses the design of the facility including:

(1) a description of the wastes to be managed at the facility, including the amount, general chemical composition and properties of the waste;

(2) any treatment processes that will be utilized to prepare the waste before land disposal, land treatment, or storage;

(3) a site plot plan that delineates the conceptual engineering plans for the facility;

(4) a section that describes and assesses as applicable:

(a) the preliminary specifications for the liners, the liners currently under consideration and the individual liners' ability to meet those specifications;

(b) the preliminary specifications for the leachate collection system, the materials currently under consideration, and the ability of those materials to meet the specifications;

(c) the preliminary design criteria for any leachate treatment system being proposed;

(5) a conceptual discussion of the operation of the proposed facility.

C. A report that:

(1) evaluates the expected effect of the vapors, gases, and dusts from the wastes on the air quality at the actual site and in the immediate vicinity of the site;

(2) appraises the expected subsurface migration of the vapors and gases from the wastes relative to conditions found in the subsurface investigations.

Subp. 4. **Hazardous waste land disposal facilities.** In addition to the information required by subpart 3, any person who submits a preliminary application for a hazardous waste facility permit for a hazardous waste land disposal facility shall submit an estimate of the cost of maintenance, monitoring, and surveillance of the facility for a 30 year period after closure of the facility, unless the agency determines that a shorter period of time is adequate.

**Statutory Authority:** *MS s 116.07 subd 4*

**7045.0770 FINAL APPLICATION.**

Subpart 1. **All hazardous waste facilities.** Any person who submits a final application to the agency for a hazardous waste facility permit, regardless of the kind of facility, shall provide the following information:

A. Any information required to respond to the comments made by the director on the preliminary application.

B. An engineering report that details the plans and specifications for the construction of the facility, which shall be referenced into the plot plans, including when applicable:

(1) A site plot plan that delineates the final engineering plans for the facility. If the facility involves progressive development of different parts of the area designated for the facility, the applicant shall submit a series of plot plans to illustrate the progressive development of the facility. All site plot plans shall be of sufficient detail, scale, and vertical contour interval to allow for actual construction from the plot plan. The site plot plan shall include:

(a) all information contained on the site plot plan submitted with the preliminary plans and specifications;

(b) a detailed layout of the facility as it is to be built, indicating buildings, fencing, utilities, storage areas, earthworks, and other applicable details;

(c) the location of any air quality, water quality, or groundwater monitoring devices located or proposed to be located at the facility;

(d) arrows delineating surface water drainage patterns after construction of the proposed facility, including the relationship of the drainage patterns to the runoff containment lagoons.

(2) Specifications for the construction of all storage areas and storage tanks, clearly delineating thickness of liners, liner material, grades, drains, sewer inlets, vehicle ramps, foundation construction, and storage tank construction.

(3) A section of the equipment that will be installed at the facility. The section shall include a discussion of underlying physical principles or chemical reactions, detailed drawings, and specifications of all equipment, expected performance data, air emissions data, and the water quality of the wastewater discharge.

(4) A section that delineates the design and specifications for the treatment of contaminated runoff or snow that would arise from the operation of the facility.

C. An operations manual that includes:

(1) a section that delineates procedures, methods, and maintenance that must be done at the facility on a daily or periodic basis to insure proper management of the waste at the facility;

(2) inventory control procedures to be utilized at the facility to properly manage the waste in inventory, including:

(a) locations for storage of each waste type, together with a clear delineation of which waste types are not compatible, the recommended maximum times of storage, and the methods for logging shipments into and out of inventory;

(b) maintenance and inspection schedules for insuring that containers in storage are properly labeled and not leaking.

(3) A thorough description of the type and frequency of inspection or maintenance that shall be done on storage areas, dikes, storage tanks, liners, cover materials, leachate collection systems, and other construction and equipment at the proposed facility.

(4) A monitoring section that describes the procedures to be used and the parameters to be analyzed by the permittee to inventory and identify incoming hazardous waste, conduct air and groundwater monitoring programs, and monitor the management of waste produced by the operation of the facility.

(5) A section on how to operate and manage holding basins for runoff or contaminated snow that arises from the operation of the facility.

(6) A description of the procedures that shall be employed by the facility personnel in responding to spills or other emergency situations that could arise during facility operation. Specific references shall be made to the training or instruction that the facility personnel shall receive, the on-site emergency and safety equipment and the arrangements for emergency services.

(7) A section outlining the specific management plan for all residuals and hazardous wastes that arise from the operation of the facility.

D. A closure manual describing the procedures and construction that will be used to close the facility, and the monitoring and maintenance required to be conducted at the facility after closure.

E. A description of the financial arrangements the applicant has made to pay for the following:

(1) the proper removal, transportation, and disposal of the total amount of hazardous waste that the applicant has requested to store; and

(2) the closure of the facility in accordance with these parts.

Subp. 2. **Noncontainerized storage land treatment and land disposal facilities.** In addition to the information required by subpart 1, any person who submits a final application for a hazardous waste facility permit for a hazardous waste noncontainerized storage facility, hazardous waste land treatment facility, or a hazardous waste land disposal facility, shall submit the following additional information:

A. A report on the subsurface conditions at the proposed facility. The report shall review the results of continued monitoring of groundwater conditions and supplement the information developed in the preliminary application. The report shall review the subsurface facility construction, including the following:

(1) Cross sections that illustrate the design of the facility in relationship to soil profiles, bedrock profiles, groundwater contours, and other

geological features and that delineate the proposed location of lysimeters and groundwater monitoring wells relative to vertical and horizontal groundwater flows.

(2) A section that describes the water balance of the facility and its impact on the existing water balance and quality in the site area, and that contains, when deemed necessary by the director, a plot plan that delineates how the groundwater contours will be affected by development of the facility.

(3) A section that reviews the effect of contaminants should a failure in the engineering design or construction occur. The section shall include an assessment of the ability of contaminants to pass through underlying soils, a description of the potential effect on the groundwater quality, and recommendations for remedial action should it be necessary.

B. A report that provides a detailed assessment of the specifications and design for liners, leachate collection systems, and leachate treatment systems.

C. A report on air and groundwater monitoring systems and other equipment that will be installed at the proposed facility and on the proposed monitoring procedures. The report shall include the location of monitoring wells and air monitoring stations, plans and specifications for the construction of the monitoring wells in accordance with requirements of the Minnesota Department of Health, plans and specifications for the construction of air monitoring stations, the procedures for sampling, the frequency of sampling, and the kind of analyses to be performed.

D. A closure report and plot plan that delineates the finished construction of the facility after closure. The report and plan shall include the following:

(1) A site plot plan of the proposed final conditions at the facility. The plot plan shall have a scale and vertical contour interval acceptable to the director and shall include:

(a) original contours;

(b) proposed final contours;

(c) original surface water drainage patterns;

(d) proposed final surface water drainage patterns;

(e) layout of the leachate collection system;

(f) layout of gas vents, gas migration barriers, and other such gas controls;

(g) access roads; and

(h) finished landscaping.

(2) Cross sections that delineate each finished cell and cross sections that delineate the disposal or storage of each major waste type. The cross sections shall depict liners, leachate collection systems, the waste, cover materials, and other applicable details.

(3) A section that provides specifications for any construction or materials to be used in closing the facility.

E. A report on the long term maintenance, monitoring, and surveillance to be performed at the facility. For a hazardous waste land disposal facility, the report shall include all of the following information. For a hazardous waste noncontainerized storage facility and a hazardous waste land treatment facility, the report shall include any of the following information that is applicable:

(1) a discussion of the long term maintenance of liners, cover material, leachate collection systems, gas controls, and other applicable construction after closure;

(2) a discussion of the operation of the leachate collection and treatment systems, gas controls, and runoff retention basins after closure;

(3) a discussion of the continued sampling and analysis of monitoring wells, leachate collection systems, emitted gases, and surface water runoff after closure;

(4) a discussion of the techniques for removal of chemical wastes from cells in case the waste poses a threat or has created a threat to air, land, or water resources of the state, or to public health or safety, after closure, regardless of the cause of that threat;

(5) a financial plan that indicates how the applicant will provide funds for maintenance, monitoring, and surveillance of the facility for 30 years after closure, unless the agency determines that a shorter period of time is adequate.

**Statutory Authority:** *MS s 116.07 subd 4*

R

### 7045.0780 WAIVER.

Any person who submits a preliminary application or final application to the agency for a hazardous waste facility permit shall not be required to submit that information which the director informs the person in writing is not pertinent to a particular application.

**Statutory Authority:** *MS s 116.07 subd 4*

## HAZARDOUS WASTE SHIPPING PAPERS

R

### 7045.0850 PURPOSE.

Parts 7045.0850 to 7045.0930 establish requirements for the preparation of hazardous waste shipping papers by generators. These parts also establish requirements for the handling, signing, and submission of hazardous waste shipping papers by generators, transporters, and facility operators.

**Statutory Authority:** *MS s 116.07 subd 4*

R

### 7045.0860 SHIPPING PAPERS REQUIRED.

No person shall release, transport, or accept a hazardous waste that is not accompanied by hazardous waste shipping papers.

**Statutory Authority:** *MS s 116.07 subd 4*

R

### 7045.0870 PREPARATION OF SHIPPING PAPERS.

Subpart 1. **By generator per shipment.** Each generator shall prepare hazardous waste shipping papers for each shipment of hazardous waste.

Subp. 2. **Original and copies.** The generator shall prepare an original and a sufficient number of copies of the hazardous waste shipping papers so that all persons who are going to participate in the management of the hazardous waste will be able to comply with the provisions of this part.

Subp. 3. **Information.** The hazardous waste shipping papers shall include the following information:

A. the names, addresses, telephone numbers, and EPA identification numbers of the generator and hazardous waste facility to which the waste is to be transported;

B. the name and EPA identification number of each transporter;

C. an identifying shipment number assigned by the generator in sequential order for each waste shipment;

D. the total quantity of each hazardous waste by units of weight or volume and the type and number of containers as loaded into or onto the transport vehicle;

E. the description of the waste(s) [e.g., proper shipping name, etc.] required by regulations of the United States Department of Transportation in Code of Federal Regulations, title 49, sections 172.101, 172.202, and 172.203 (1979) if applicable; otherwise the description of the waste(s) as listed on the Minnesota hazardous waste disclosure;

F. signature and date blocks for the generator, the transporter, and the facility operator; and

G. the following certification shall appear on the shipping papers:

"This is to certify that the above named materials are properly classified, described, packaged, marked, and labeled and are in proper condition for transportation according to the applicable regulations of the United States Department of Transportation and the EPA."

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0880 SUPPLEMENTAL COVER SHEET.

Each transporter or facility operator who commingles or consolidates more than one shipment of hazardous waste shall prepare a supplemental cover sheet. The supplemental cover sheet shall provide procedures for handling spills, fires, and other emergencies and shall accompany the hazardous waste shipping papers for each individual shipment of hazardous waste until ultimate disposition.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0890 SIGNING AND SUBMISSION OF SHIPPING PAPERS.

Subpart 1. **Procedures.** Prior to relinquishing possession of a shipment of hazardous waste each generator, transporter, and facility operator shall obtain the dated signature of the transporter or facility operator who accepts the shipment of hazardous waste on the original and each copy of the hazardous waste shipping papers, give the original and at least three copies to the transporter and shall send one copy to the following address: Hazardous Waste, MIS, 322 Washington Avenue South, Hopkins, Minnesota 55343.

The transporter shall retain one copy of the hazardous waste shipping papers after relinquishing possession of the hazardous waste and give the original and two copies to the hazardous waste facility operator. The facility operator shall sign the original and the remaining copies of the hazardous waste shipping papers upon gaining possession of the hazardous waste, retaining one copy, sending one copy to the above address and returning the original to the generator within five working days of gaining possession. Generators, transporters, and facility operators shall retain their copies of the shipping papers in accordance with subpart 4.

Subp. 2. **Acceptance of shipment.** Each transporter or facility operator who accepts a shipment of hazardous waste shall sign and date the hazardous waste shipping papers.

Subp. 3. **Outstate shipments.** When a shipment of hazardous waste is to be delivered to a hazardous waste facility located outside the state of Minnesota, the generator shall ensure that the copy of the hazardous waste shipping papers signed by the facility operator is sent to the address listed in subpart 1 within 35 days of the acceptance of the hazardous waste by the hazardous waste facility. If the generator is unable to comply with this deadline, a letter of explanation must be sent to the address listed in subpart 1 within five working days after the expiration of the 35-day period.

Subp. 4. **Retention of copies.** The generator of a shipment of hazardous waste shall maintain the original of the hazardous waste shipping papers for a period of five years after it is returned. Each transporter and facility operator who accepted the shipment of hazardous waste shall maintain a copy of the hazardous waste shipping papers for a period of five years after accepting the hazardous waste.

Subp. 5. **Request of director.** Upon the request of the director any generator, transporter, or facility operator shall submit the original or a copy of hazardous waste shipping papers to the agency at the time and in the manner specified by the director. If the request requires the generator to inform all transporters and facility operators managing the hazardous waste of the request, the generator shall so inform the transporters and facility operators, and the generator, transporters, and facility operators shall comply with the director's request.

Subp. 6. **Ordinance requirement.** In the event that a county ordinance is approved pursuant to parts 7045.1000 to 7045.1030, the generator, transporter, and facility operator of a waste generated within that county or transported to a hazardous waste facility within that county shall sign and submit hazardous waste shipping papers as required by that ordinance.

**Statutory Authority:** *MS s 116.07 subd 4*

R

## 7045.0900 EXEMPTIONS.

A generator who manages the hazardous waste at an on-site hazardous waste processing facility, hazardous waste land disposal facility, or hazardous waste land treatment facility is not required to comply with the requirements of this rule with respect to those wastes on-site.

Generators and transporters of used crankcase oil are not required to have hazardous waste shipping papers accompany the used crankcase oil that they generate or transport unless the used crankcase oil has been mixed with other wastes.

Persons who release or accept hazardous waste that is discharged directly to a sewer system operated pursuant to an NPDES permit or state disposal system permit are exempted from the requirements of this rule with respect to such a discharge.

**Statutory Authority:** *MS s 116.07 subd 4*

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## 7045.0910 MAILING SHIPPING PAPERS.

Any generator in Minnesota who ships his hazardous waste to a hazardous waste facility outside the state of Minnesota or any generator outside the state of Minnesota who ships his hazardous waste to a hazardous waste facility in Minnesota shall mail the required copies of the shipping papers to the hazardous waste facility if both of the following conditions are present:

A. a transporter that neither originates nor terminates a shipment in the state of Minnesota refuses to sign and carry the shipping papers; and

B. there is neither a federal law nor a state law of the state in which the shipping papers were proffered that requires the transporter to sign and carry similar shipping papers.

When the facility operator receives the shipment of hazardous waste, he shall then treat the shipping papers in the same manner as if they had physically arrived with the shipment of hazardous waste. In addition, the generator shall provide all persons who will be transporting the waste within the state of Minnesota with appropriate written information on the procedures for handling spills, fires, and other emergencies.

**Statutory Authority:** *MS s 116.07 subd 4*

R

## 7045.0920 HAZARDOUS WASTE SHIPPING PAPER FORMAT.

Nothing in this rule shall be construed to require the shipping paper to be a document that is separate from and in addition to other documents already being utilized for the transportation of hazardous wastes. A generator may use a bill of lading or any other such document to fulfill the requirements of this rule so long as it contains all the required information and is handled as required by this rule. The information shall, however, be in a format that will make it amenable to computerized data processing and that has been approved by the agency.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.0930 CERTIFIED BILL OF LADING.

Any generator that gives a bill of lading to a transporter shall, prior to the execution of the bill of lading, determine whether the facility operator can and will accept the shipment of hazardous waste and whether the facility, if within Minnesota, has a hazardous waste facility permit. The generator shall certify on the bill of lading that the facility operator can and will accept the shipment of hazardous waste and that the facility has a hazardous waste facility permit.

**Statutory Authority:** *MS s 116.07 subd 4*

## COUNTY REGULATION OF HAZARDOUS WASTE MANAGEMENT

### 7045.1000 SCOPE AND PURPOSE.

Parts 7045.1000 to 7045.1030 establish procedures for submission of documents in the event the county ordinance is approved by the agency. Issuing, denying, modifying, imposing conditions upon, or revoking hazardous waste generator licenses or permits, and county hazardous waste rules, shall be subject to review, denial, suspension, and reversal by the agency.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.1010 REVIEW OF COUNTY ORDINANCES.

Subpart 1. **Agency approval.** A county that seeks agency approval of a hazardous waste ordinance shall submit a copy of the ordinance to the agency. The agency shall advise the county in writing whether the ordinance is approved. The agency shall approve a county ordinance that embodies the standards and requirements set forth in these hazardous waste rules.

Subp. 2. **Procedures.** If a metropolitan county submits a county hazardous waste ordinance to the agency for approval pursuant to Minnesota Statutes, section 473.811, subdivision 5, the procedure established in part 7045.1030, subpart 1 for agency review of county hazardous waste licenses and permits shall be followed. Any action by the agency pursuant to such submission for agency review of the county ordinance shall not be deemed to be agency approval of such ordinance unless such approval is explicit and is in writing.

Subp. 3. **Metropolitan counties.** For the purposes of these parts a metropolitan county is any one of the following counties: Anoka, Carver, Dakota, Hennepin, Ramsey, Scott, and Washington.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.1020 EFFECT OF AGENCY APPROVAL OF COUNTY ORDINANCE.

In the event that a county has adopted a hazardous waste ordinance that is approved in writing by the agency:

A. Each generator who produces a hazardous waste within the county shall not be required to submit a disclosure to the agency for that waste unless specifically requested in writing by the director to do so.

B. Each generator shall submit the required copies of the hazardous waste shipping papers to the county as required by the county ordinance for each shipment of hazardous waste that is transported.

C. All persons shall comply with all other requirements of these parts and all requirements of the county ordinance.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.1030 DUTIES OF COUNTIES.

Subpart 1. **Notice; agenda.** A county shall submit to the agency written notification of all hazardous waste generator licenses or permits approved or reviewed by the county during the previous month. The notification shall be submitted to the agency on the fifteenth day of each month. Upon the request of the director, the county shall provide the agency with a copy of all the information that it considered in reaching its decision. The agency shall place the matter on the agenda of the next regularly scheduled meeting of the agency, which will be on the fourth Tuesday of the month. The agency may amend, modify, suspend, or reverse the action of the county. The action of the agency in reviewing the county decision to grant the license or permit shall not affect the agency's consideration of a hazardous waste facility permit for the same facility under these parts.

Subp. 2. **Disclosures.** A county shall submit to the director, upon request, a copy of any disclosure that has been submitted to the county.

Subp. 3. **Annual summary.** A county shall submit to the agency a yearly summary of hazardous waste management in the county. The yearly summary shall be submitted by March 1 for the year that ended on the previous December 31. The summary shall contain:

A. the name and identification numbers assigned by the county to each generator in the county;

B. the total number of hazardous waste shipments;

C. the total quantities shipped for each type of hazardous waste;

D. the identification numbers pursuant to part 7045.0560 and names of the transporters used;

E. facilities at which the waste was stored, processed, or disposed;

F. number of spills and accidents; and

G. any other information requested by the director.

**Statutory Authority:** *MS s 116.07 subd 4*

## SPILLS AND LEAKS

### 7045.1110 DUTY TO REPORT.

Any person who owns, has possession of, or otherwise has control of a hazardous waste that spills, leaks, or otherwise escapes from a container, vehicle tank, storage tank, portable tank, or other containment system, including its associated piping, shall immediately notify the agency if the hazardous waste may cause pollution of the air, land, or waters of the state. The person shall use, when applicable, the agency's 24-hour telephone notification service.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.1120 DUTY TO RECOVER.

Any person who owns, has possession of, or otherwise has control of a hazardous waste that spills, leaks, or otherwise escapes from a container, vehicle tank, storage tank, portable tank, or other containment system, including its associated piping, shall recover the hazardous waste as rapidly and as thoroughly as possible and shall immediately take such other action as may be reasonably possible to protect human life and health and minimize or abate pollution of the water, air, or land resources of the state caused thereby.

**Statutory Authority:** *MS s 116.07 subd 4*

### 7045.1130 OPEN BURNING.

No person shall undertake open burning of such hazardous waste unless the open burning has been approved in writing by the director. The director may approve open burning of the following substances: distilled petroleum products, or crude oil, if it is not possible to recover the crude oil.

The director shall consider the location of such hazardous waste and the wind conditions and may approve such open burning only if there will be no adverse effect on residential areas or on traffic conditions.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.1140 APPENDIX A.

AMERICAN NATIONAL STANDARD ANSI/ASTM D 3243 - 76

# Standard Test Method for FLASH POINT OF AVIATION TURBINE FUELS BY SETAFLASH CLOSED TESTER[1]

This Standard is issued under the fixed designation D 3243; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

## 1. Scope

1.1 This method describes a procedure for the determination of the flash point, by a Setaflash* Tester, of aviation turbine fuels. The procedure may be used to determine whether a fuel will or will not flash at a specified temperature (go-no-go flash) or the actual flash point temperature.

1.2 Results by using the Setaflash Tester have been shown to be comparable in magnitude to those obtained by using the Tag Closed-Cup procedure as described in Method D 56.

1.3 *This standard should be used solely to measure and describe the properties of materials, products, or systems in response to heat and flame under controlled laboratory conditions and should not be considered or used for the description, appraisal, or regulation of the fire hazard of materials, products, or systems under actual fire conditions.*

## 2. Applicable Documents

2.1 *ASTM Standards:*

D 56 Test for Flash Point by Tag Closed Tester[2]

E 1 Specification for ASTM Thermometers[3]

## 3. Summary of Method

3.1 By means of a syringe, 2 ml of sample is introduced through a leakproof entry port into the tightly closed Setaflash Tester that has been brought to within about 5°F (3°C) below the expected flashpoint. After 1 min, to allow the sample to come to the same temperature as the aluminum alloy body of the tester, in which the thermometer is inserted, the temperature of the apparatus is raised to the "expected" flash point temperature. A test flame is applied inside the cup and note is taken as to whether the test sample flashes or not.

3.2 As a go-no-go test, the "expected" flash point temperature is a specification or other operating target value. For a finite flash measurement, the temperature is moved through the anticipated temperature range, the test flame being applied at 2°F (1.0°C) intervals until a flash is observed; a second trial is made using the first value as the "expected" flash point temperature, this time making tests at 1°F (0.5°C) intervals.

## 4. Apparatus

4.1 *Flash cup and operating mechanism*, illustrated in Fig. 1 and dimensioned in detail in Annex A1. Fasten electrical heaters to the cup in such a way so as to provide for efficient transfer of heat. Provide a variable heater control device with a scaled dial and a visible signal indicating when energy is or is not being applied. Energy may be supplied from a 115 or 230-V a-c main service (for stationary use) or by a 12-V d-c battery service (for field use).

4.2 *Test Flame and Pilot Flame*—Regulatable test flame, for dipping into the test cup to try for flash, and a pilot flame to maintain the test flame, are required. These flames may be fueled by piped gas service (fixed location) or by a self-contained tank (4.2.1) of LP gas lighter fluid (for portability). Provide a gage ring, 1/4 in. (4 mm) in diameter, mounted on a post near the test flame against which the size of the flame may be judged.

4.2.1 Never recharge the gas tank with the pilot or test flames alight, nor in the vicinity of other naked flames.

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[1] This method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants.

Current edition approved Aug. 27, 1976. Published November 1976. Originally published as D 3243 - 73 T. Last previous edition D 3243 - 73 T.

[2] *Annual Book of ASTM Standards*, Part 23.

[3] *Annual Book of ASTM Standards*, Parts 25 and 44.

for a flash at the cup opening.

7.3.5 The sample is deemed to have flashed only if a comparatively large blue flame appears and propagates itself over the surface of the sample. Occasionally, particularly near the temperature of the actual flash point, the application of the test flame will cause a halo, which should be ignored.

7.3.6 Record the test result as flash (or no flash) at the temperature and the barometric pressure. Make a barometric pressure correction, if necessary (9.1).

7.4 *Test Procedure for Actual Flash Point:*

7.4.1 When a finite flash point is to be determined, make a trial test beginning at about 10°F (6°C) below the expected value. If no flash is observed, continue heating and test for flash at each 2°F (1°C); the rate of temperature rise may be as rapid as can be conveniently followed when watching both the thermometer and the test flame. When a flash is observed, use it as the expected flash point value in setting the test cup temperature for the finite flash point.

7.4.2 After cleaning the test cup, adjust the temperature so that it is stable at about 5°F (3°C) below the expected flash point (7.3); charge the syringe with a fresh test sample and transfer the sample to the filling orifice (Fig. 1) taking care not to lose any sample. Then discharge the sample into the test cup by depressing the syringe plunger to its lowest position.

NOTE 3—The test cup temperature is stable when the signal light slowly cycles ON/OFF.

7.4.3 Move the 1-min timing device by rotating its knob clockwise to the required setting. In the meantime, open the gas control valve, and light the pilot and test flames. Adjust the test flame size with the pinch valve so as to match the size of the $\frac{1}{8}$ in. (4 mm) diameter of the gage.

7.4.4 After 1 min has elapsed, turn the heater control toward FULL ON. Apply the test flame at each 1°F (0.5°C) interval by slowly and uniformly opening the slide fully and closing completely over a period of approximately $2\frac{1}{2}$ s. Watch for a flash at each application of the test flame.

7.4.5 When the flash point (7.3.5) is observed, record the flash point temperature and barometric pressure. If the barometric pressure differs from 760 mm (101.3 kPa), make necessary corrections according to 9.1.

7.4.6 Turn off pilot and test flames and clean the testing apparatus.

7.4.7 Never make a repeat flash point determination using the same sample; always use a fresh portion of sample for each new test.

## 8. Clean-Up of Apparatus

8.1 To prepare for the next test, unlock the lid assembly of the tester and raise to the hinge stop. Soak up sample with an absorbent paper tissue. Clean the underside of the lid and the filling orifice; a pipe cleaner may be of assistance in cleaning the latter.

8.1.1 Any further cleaning necessary may be carried out by complete removal of the lid and shutter assembly. Disconnect the silicone rubber gas tube and slide the lid assembly to the right to remove. If warm, handle gingerly.

8.2 After the cup has been cleaned, its temperature may be rapidly reduced to some stand-by value by turning the temperature control dial to an appropriate point.

8.2.1 It is convenient to hold the test cup at some stand-by temperature (depending on planned usage) to conserve time in bringing the cup within the test temperature range. The cup temperature may be quickly lowered by placing an ice cube in the cup, sopping up the water before it spills over the edge. A neater operation is possible by inserting the aluminum cooling block (4.6) which has been kept in an ice-water bath.

8.3 The syringe is easily cleaned by filling with acetone twice, discharging each time, and allowing to air dry with the plunger removed. Replace the plunger, and pump several times to replace any acetone vapor with air.

## 9. Correction for Barometric Pressure

9.1 When the barometric pressure differs from 760 mm Hg (101.3 kPa), calculate the flash temperature by means of the following equation:

$$\text{Calculated flash point} = T_F + 0.06\,(760 - P)$$
$$= T_C + 0.03\,(760 - P)$$

where:

$T_F, T_C$ = observed flash point, °F or °C, and
$P$ = barometric pressure, mm Hg

## 10. Precision

10.1 The following criteria should be used

4.3 *1-min Audible Signal*, a desirable accessory.

4.4 *Thermometer*, conforming to specifications given in Annex A2: test to determine that the scale error does not exceed 0.5°F (1.25°C). The use of a magnifying lens significantly assists in making temperature observations.

4.5 *Syringe*, a 2 ± 0.05-ml capacity at 77°F (25°C), is needed for selecting a uniform size sample. Check the capacity by discharging water into a beaker and weighing. Adjust plunger stop if necessary.

NOTE 1—A unit[4] meeting all of the requirements described above is shown in Fig. 2.

4.6 *Aluminum Alloy Cooling Block* (*with knob handle*), that fits snugly within the test cup and is about 2½ times the volume of the cup, is a useful accessory for rapid cooling of the sample cup between tests.

## 5. Sample

5.1 The size of sample required for each test is 2 ml. Obtain at least a 25-ml sample at the bulk test site and store in a tight screw cap clean glass container.

5.2 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open sample containers unnecessarily and do not transfer sample unless its temperature is at least 20°F (11°C) below the expected flash point. Discard samples in leaky containers.

## 6. Preparation of Apparatus

6.1 After thermometer removal or replacement, fix in place in its pocket (Fig. 1) with a good heat transfer paste.

6.2 Before initial use, determine the relationship between the temperature control dial and thermometer readings at intervals not exceeding 10°F (6°C) throughout the scale range of the heater. This calibration is useful in making the desired settings to establish a desired test temperature during a test.

6.3 As nearly as possible, place the tester in a position where it is not exposed to disturbing drafts. Provide a shield if necessary.

6.4 Read the manufacturers operating and maintenance instructions on the care and servicing of the tester, and for specific suggestions on the operation of its various controls.

## 7. Procedure

7.1 Inspect the inside of the test cup, lid and shutter mechanism for cleanliness and freedom from contamination. Use an absorbent paper tissue to wipe clean, if necessary. Put cover in place and lock securely.

7.2 To use the tester, switch the instrument on and turn the heater dial to a position corresponding to the desired test temperature.

NOTE 2—A calibration curve of Temperature Control Dial setting *versus* Sample Well Temperature is furnished with each instrument. If such a calibration is not available, one should be prepared from observations of test cup temperature for each dial setting as this will be useful in making desired temperature settings.

7.3 *Procedure for Go-No-Go Flash Point:*

7.3.1 Switch the instrument ON and turn the heater dial fully clockwise (FULL ON) causing the heater signal light to glow. When the thermometer reaches a temperature of about 5°F (3°C) below the expected or target flash point temperature (7.3.1.1) reduce the heat input to the test cup by turning the heater control dial, counterclockwise to the point at which the signal light is just extinguished.

7.3.1.1 When the tester is used in the go-no-go configuration, the specification or target value is the expected flash point.

7.3.1.2 When the correct temperature can be dialed on the temperature controller (6.2), the elapsed time to reach it may be greater than when turned to FULL ON, but less attention will be required in the intervening period.

7.3.2 When the test temperature is reached and the signal light is just extinguished, charge the syringe with a sample of the fuel to be tested and transfer the charge to the filler orifice. Discharge the test sample into the test cup by fully depressing the plunger of the syringe and then removing the syringe.

7.3.3 Move the 1-min timing device by rotating the knob clockwise to its stop. Open the gas control valve and light the test and pilot control flames. Adjust the pilot flame with the pinch valve to conform to the size of the 5/32-in. (4-mm) gage.

7.3.4 After 1 min has elapsed, apply the test flame by slowly and uniformly opening the slide valve and then closing it completely over a period of approximately 2½ s. Watch closely

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[4] A Setaflash Low Temperature Tester, Model SF LV 1374, as shown in Fig. 2 is manufactured by Stanhope-Seta, Ltd., Egham, Surrey, England, and available in the U.S. from Erdco Engineering Corp., 136 Official Rd., Addison, Ill., 60101.

for judging the acceptability of results (95% confidence):

10.1.1 *Repeatability*[4]—Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

| Range | Repeatability |
|---|---|
| 70 to 200°F (21 to 94°C) | 2°F (1.1°C) |

10.1.2 *Reproducibility*[4]—The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amounts:

| Range | Reproducibility |
|---|---|
| 70 to 200°F (21 to 94°C) | 7°F (3.9°C) |

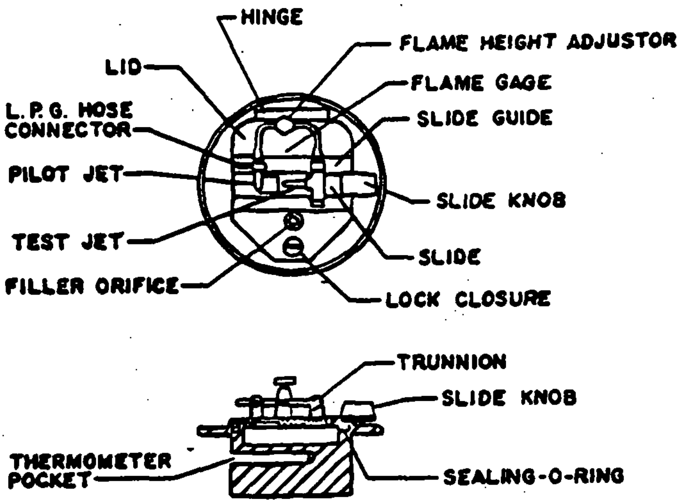

FIG. 1 Setaflash Cup Unit.

**Statutory Authority:** *MS s 116.07 subd 4*

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[4] The results of the cooperative test program, from which these values have been derived, are filed at ASTM Headquarters as Research Report No. RR-1008: D-2.

## 7045.1150 APPENDIX B.

**Designation: D 56 – 70 (Reapproved 1975)**

American National Standard Z11.24-1971
Approved Aug. 17, 1971
By American National Standards Institute
Method 1101—Federal Test
Method Standard No. 791b
Replaces Method 4291 of Federal Test
Method Standard No. 141A

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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### Standard Method of Test for

# FLASH POINT BY TAG CLOSED TESTER[1]

This Standard is issued under the fixed designation D 56; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

## 1. Scope

1.1 This method covers the determination of the flash point, by Tag closed tester, of liquids with a viscosity of below 45 SUS at 100 F (37.8 C) and a flash point below 200 F (93 C) except cut-back asphalts and those liquids which tend to form a surface film under test conditions.

NOTE 1—For the closed cup flash point of liquids with a viscosity of 45 SUS or more at 100 F (37.8 C) or a flash point of 200 F or higher, and those liquids which tend to form a surface film under test conditions use ASTM Method D 93, Test for Flash Point by Pensky-Martens Closed Tester.[2] For cut-back asphalts refer to ASTM Method D 1310, Test for Flash Point of Liquids by Tag Open-Cup Apparatus.[2]

NOTE 2—The U. S. Department of Transportation (OHM)[3] and U. S. Department of Labor (OSHA) have established that liquids with a flash point under 100 F (37.8 C) are flammable as determined by this method for those liquids which have a viscosity less than 45 SUS at 100 F (37.8 C) or do not contain suspended solids or do not have a tendency to form a surface film while under test. Other classification flash points have been established by these Departments for liquids using this test.

Liquids having viscosities more than 45 SUS at 100 F (37.8 C) and contain suspended solids or have a tendency to form a surface film while under test are regulated in accordance with Method D 93.

Coast Guard and Consumer Product Safety Commission Regulations are required by law to define flammable liquids as those having a flash point under 80 F (26.7 C) by an Open Cup Method (Method D 1310).

1.2 *This standard should be used solely to measure and describe the properties of materials, products, or systems in response to heat and flame under controlled laboratory conditions and should not be considered or used for the description, appraisal, or regulation of the fire hazard of materials, products, or systems under actual fire conditions.*

## 2. Summary of Method

2.1 The sample is placed in the cup of the tester and, with the lid closed, heated at a specified constant rate. A small flame of specified size is directed into the cup at regular intervals. The flash point is taken as the lowest temperature at which application of the test flame causes the vapor above the sample to ignite.

## 3. Apparatus

3.1 *Tag Closed Tester*—The apparatus is shown in Fig. 1 and described in detail in Appendix A1. Refer to Appendix A2 for directions for checking the condition and operation of the tester.

3.2 *Shield*—A shield 18 in. (460 mm) square and 24 in. (610 mm) high, open in front, is recommended.

3.3 *Thermometers*—For the test cup thermometer, use one as prescribed in Table 1. For the bath thermometer, any convenient type which has an adequately open scale covering the required range may be used; it is often convenient to use the same type of thermometer as used in the test cup.

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[1] This method is under the joint jurisdiction of ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products, and Committee D-2 on Petroleum Products and Lubricants.

Current edition effective Sept. 11, 1970. Originally issued 1918. Replaces D 56 - 64.

[2] *Annual Book of ASTM Standards*, Part 29.

[3] For information on U.S. Department of Transportation's regulations, see Codes of U.S. Regulations 49 CFR Chapter I and for information on U.S. Department of Labor's regulations see Code of U.S. Regulations 29 CFR Chapter XVII. Each of these items are revised annually and may be procured from the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.

NOTE 3—Whenever thermometers complying with ASTM requirements are not available, thermometers complying with the requirements for The Institute of Petroleum thermometer IP 15F PM-Low may be used.

NOTE 4—There are automatic flash point testers available and in use which may be advantageous in the saving of testing time, permit the use of smaller samples, and other factors which may merit their use. If automatic testers are used, the user must be sure that all of the manufacturer's instructions for calibrating, adjusting, and operating the instrument are followed. In any cases of dispute, the flash point as determined manually shall be considered the referee test.

## 4. Sample

4.1 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Containers shall not be opened unnecessarily and transfers shall not be made unless the sample temperature is at least 20 F (11 C) below the expected flash point. Samples in leaky containers shall be discarded.

## 5. *Preparation of Apparatus*

5.1 Support the tester on a level steady table. Unless tests are made in a draft-free room or compartment, surround the tester on three sides by the shield for protection from drafts. Tests made in a laboratory draft hood or near ventilators are not to be relied upon.

5.2 Gas is recommended for the test flame. If gas is not available, insert a wick of cotton in the burner tip, place small quantity of cotton waste in the chamber to which the burner tip is attached, and fill the chamber with signal, sperm, or lard oil.

## 6. Procedure

6.1 For flash points below 55 F (13 C) or above 140 F (60 C), use as bath liquid a 1 + 1 mixture of water and ethylene glycol. For flash points between 55 F (13 C) and 140 F (60 C), either water or water-glycol mixture may be used as bath liquid (Note 4). The temperature of the liquid in the bath shall be at least 20 F (11 C) below the expected flash point at the time of introduction of the sample into the test cup. Do not cool the bath liquid by direct contact with carbon dioxide or "dry ice." Place the test cup in position in the bath.

NOTE 5—Due to possible difficulty in maintaining the prescribed rate of temperature rise and due to the formation of ice on the lid, results by this method for samples having flash points below 32 F (0 C) may be somewhat unreliable. Trouble due to ice formation on the slide may be minimized by carefully lubricating the slide shutter with high-vacuum silicone lubricant.

6.2 Using a graduate and taking care to avoid wetting the cup above the final liquid level, measure 50 ± 0.5 ml of the sample into the cup, both the sample and graduate being precooled, if necessary, so that the sample temperature at the time of measurement will be 80 ± 10 F (27 ± 5.6 C) or at least 20 F (11 C) below the expected flash point, whichever is lower. It is essential that the sample temperature be maintained at least 20 F (11 C) below the expected flash point during the transfers from the sample container to the graduate and from the graduate to the test cup. Destroy air bubbles on the surface of the sample. Wipe the inside of the cover with a clean cloth or absorbent tissue paper; then attach the lid, with the thermometer in place, to the bath collar.

6.3 Light the test flame, adjusting it to the size of the small bead on the cover. Operate the mechanism on the cover in such a manner as to introduce the test flame into the vapor space of the cup, and immediately bring it up again. The time consumed for the full operation shall be about 1 s, or the time required to pronounce distinctly the words "thousand and one." Avoid any jerkiness in the operation of depressing and raising the test flame.

6.4 *Flash Points Below 140 F (60 C)*—If the flash point of the sample is known to be below 140 F (60 C), apply and adjust the heat so that the temperature of the sample will rise at a rate of 2 F (1 C)/min ± 6 s. When the temperature of the sample in the test cup is 10 F (5.6 C) below its expected flash point, apply the test flame in the manner just described in 6.3, and repeat the application of the test flame after each 1 F (0.6 C) rise in temperature of the sample.

6.5 *Flash Points at or above 140 F (60 C)*—If the flash point of the sample is known to be 140 F or higher, apply and adjust the heat so that the temperature of the sample will rise at a rate of 5 F (3 C)/min ± 6 s. When the temperature of the sample in the test cup is 10 F (5.6 C) below its expected flash point, apply the test flame in the manner described in 6.3, and repeat the application of the test flame after each 2 F (1 C) rise in temperature of the sample, at each temperature reading that is a multiple of 2 F (1 C).

6.6 When the test flame application causes a distinct flash in the interior of the cup, observe and record the temperature of the sample as the flash point. Do not confuse the true flash with the bluish halo which sometimes surrounds the test flame at applications immediately preceding the actual flash.

6.7 Discontinue the test and remove the source of heat. Lift the lid and wipe off the thermometer bulb. Remove the sample cup, empty, and wipe dry.

6.8 If, at any time between the first introduction of the test flame and the observation of the flash point, the rise in temperature of the sample is not within the specified rate or if the actual flash point differs from the expected flash point by an amount greater than 4 F (2 C), discard the result and repeat the test, adjusting the source of heat to secure the proper rate of temperature rise and/or using a modified "expected flash point," as required.

NOTE 6—Never make a repeat test on the same portion of sample once used; always take a fresh portion of sample for each test.

## 7. Correction for Barometric Pressure

7.1 Observe and record the barometric pressure at the time of the test. Make a correction on the following basis: for each 25 mm (1 in.) below 760 mm (29.92 in.) barometric reading, add 0.9 C (1.6 F) to the observed flash point; for each 25 mm (1 in.) above 760 mm (29.92 in.) barometric reading, subtract 0.9 C (1.6 F) from the observed flash point. After applying the correction, round the value obtained to the nearest whole number.

## 8. Precision

8.1 The following criteria should be used for judging the acceptability of results (95 percent probability).[4]

8.1.1 *Repeatability*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts:

| Flash Point | Repeatability |
|---|---|
| Below 140 F (60 C) | 2 F (1.1 C) |
| 140 F (60 C) to 199 F (93 C) | 3 F (1.7 C) |

8.1.2 *Reproducibility*—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts:

| Flash Point | Reproducibility |
|---|---|
| Below 55 F (13 C) | 6 F (3.3 C) |
| 55 F (13 C) to 139 F (59 C) | 4 F (2.2 C) |
| 140 F (60 C) to 199 F (93 C) | 6 F (3.3 C) |

TABLE 1 Thermometers

| For Tests ....... | Below 40 F (4 C) | At 40 to 120 F (4 to 49 C) | Above 120 F (49 C) |
|---|---|---|---|
| Use ASTM Thermometer[A] ....... | 57F or 57C | 9F or 9C<br>57F or 57C | 9F or 9C |

[A] Complete specifications for these thermometers are given in ASTM Specification E 1, for ASTM Thermometers.[5]

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[4] Supporting data for this method have been filed at ASTM Headquarters as RR: D-2-1003.

[5] *Annual Book of ASTM Standards*, Part 25.

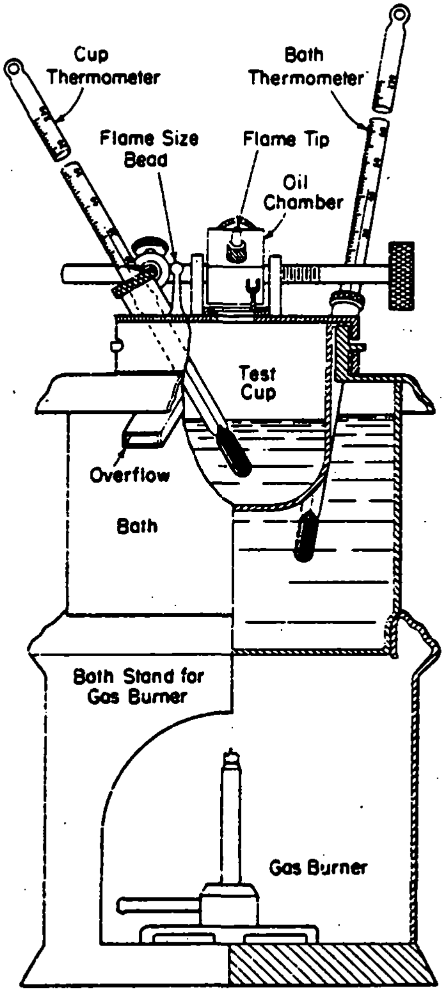

FIG. 1 Tag Closed Flash Tester.

## APPENDIXES

### A1. APPARATUS

A1.1 The Tag closed tester shall consist of the test cup, lid with test flame, and liquid bath conforming to the following requirements:

A1.1.1 *Test Cup*, of brass or other nonrusting metal of equivalent heat conductivity, conforming to dimensional requirements prescribed in Table A1. It shall weigh 68 ± 1 g.

A1.1.2 *Lid:*

A1.1.2.1 The lid comprises a circle of nonrusting metal with a rim projecting downward about ⅝ in. (15.9 mm), a slide shutter, a device which simultaneously opens the shutter and depresses the tip of the tube which carries fuel through to the test flame, and a slanting collar in which the cup-thermometer ferrule is inserted. Figure A1 gives a diagram of the upper surface of the lid, showing dimensions and positions of the three holes opened and closed by the shutter, and the size and position of the opening for the cup thermometer.

A1.1.2.2 The rim shall fit the collar of the liquid bath with a clearance not exceeding 0.002 in. (0.05 mm) and shall be slotted in such a manner as to press the lid firmly down on the top of the cup when the latter is in place in the bath. When this requirement is not met, the vertical position of the cup in the bath shall be suitably adjusted, as by placing a thin ring of metal under the flange of the cup.

A1.1.2.3 The shutter shall be of such size and shape that it covers the three openings in the lid when in the closed position and uncovers them completely when in the open position. The nozzle of the flame-exposure device shall conform to the dimensions given in Table A1. The device shall be designed and constructed so that opening the shutter depresses the tip to a point approximately 0.08 in. (2 mm) to the right of the horizontal center of the middle opening of the lid. (Refer to lower part of Fig. A2.) This will bring the test flame to the approximate center of the opening. The plane of the underside of the lid shall be between the top and bottom of the opening in the tip of the flame-exposure device when the latter is fully depressed.

A1.1.2.4 The collar for the cup-thermometer ferrule shall be set at an angle which permits placement of the thermometer with its bulb approximately in the horizontal center of the cup, at a depth prescribed in Table A1.

A1.1.3 *Liquid Bath*, conforming to the limiting or minimum dimensions shown in Fig. A2. It shall be of brass, copper, or other noncorroding metal of substantial construction. Sheet metal of about No. 20 B & S gage (0.812 mm) is satisfactory. It may, if desired, be lagged with heat-insulating material to facilitate control of temperature.

A1.1.4 *Heater*, of any type (electric, gas, alcohol, etc.) capable of controlling temperature as required in Section 6. An external electric heater, controlled by a variable voltage transformer, is recommended.

A1.1.5 *Bath Stand*—For electric heating, any type of stand may be used. For alcohol lamp or gas burner, a stand, as illustrated in Fig. 1, to protect the flame from air currents (unless tests can be made in a draft-free room) is required.

## A2. CHECKING CONDITION AND OPERATION OF TAG CLOSED TESTERS

### A2.1 Material

A2.1.1 *p-Xylene*,[6] conforming to the following requirements:

Specific gravity (60/60 F) (15.6/15.6 C), 0.860 min, 0.866 max.

Boiling range.....2 C max from start to dry point, when tested by ASTM Method D 850, Test for Distillation of Industrial Aromatic Hydrocarbons and Related Materials,[7] or Method D 1078, Test for Distillation Range of Volatile Organic Liquids.[7] The range shall include the boiling point of pure *p*-xylene, which is 138.35 C (281.03 F).

Freezing point .....11.23 C, min (95 percent molal purity) as determined by ASTM Method D 1015, Test for Freezing Points of High-Purity Hydrocarbons.[7]

### A2.2 Procedure

A2.2.1 Determine the flash point of the *p*-xylene, following the directions in Sections 4 to 7. When the tester is operating properly, a value of 81 ± 1 F (27.2 ± 0.6 C) will be obtained.

A2.2.2 If the flash point obtained on *p*-xylene is not within the limits stated in A2.2.1, check the condition and operation of the apparatus to ensure conformity with the details listed in Appendix A1, especially with regard to tightness of the lid (A1.1.2.2), the action of the shutter and the position of the test flame (A1.1.2.3), and the angle and position of the thermometer (A1.1.2.4). After adjustment, if necessary, repeat the test, with special attention to procedural details prescribed in Section 6.

## A3. MANUFACTURING STANDARDIZATION

A3.1 The cup thermometer, which conforms also to the specifications for the low-range thermometer used in the Pensky-Martens flash tester, Method D 93, is frequently supplied by the thermometer manufacturer with a metal ferrule intended to fit the collar on the lid of the flash tester. This ferrule is frequently supplemented by an adapter which is used in the larger-diameter collar of the Pensky-Martens apparatus. Differences in dimensions of these collars, which are immaterial in their effect on the results of tests, are a source of considerable unnecessary trouble to manufacturers and suppliers of instruments, as well as to users.

A3.2 Subcommittee 21 on Metalware Laboratory Apparatus, of ASTM Committee E-1 on Methods of Testing, has studied this problem and has established some dimensional requirements which are shown, suitably identified, in Figs. A1, A3, and A4. Conformity to these requirements is not mandatory but is desirable to users as well as suppliers of Tag closed testers.

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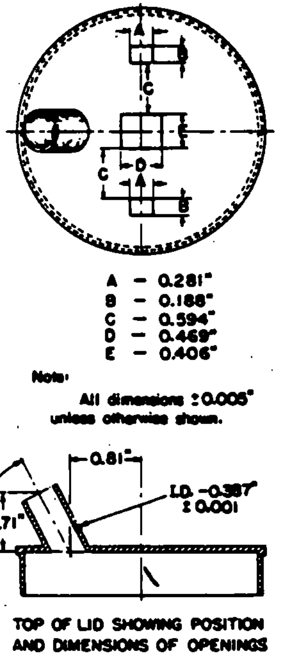
[6] Available as Flash Point Check Fluid (*p*-xylene) from Special Products Div., Phillips Petroleum Co., Bartlesville, Okla.

[7] *Annual Book of ASTM Standards*, Part 23.

TABLE A1 Dimensional Requirements

| | |
|---|---|
| Depth of bath liquid surface below top of test cup | 1.094 ± 0.016<br>(27.8 ± 0.4 mm) |
| Depth of sample surface below top of test cup | 1.156 + 0.031 in.<br>(29.4 ± 0.8 mm) |
| Depth of bottom of bulb of test thermometer below top of cup when in place | 1.77 ± 0.03 in.<br>(45.0 ± 0.8 mm) |
| Inside diameter of test cup at top | 2.125 ± 0.005 in.<br>(54.0 ± 0.1 mm) |
| Diameter of bead on top of cover | 0.156 ± 0.031 in.<br>(4.0 ± 0.8 mm) |
| Diameter of opening in tip of test flame nozzle | 0.049 ± 0.010 in.<br>(1.2 ± 0.3 mm) |
| Outside diameter of tip of test flame nozzle | 0.079 in. max<br>(2.0 mm max) |

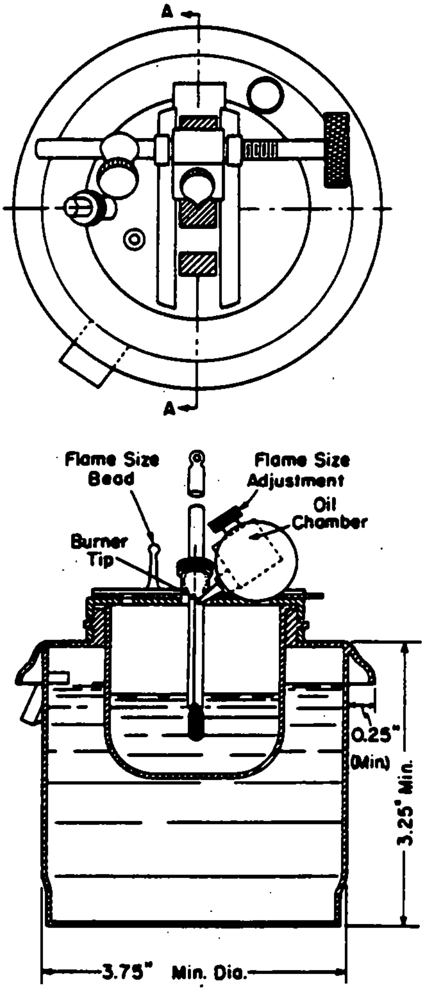


Metric Equivalents

| in. | mm | in. | mm |
|---|---|---|---|
| 0.001 | 0.03 | 0.406 | 10.32 |
| 0.005 | 0.13 | 0.469 | 11.92 |
| 0.188 | 4.78 | 0.594 | 15.10 |
| 0.281 | 7.15 | 0.71 | 18.0 |
| 0.387 | 9.84 | 0.81 | 20.6 |

NOTE—Dimensions relating to the size and position of the thermometer collar are recommended but not mandatory.

FIG. A1 Top of Lid Showing Position and Dimensions of Openings



Metric Equivalents

| | | | |
|---|---|---|---|
| in. | 0.25 | 3.25 | 3.75 |
| mm | 6.4 | 82.6 | 95.3 |

FIG. A2 Section of Liquid Bath and Cup. Metric Equivalents

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.1160 APPENDIX C.

Designation: D 3278 – 73

**AMERICAN SOCIETY FOR TESTING AND MATERIALS**

1916 Race St., Philadelphia, Pa., 19103

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## Standard Methods of Test for
# FLASH POINT OF LIQUIDS BY SETAFLASH CLOSED TESTER[1]

This Standard is issued under the fixed designation D 3278; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

### 1. Scope

1.1 This method covers the determination of the flash point, by Setaflash® Closed Tester, of paints, enamels, lacquers, varnishes, and related products and their components having flash points, between 32 and 230°F (0 to 110°C) having a viscosity lower than 150 stokes at 77°F (25°C).[2]

NOTE 1—Tests at higher or lower temperatures are possible.

1.2 The procedure may be used to determine whether a material will or will not flash at a specified temperature or to determine the finite temperature at which a material will flash.

1.3 The results from this method are comparable to those obtained by the Tag Closed Tester procedure described in Method D 563 and the Pensky-Martens Tester method described in Method D 93.

### 2. Applicable Documents

2.1 *ASTM Standards:*

D 56 Test for Flash Point by Tag Closed Tester[2]

D 93 Test for Flash Point by Pensky-Martens Closed Tester[3]

D 850 Test for Distillation of Industrial Aromatic Hydrocarbons and Related Materials[2]

D 1015 Test for Freezing Points of High-Purity Hydrocarbons[3]

D 1078 Test for Distillation Range of Volatile Organic Liquids[2]

### 3. Summary of Method

3.1 By means of a syringe, 2 ml of sample is introduced through a leakproof entry port into the tightly closed Setaflash Tester or directly into the cut that has been brought to within 5°F (3°C) below the expected flash point. As a flash/no flash test, the *expected* flash point temperature may be a specification or other operating requirements. The temperature of the apparatus is raised to the precise temperature of the expected flash point by slight adjustment of the temperature dial. After 1 min, a test flame is applied inside the cup and note is taken as to whether the test sample flashes or not. If a repeat test is necessary, a fresh sample should be used.

3.2 For a finite flash measurement, the temperature is sequentially increased through the anticipated range, the test flame being applied at 9°F (5°C) intervals until a flash is ovserved. A repeat determination is then made using a fresh sample, starting the test at the temperature of the last interval before the flash point of the material and making tests at increasing 1°F (0.5°C) intervals.

### 4. Apparatus

4.1 *Setaflash Tester*[4], shown in Fig. X1, and described in Appendix X1.

4.2 *Thermometers*[5] conforming to specifica-

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[1] These methods are under the jurisdiction of ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

Current edition approved Oct. 29, 1973. Published December 1973.

[2] *1974 Annual Book of ASTM Standards*, Part 29.

[3] *1973 Annual Book of ASTM Standards*, Part 18.

[4] Unit shown in Fig. X1 is manufactured by Stanhope-Seta Ltd., Park Close, Egham, Surrey, England. It is available in the USA from Erdco Engineering Corp., 136 Official Road, Addison, Ill. 60101, or from Paul N. Gardner Co., Station 9, P. O. Box 6633, Fort Lauderdale, Fla. 33316.

[5] Thermometers may be obtained from the suppliers of the Setaflash.

tions given in Table X1. Test to determine that the scale error does not exceed 0.5°F (0.25°C). The use of a magnifying lens significantly assists in making temperature observations.

4.3 *Glass Syringe*, 2 ± 0.1-ml capacity at 77°F (25°C), to provide a means of taking a uniform sample. Check the capacity by discharging water into a weighing bottle and weighing. Adjust plunger if necessary. A disposable syringe of equal precision may be used.

4.4 *Cooling Block*, aluminum (described in Appendix X2) which fits snugly within the test cup for rapid cooling of the sample cup.

4.5 *Barometer*.

### 5. Reagents and Materials

5.1 *p-Xylene*[6]—Reference standard for checking the Setaflash Tester.

5.2 *Cooling Mixture* of ice water or dry ice (solid $CO_2$) and acetone.

5.3 *Liquefied Petroleum Gas*.

5.4 *Heat Transfer Paste*[7]

### 6. Sampling

6.1 The sample size for each test is 2 ml. Obtain at least a 25-ml sample from the bulk source and store in a nearly full tightly closed clean glass container or in other container suitable for the type of liquid being sampled.

6.2 Erroneously high flash points may be obtained if precautions are not taken to avoid loss of volatile material. Do not open sample containers unnecessarily and do not transfer the sample to the cup unless its temperature is at least 20°F (10°C) below the expected flash point. Discard samples in leaky containers.

### 7. Preparation of Apparatus

7.1 Prior to initial use or after removal of the thermometer, insert the thermometer into its pocket, Fig. X2, with a good heat transfer paste.

7.2 To help in making the necessary settings during a test, determine the relationship between the temperature control dial and thermometer readings at intervals not over 10°F (5°C) throughout the scale range of heater before the initial use.

7.3 Place the tester in a subdued light and in a position where it is not exposed to disturbing drafts. Provide a black-coated shield, if necessary.

7.4 Read the manufacturer's operating and maintenance instructions on the care and servicing of the tester. Observe the specific suggestions regarding the operation of its various controls.

7.5 Check the accuracy of the tester by determining the flash point of the *p*-xylene reference standard in duplicate (Appendix X3). The average of the results should be 81 ± 1.5°F (27.2 ± 0.8°C). If not, remove the thermometer and observe whether sufficient heat transfer paste surrounds the thermometer to provide good heat transfer from the cup to the thermometer.

## METHOD A—FLASH/NO FLASH

### 8. Procedure—Ambient to 230°F (110°C)

8.1 Inspect the inside of the test cup, lid, and shutter mechanism for cleanliness and freedom from contamination. Use an absorbent tissue to wipe clean, if necessary. Lock the cover lid tightly in place.

8.2 Switch the tester on, if not already at stand-by. To rapidly approach the specification flash temperature of the charged sample, turn the heater dial fully clockwise (Note 2) causing the heater signal (red) light to glow. When the thermometer indicates a temperature of about 5°F (3°C) below the specification or target flash point temperature, reduce the heat input to the test cup by slowly turning the heater control dial counter clockwise until the signal light goes out (Note 3).

NOTE 2—When the correct temperature is dialed on the temperature controller, the elapsed time to reach it may be greater than when turned Full On, but less attention will be required in the intervening period.

NOTE 3—The test cup temperature is stable when the signal light slowly cycles on and off.

8.3 Determine the barometric pressure to determine the corrected specification temperature at that barometric pressure (see 13.2).

8.4 After the test cup temperature has stabilized at the specification or target flash point, charge the syringe with the sample to be tested and transfer the syringe to the filling orifice

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[6] *P*-xylene is available as "Flash Point Check Fluid" from Special Products Div., Phillips Petroleum Co., Bartlesville, Okla.

[7] Heat transfer paste is available from the supplies of the Setaflash Tester. Dow Corning also can supply a similar paste as their no 340 silicone.

(Fig. X2) taking care not to lose any sample. Discharge the sample into the test cup by depressing the syringe plunger to its lowest position, then remove the syringe. If the sample has a viscosity greater than 45 SUS at 100°F (37.8°C) or equivalent of 9.5 cSt at 77°F (25.0°C), discharge the contents of the syringe directly into the cup. Immediately close tightly the lid and shutter assembly.

8.5 Set the 1-min timing device by rotating its knob clockwise to the required setting. In the meantime, open the gas control valve and light the pilot and the test flames. Adjust the test flame size with the pinch valve so as to match the size of the $\frac{5}{32}$ in. (4-mm) diameter flame gage.

8.6 After 1 min has elapsed, observe the temperature. If at the specification temperature (accounting for the differences of the barometer reading from 760 mm), apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of approximately 2½ s. Watch for a flash.

NOTE 4—The sample is considered to have flashed only if a comparatively large blue flame appears and propagates itself over the surface of the liquid. Occasionally, particularly near the actual flash point temperature, application of the test flame may give rise to a halo; this should be ignored.

8.7 Turn off the test and the pilot flame. Clean the apparatus in preparation for the next test.

### 9. Procedure—32°F (0°C) to Ambient

9.1 If the specification or target flash point is at or below ambient temperature, cool the sample to 10 to 20°F (5 to 10°C) below that point by some convenient means.

9.2 Cool the tester to approximately the temperature of the sample by inserting the cooling block (Appendix X1.2) filled with a cooling mixture (Notes 5 and 6) into the sample well. Dry the cup with a paper tissue to remove any collected moisture prior to adding the sample.

NOTE 5: Caution—Be careful in handling the cooling mixture and cooling block; wear gloves and goggles. Mixtures such as dry ice and acetone can produce severe frost bite.

NOTE 6: Caution—Be careful in inserting the cooling block into the tester cup to prevent damage to the cup.

9.3 Introduce the sample as in 8.4. Allow the temperature to rise under ambient conditions or increase the temperature of the cup by rotating the heater controller clockwise slowly until the specification temperature adjusted for barometric pressure is reached. Determine whether the sample flashes as in 8.5 and 8.6.

9.4 Turn off the test and pilot flames. Clean up the apparatus.

## METHOD B—FINITE FLASH POINT

### 10. Procedure—Ambient to 230°F (110°C)

10.1 *Preliminary or Trial Test*—Follow steps 8.1 to 8.5 omitting the barometric reading and using an estimated finite flash point instead of a specification flash point temperature.

10.2 After 1 min has elapsed, observe the temperature, apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of 2½ s. Watch for a flash (Note 3).

10.3 *Finite Flash Point*—If a flash is observed proceed as below.

10.3.1 Using a temperature of 9°F (5°C) lower than the temperature observed in 10.2, repeat 10.1 and 10.2 (Note 6). If a flash is still observed, repeat at 9°F (5°C) lower intervals until no flash is observed.

NOTE 7—Never make a repeat test on the same sample. Always take a fresh portion for each test.

10.3.2 Repeat 10.1 and 10.2 with a new sample, stabilizing the test cup temperature at the temperature at which no flash occurred previously. Observe if a flash occurs at this temperature. If no flash occurs, increase the temperature at 1°F (0.5°C) intervals by making small incremental adjustment to the temperature controller and allowing 1-min intervals between each increment and the flash point test. Record the temperature at which the flash acutally occurs. Record the barometric pressure. Turn off pilot and test flames and clean up tester.

10.4 *Finite Flash Point*—If no flash point is observed in 10.2, proceed as follows:

10.4.1 Using a test temperature of 9°F (5°C) higher than the temperature observed in 10.2, repeat steps 10.1 and 10.2 (Note 7). If no flash is observed, repeat at 9°F (5°C) higher intervals until a flash is observed.

10.4.2 Repeat step 10.3.2 with a new sample.

## 11. Procedure—32°F (0°C) to Ambient Temperature

11.1 *Preliminary or Trial Test*—Cool the sample to 5 to 10°F (3 to 5°C) below the expected flash point.

11.2 Cool the tester to approximately the temperature of the sample by inserting the cooling block filled with a cooling medium, into the sample well (Notes 4 and 5).

11.3 Insert the sample as in 8.4. Set the 1-min timing device. After 1 min, apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of approximately 2½ s. Observe for a flash (Note 3). Record the temperature.

11.4 *Finite Flash Point*—If a flash is observed, proceed as follows:

11.4.1 Cool a new sample and the sample cup to 9°F (5°C) below the previous temperature (11.3). After 1 min, check for a flash as in 11.3. If the sample flashes, repeat test at 9°F (5°C) lower intervals until no flash is observed.

11.4.2 Repeat with a new sample, cooling both sample and tester to the temperature at which the sample did not flash. After 1 min, observe if a flash occurs at this temperature, if not, increase the temperature at 1°F (0.5°C) intervals by making small incremental adjustments to the temperature controller, allowing 1 min between each increment and the test for the flash point. Record the temperature at which the flash actually occurs. Record the barometric pressure.

11.5 *Finite Flash Point*—If no flash point is observed proceed as follows:

11.5.1 Using a test temperature of 9°F (5°C) higher than the temperature observed in 11.3, repeat step 11.3 (Note 6). If no flash is observed, repeat at 9°F (5°C) higher intervals until flash is observed.

11.5.2 Using a new sample, repeat 11.4.2 until a flash occurs. Record the temperature at which the flash occurs and the barometric pressure.

## 12. Clean Up Of Apparatus and Preparation for Next Test

12.1 To prepare for the next test, unlock the lid assembly of the tester and raise to the hinge stop. Soak up liquid samples with an absorbent paper tissue and wipe dry. Clean the underside of the lid and filling orifice. A pipe cleaner may be of assistance in cleaning the orifice.

12.2 If the sample is a viscous liquid or contains dispersed solids, after soaking up most of the sample, add a small amount of a suitable solvent for the sample to the cup and then soak up the solvent and wipe clean the interior surfaces of the cup with an absorbent tissue paper.

NOTE 8—If necessary to remove residual high boiling solvent residues, moisten tissue with acetone and wipe clean.

NOTE 9—If any further cleaning is necessary, remove the lid and shutter assembly. Disconnect the silicone rubber hose and slide the lid assembly to the right to remove. If warm, handle carefully.

12.3 After the cup has been cleaned, its temperature may be rapidly increased to some stand-by value by turning the temperature control dial to an appropriate point.

NOTE 10—It is convenient to hold the test cup at some stand-by temperature (depending on planned usage) to conserve time in bringing the cup within the test temperature range. The cup temperature may be quickly lowered by inserting the aluminum cooling block filled with an appropriate cooling mixture into the cup.

12.4 The syringe is easily cleaned by filling it several times with acetone or any compatible solvent, discharging the solvent each time, and allowing the syringe to air dry with the plunger removed. Replace the plunger, and pump several times to replace any solvent vapor with air.

## 13. Correction for Barometric Pressure

13.1 When the barometric pressure differs from 760 mm Hg (101.3 kPa), calculate the flash point temperature by means of the following equations:

$$\text{Calculated flash point} = F + 0.06\,(760 - P)$$
$$= C + 0.03\,(760 - P)$$

where:

$F, C$ = observed flash point, °F (or °C), and
$P$ = barometric pressure, mm Hg.

13.2 Likewise determine the corrected specification flash point by the following equation:

$$F = S - 0.06\,(760 - P)$$
$$C = S - 0.03\,(760 - P)$$

where:

$F, C$ = flash point to be observed to obtain the specification flash point at standard pressure ($S$),
$S$ = specification flash point.

## 14. Report

14.1 When using the flash/no flash method, report whether the sample flashed at the required flash point and that the flash/no flash method was used.

14.2 If an actual flash point was determined, report the average of duplicate runs to nearest 1°F (0.5°C) provided the difference between the two values does not exceed 2°F (1°C).

## 15. Precision[8]

15.1 The following criteria should be used for judging the acceptability of results (95 % confidence):

15.1.1 Liquids at or below 45 SUS at 100°F or equivalent viscosity measurements.

15.1.1.1 *Repeatability*—The average of duplicate results obtained by the same operator on different days should be considered suspect if they differ by more than 3°F (1.7°C).

15.1.1.2 *Reproducibility*—The average of duplicate results, obtained by each of two laboratories should not be considered suspect unless they differ by more than 6°F (3.3°C).

15.1.2 Viscous liquids above 45 SUS at 100°F or liquids with dispersed solids.

15.1.2.1 *Repeatability*—Duplicate results obtained by the same operator on different days should be considered suspect if they differ by more than 6°F (3.3°C).

15.1.2.2 *Reproducibility*—The average of duplicate results obtained by each of two laboratories should not be considered suspect unless they differ by more than 9°F (5°C).

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[8] Supporting data for this method has been filed at ASTM Headquarters RR 0-1-1000 and reported in Journal of Paint Technology, Vol 45, No. 581 Page 44.

# APPENDIXES

## X1. APPARATUS SPECIFICATIONS

X1.1 A typical apparatus is shown in Fig. X1 and X2. Electrical heaters are fastened to the cup in such a way so as to provide for efficient transfer of heat. The tester includes a variable heater control device with a scaled dial and a visible signal to indicate when energy is or is not being applied. Energy may be supplied from a 115 or 230-V a-c main service (for stationary use) or by a 12-V d-c battery service (for field use). A regulatable test flame and a pilot flame to maintain the test flame, are provided. These flames may be fueled by piped gas service (fixed location) or by a self-contained tank of liquefied petroleum gas (5.3) (for portability). A test flame, 5/32 in. (4 mm) in diameter, is provided against which the size of the flame may be judged. Never recharge the gas tank with the pilot or test flames lighted, nor in the vicinity of other naked flames. A 1-min audible signal is a desirable accessory.

## X2. COOLING BLOCK

X2.1 The cooling block with dimensions as shown in Fig. X3, is made of aluminum and covered with pipe insulation.

## X3. SPECIFICATIONS FOR *P*-XYLENE REFERENCE STANDARD

X3.1 *Specific Gravity (60/60°F) (15.6/15.6°C)*—0.860 min, 0.866 max.

X3.2 *Boiling Range*—2°C max from start to dry point, when tested by Method D 850 or Method D 1078. The range shall include the boiling point of pure *p*-xylene, which is 138.35°C (281.03°F).

X3.3 *Freezing Point*—11.23°C min (95 % molal purity) as determined by Method D 1015.

TABLE X1 Setaflash® Thermometers

| ASTM No., Name | XXF (XXC), Setaflash Medium | XXF (XXC) Setaflash Low |
|---|---|---|
| Range | 32 to 230°F (0 to 110°C) | −10°C – 160°<br>(−23 to 70°C) |
| Immersion | 44.5 ± 1 mm | 44.5 ± 1 mm |
| Graduations: | | |
| Subdivisions | 1 ± F (1°C) | 1°F (1°C) |
| Long lines at each | 10°F (10°C) | 10°F (10°C) |
| Number at each[a] | 10°F (10°C) | 10°F (10°C) |
| Scale error, max | 0.5°F (0.25°C) | 0.5°F (0.25°C) |
| Expansion chamber, for heat to[b] | 248°F (120°C) | 176°F (80°C) |
| Total length | 204 ± 3 mm | 204 ± 3 mm |
| Stem, OD | 6 to 7 mm | 6 to 7 mm |
| Bulb: | | |
| Length | 11.7 to 13.7 mm | 11.7 to 13.7 mm |
| OD | 4.7 to 5.7 mm | 4.7 to 5.7 mm |
| Scale location: | | |
| Bottom of bulb to 32°F (0°C) | 49 to 51 mm | |
| Bottom of bulb to −10°F (−23°C) | . . . | 59 mm to 61 mm |
| Bottom of bulb to 230°F (110°C) | 163 to 176 mm | . . . |
| Bottom of bulb to 160°F (71°C) | . . . | 183 to 185 mm |

[a] Number so that figures are read from right to left in a horizontal plane.

[b] Expansion bulb to be nitrogen filled for horizontal operation.

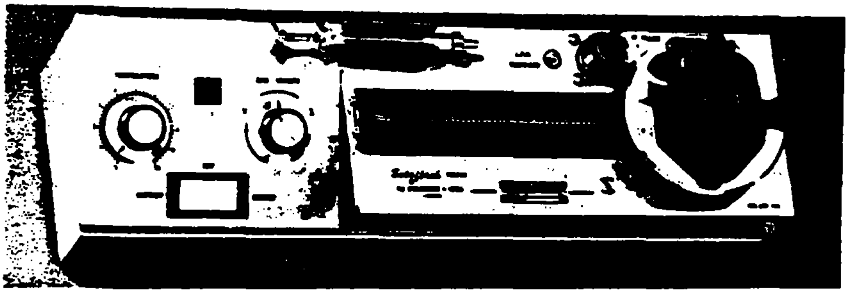

FIG. X1 Setaflash Tester.

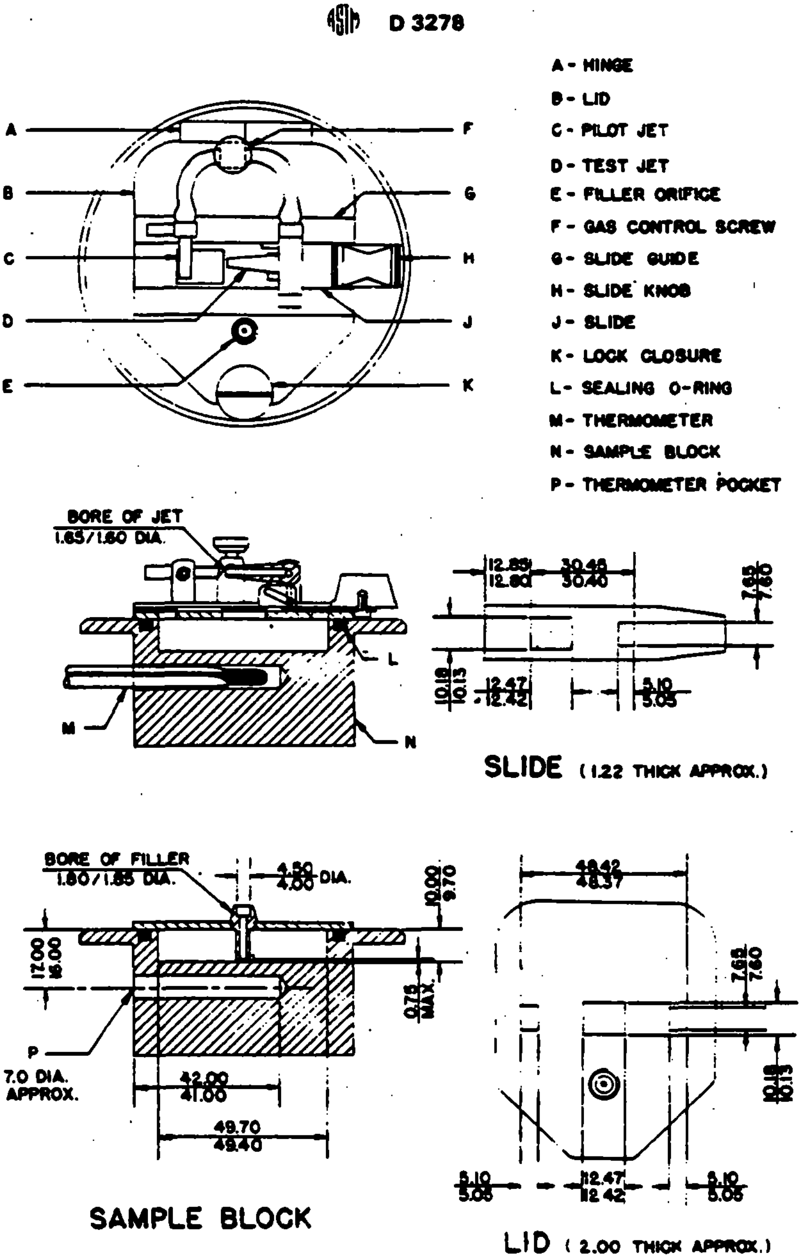

FIG. X2 Setaflash Unit.

D 3278

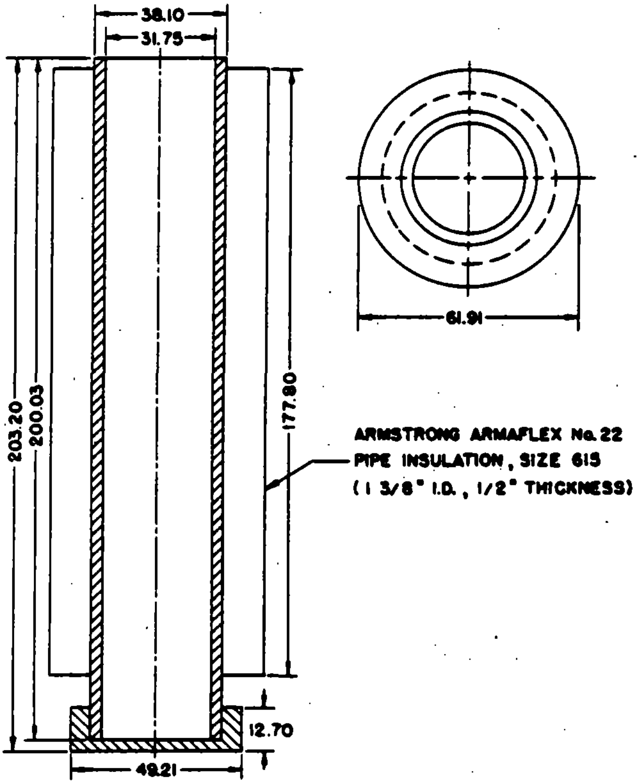

FIG. 3 Cooling Block

**Statutory Authority:** *MS s 116.07 subd 4*

7045.1170 APPENDIX D.

**Designation: D 93 – 73** **IP Designation: 34/71**

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American National Standard Z11.7
American National Standards Institute
Method 1102—Federal Test
Method Standard No. 791b
Federation of Societies for
Paint Technology Standard No. Dt-8-66
British Standard 2839

## Standard Method of Test for
# FLASH POINT BY PENSKY-MARTENS CLOSED TESTER[1]

ADOPTED (as method GO-7), 1924; LAST REVISED, 1971

This Standard of the American Society for Testing and Materials is issued under the fixed designation D 93; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. This is also a standard of the Institute of Petroleum issued under the fixed designation IP 34. The final number indicates the year of last revision.

*This method was adopted as a joint ASTM-IP Standard in 1967.*

### 1. Scope

1.1 This method covers the determination of the flash point by Pensky-Martens Closed Cup Tester of fuel oils, lube oils, suspensions of solids, liquids that tend to form a surface film under test conditions, and other liquids. For the determination of the flash point of drying oils and solvent-type waxes refer to Note 1.

NOTE 1—The flash point of drying oils may be determined using Method D 1393 and the flash point of solvent-type liquid waxes may be determined using Method D 1437.

NOTE 2—This method may be employed for the detection of contamination of lubricating oils by minor amounts of volatile materials.

### 2. Applicable Documents

2.1 *ASTM Standards:*

D 56 Test for Flash Point by Tag Closed Tester[2]

D 1310 Test for Flash Point of Liquids by Tag Open-Cup Apparatus[2]

D 1393 Test for Flash Point of Drying Oils[3]

D 1437 Test for Flash Point of Solvent-Type Liquid Waxes[4]

E 1 Specification for ASTM Thermometers[5]

### 3. Summary of Method

3.1 The sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite.

### 4. Apparatus

4.1 *Pensky-Martens Closed Flash Tester*, as described in Appendix A1.

NOTE 3—There are automatic flash point testers available and in use which may be advantageous in the saving of testing time, permit the use of smaller samples, and have other factors which may merit their use. If automatic testers are used, the user must be sure that all of the manufacturer's instructions for calibrating, adjusting, and operating the instrument are followed. In any cases of dispute, the flash point as determined manually shall be considered the referee test.

4.2 *Thermometers*—Two standard thermometers shall be used with the ASTM Pensky-Martens tester, as follows:

4.2.1 For tests in which the indicated reading falls within the limits 20 to 200 F (−7 to +93 C), inclusive, an ASTM Pensky-Martens Low Range or Tag Closed Tester Thermometer having a range from 20 to 230 F (−5 to +110 C) and conforming to the requirements for Thermometers 9F (9C) and as prescribed in ASTM Specification E 1 or IP Thermometer 15F (15C) conforming to specifications given in Appendix A3, shall be used.

3.2.2 For tests in which the indicated reading falls within the limits 230 to 700 F (110 to 371 C), an ASTM Pensky-Martens High

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[1] This method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants.
Current edition approved Aug. 27, 1973. Published October 1973. Originally published as D 93 – 21 T. Last previous edition D 93 – 72.
In the IP, this method is under the jurisdiction of the Standardization Committee.
In 1971 the scope was revised.

[2] *Annual Book of ASTM Standards*, Part 20.

[3] *Annual Book of ASTM Standards*, Part 11.

[4] *Annual Book of ASTM Standards*, Part 22.

[5] *Annual Book of ASTM Standards*, Parts 18 and 30.

Range Thermometer having a range from 200 to 700 F (90 to 370 C) and conforming to the requirements for Thermometers 10F (10C) as prescribed in Specification E 1 or IP Thermometer 16F (16C) conforming to specifications given in Appendix A3, shall be used.

4.2.3 For the range 200 to 230 F (93 to 110 C) either thermometer may be used.

### 5. Preparation of Apparatus

5.1 Support the tester on a level, steady table. Unless tests are made in a draft-free room or compartment, it is good practice, but not required, to surround the tester on three sides with a shield, each section of which is about 18 in. (46 cm) wide and 24 in. (61 cm) high.

### 6. Preparation of Sample

6.1 Samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than is absolutely necessary. It shall never be heated above a temperature of 30 F (16 C) below its expected flash point.

6.2 Samples containing dissolved or free water may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Warming the sample is permitted, but it shall not be heated for prolonged periods or above a temperature of 30 F (16 C) below its expected flash point.

NOTE 4—If the sample is suspected of containing volatile contaminants, the treatment described in 6.1 and 6.2 should be omitted.

### 7. Procedure

7.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent which had been used to clean the apparatus. Fill the cup with the sample to be tested to the level indicated by the filling mark. Place the lid on the cup and set the latter in the stove. Be sure to have the locating or locking device properly engaged. Insert the thermometer. Light the test flame and adjust it to $^{5}/_{32}$ in. (4 mm) in diameter. Supply the heat at such a rate that the temperature as indicated by the thermometer increases 9 to 11 F (5 to stirring in a downward direction.

7.2 If the sample is known to have a flash point of 220 (104 C) or below, apply the test flame when the temperature of the sample is from 30 F (17 C) to 50 F (28 C) below the expected flash point and thereafter at a temperature reading that is a multiple of 2 F (1 C). Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cup in 0.5 s, left in its lowered position for 1 s, and quickly raised to its high position. Do not stir the sample while applying the test flame.

7.3 If the sample is known to have a flash point above 220 F (104 C) apply the test flame in the manner just described at each temperature that is a multiple of 5 F (3 C), beginning at a temperature of 30 F (17 C) to 50 F (28 C) below the expected flash point.

NOTE 5—When testing materials to determine if volatile contaminants are present, it is not necessary to adhere to the temperature limits for initial flame application as stated in 7.2 and 7.3.

7.4 Record as the flash point the temperature read on the thermometer at the time the test flame application causes a distinct flash in the interior of the cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash.

## DETERMINATION OF FLASH POINT OF SUSPENSIONS OF SOLIDS

### 8. Procedure

8.1 Bring the material to be tested and the tester to a temperature of 60 ± 10 F (15 ± 5 C) or 20 F (11 C) lower than the estimated flash point, whichever is lower. Completely fill the air space between the cup and the interior of the air bath with water at the temperature of the tester and sample. Turn the stirrer 250 ± 10 rpm, stirring in a downward direction. Raise the temperature throughout the duration of the test at a rate of not less than 2 nor more than 3 F (1 to 1.5 C)/min. With the exception of these requirements for rates of stirring and heating, proceed as prescribed in Section 7.

NOTE 6—Solid carbon dioxide ($CO_2$) (dry ice) shall in no case be used to obtain the proper rate of temperature rise, since $CO_2$ has a blanketing effect which leads to a false flash point.

## 9. Calculation and Report

9.1 Observe and record the barometric pressure at the time of the test. When the pressure differs from 760 mm Hg, correct the flash point by means of the following equations:

$$\text{Corrected flash point} = F + 0.06\,(760 - P)$$

or

$$\text{Corrected flash point} = C + 0.03\,(760 - P)$$

where:

$F$ = observe flash point, deg F.

$C$ = observe flash point, deg C.

$P$ = barometric pressure, mm Hg.

9.2 Record the corrected flash point to the nearest 1 F or 0.5 C.

9.3 Report the recorded flash point as the Pensky-Martens Closed Cup Flash Point, ASTM D 93 - IP 34, of the sample tested.

## 10. Precision

10.1 The following criteria should be used for judging the acceptability of results (95 percent probability):

10.1.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

| Material | Flash Point Range | Repeatability |
|---|---|---|
| Suspensions of solids | 95 to 110 F<br>(35 to 43.3 C) | 4 F<br>(2.0 C) |
| All others | 220 F (104.4 C) and under | 4 F<br>(2.0 C) |
| | Above 220 F<br>(104.4 C) | 10 F<br>(5.5 C) |

10.1.2 *Reproducibility*—The results submitted by each of two laboratories should be considered suspect if the two results differ by more than the following amounts:

| Material | Flash Point Range | Reproducibility |
|---|---|---|
| Suspensions of solids | 95 to 110 F<br>(35 to 43.3 C) | 6 F<br>(3.5 C) |
| All others | 220 F (104.4 C) and under | 6 F<br>(3.5 C) |
| | Above 220 F<br>(104.4 C) | 15 F<br>(8.5 C) |

10.2 The following criteria should be used for judging the acceptability of results (95 percent confidence) obtained on viscous and/or heavily pigmented (paint or varnish) materials which tend to form a surface film:

10.2.1 *Repeatability*—The average of two tests by the same operator on the same day compared to two tests on another day should be considered suspect if they differ by more than 9 F (5 C).

10.2.2 *Reproducibility*—The average of two tests by an operator on the same day compared to the average of two tests by another operator (or another laboratory) on any one day should be considered suspect if they differ by more than 18 F (10.0 C).

10.2.3 The definition of Repeatability and Reproducibility given here represents different parameters of the variance of the method; those given in 10.1 are derived from standards of ASTM Committee D-2 and the Institute of Petroleum, while those in 10.2 are from ASTM Committee D-1.

## APPENDIXES

### A1. APPARATUS SPECIFICATIONS

A1.1 A typical assembly of the apparatus, gas heated, is shown in Fig. A1. The apparatus shall consist of a test cup, cover, and stove conforming to the following requirements:

A1.1.1 *Cup*—The cup shall be of brass, or other nonrusting metal of equivalent heat conductivity, and shall conform to the dimensional requirements in Fig. A2. The flange shall be equipped with devices for locating the position of the cup in the stove. A handle attached to the flange of the cup is a desirable accessory. The handle shall not be so heavy as to tip over the empty cup.

A1.1.2 *Cover:*

A1.1.2.1 *Cover Proper*—The cover shown in Fig. A3 shall be of brass (A1.1.1.1), and shall have a rim projecting downward almost to the flange of the cup. The rim shall fit the outside of the cup with a clearance not exceeding 0.014 in. on the diameter. There shall be a locating or locking device, or both engaging with a corresponding device on the cup. The four openings in the cover, *A*, *B*, *C*, and *D*, are shown in Fig. A3. The upper edge of the cup shall be in close contact with the inner face of the cover throughout its circumference.

A1.1.2.2 *Shutter*—The cover shall be equipped with a brass (Section 3) shutter (Fig. A4), approximately $^{3}/_{32}$ in. thick, operating on the plane of the upper surface of the cover. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal center of the cover between two stops, so placed, that when in one extreme position, the *openings A, B, and C in the* cover are completely closed, and when in the other extreme position, these openings are completely opened. The mechanism operating the shutter should be of the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme, the three cover openings shall be exactly open and the tip of the exposure tube shall be fully depressed.

A1.1.2.3 *Flame-Exposure Device*—The flame-exposure device (Fig. A4) shall have a tip with an opening 0.027 to 0.031 in. in diameter. This tip shall be made preferably of stainless steel, although it may be fabricated of other suitable metals. The flame-exposure device shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the center of the orifice is between the planes of the under and upper surfaces of the cover proper at a point on a radius passing through the center of the larger opening A (Fig. A3).

A1.1.2.4 *Pilot Flame*—A pilot flame shall be provided for automatic relighting of the exposure flame. A bead 5/32 in. in diameter may be mounted on the cover so that the size of the test flame can be regulated by comparison. The tip of the pilot flame shall have an opening the same size as the tip of the flame exposure device (0.027 to 0.031 in. in diameter).

A1.1.2.5 *Stirring Device*—The cover shall be equipped with a stirring device (Fig. A4) mounted in the center of the cover and carrying two 2-bladed metal propellers. A stirrer shaft may be coupled to the motor by a flexible shaft or a suitable arrangement of pulleys.

A1.1.3 *Stove*—Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. The stove shall consist of an air bath and a top plate on which the flange of the cup rests.

A1.1.3.1 *Air Bath*—The air bath shall have a cylindrical interior and shall conform to the dimensional requirements in Fig. A1. The air bath may be either a flame or electrically heated metal casting (Note A1), or an electric-resistance element (Note A2). In either case, the air bath must be suitable for use at the temperatures to which it will be subjected without deformation.

NOTE A1—If the heating element is a flame or electrically heated metal casting, it shall be so designed and used that the temperatures of the bottom and the walls are approximately the same. On this account it should be not less than 1/4 in. in thickness. The casting shall be designed so that products of combustion of the flame cannot pass up and come into contact with the cup.

NOTE A2—If the air bath is of the electric-resistance heated type, it shall be constructed so that all parts of the interior surface are heated uniformly. The wall and bottom of the air bath shall be not less than 1/4 in. in thickness.

A1.1.3.2 *Top Plate*—The top plate shall be of metal, and shall be mounted with an air gap between it and the air bath. It may be attached to the air bath by means of three screws and spacing bushings. The bushings should be of proper thickness to define an air gap of 3/16 in., and they shall be not more than 3/8 in. in diameter.

## A2. MANUFACTURING STANDARDIZATION OF THERMOMETER AND FERRULE

A2.1 The low-range thermometer, which conforms also to the specification for the cup thermometer in the Tag closed tester (Method D 56) and which frequently is fitted with a metal ferrule intended to fit the collar on the cover of the Tag flash tester, can be supplemented by an adapter (Fig. A5) to be used in the larger diameter collar of the Pensky-Martens apparatus. Differences in dimensions of these collars, which do not affect test results, are a source of unnecessary trouble to manufacturers and suppliers of instruments, as well as to users.

A2.2 Subcommittee 21 on Metalware Laboratory Apparatus, of ASTM Committee E-1 on Methods of Testing, has studied this problem and has established some dimensional requirements which are shown in Fig. A5. Conformity to these requirements is not mandatory, but is desirable to users as well as suppliers of Pensky-Martens Testers.

D 93 — 34

## A3. THERMOMETER SPECIFICATIONS

TABLE A1 IP Thermometer Specifications

NOTE—The stem shall be made with an enlargement having a diameter of 1.5 to 2.0 mm greater than the stem and a length of 3 to 5 mm, the bottom of the enlargement being 64 to 66 mm from the bottom of the bulb. These dimensions shall be measured with the test gage shown in Fig. 1 of Specification E 1.[1]

| Name | IP 15F | IP 15C | IP 16F | IP 16C |
|---|---|---|---|---|
| | Pensky-Martens Low | | Pensky-Martens High | |
| Range | 20 to 230 F | −7 to +110 C | 200 to 700 F | 90 to 370 C |
| Graduation | 1 F | 0.5 C | 5 F | 2 C |
| Immersion, mm | 57 | 57 | 57 | 57 |
| Over-all length ±10 mm | 280 | 280 | 280 | 280 |
| Stem diameter, mm | 5.5 to 8.0 | 5.5 to 8.0 | 5.5 to 8.0 | 5.5 to 8.0 |
| Bulb shape | cylindrical | cylindrical | cylindrical | cylindrical |
| Bulb length, mm | 9 to 13 | 9 to 13 | 10 max | 10 max |
| Bulb diameter, mm | not less than 5.5 and not greater than stem | not less than 5.5 and not greater than stem | not less than 5.5 and not greater than stem | not less than 5.5 and not greater than stem |
| Length of graduated portion, mm | 143 to 177 | 143 to 177 | 143 to 177 | 143 to 177 |
| Distance bottom of bulb to, mm | 20 F<br>75 to 90 | −7 C<br>75 to 90 | 200 F<br>75 to 90 | 90 C<br>75 to 90 |
| Longer lines at each | 5 F | 1 C and 5 C | 25 F | 10 and 20 C |
| Figured at each | 10 F | 5 C | 50 F | 20 C |
| Expansion chamber | Required | Required | Required | Required |
| Top finish | Ring | Ring | Ring | Ring |
| Scale error not to exceed ± | 1 F | 0.5 C | 2.5 to 500 C<br>3.5 F above 500 F | 1 to 260 C<br>2 C above 260 C |
| See notes | 1 and see table for emergent stem temperatures | 1 and see table for emergent stem temperatures | 1 and see table for emergent stem temperatures | 1 and see table for emergent stem temperatures |

TABLE A3 Standardization Temperatures

NOTE—The emergent column temperatures are those attained when using the thermometers in the test equipment for which the thermometers were originally designed. In some cases these temperatures are markedly different from those realized during standardization.

| Temperature | Average Temperature of Emergent Column | Temperature | Average Temperature of Emergent Column | Temperature | Average Temperature of Emergent Column | Temperature | Average Temperature of Emergent Column |
|---|---|---|---|---|---|---|---|
| Thermometer 9F (20 to 230 F) | | Thermometer 9C (−5 to +100 C) | | Thermometer 10F (200 to 700 F) | | Thermometer 10C (90 to 370 C) | |
| 32 F | 66 F | 0 C | 19 C | 212 F | 141 F | 100 C | 61 C |
| 100 F | 86 F | 35 C | 28 C | 390 F | 159 F | 200 C | 71 C |
| 160 F | 106 F | 70 C | 40 C | 570 F | 180 F | 300 C | 87 C |
| 220 F | 123 F | 105 C | 50 C | 700 F | 220 F | 370 C | 104 C |
| IP 15F (20 to 230 F) | | IP 15C (−7 to 110 C) | | IP 16F (20 to 700 F) | | IP 16C (90 to 370 C) | |
| 32 F | 66 F | 0 C | 19 C | 200 F | 140 F | 100 C | 61 C |
| 70 F | 70 F | 20 C | 20 C | 300 F | 149 F | 150 C | 65 C |
| 100 F | 86 F | 40 C | 31 C | 400 F | 160 F | 200 C | 71 C |
| 150 F | 104 F | 70 C | 40 C | 500 F | 175 F | 250 C | 78 C |
| 212 F | 118 F | 100 C | 48 C | 600 F | 195 F | 300 C | 87 C |
| | | | | 700 F | 220 F | 350 C | 99 C |

TABLE A2 Specifications for ASTM Thermometers
All dimensions are in millimeters.
See Table A3 for Standardization Temperatures.

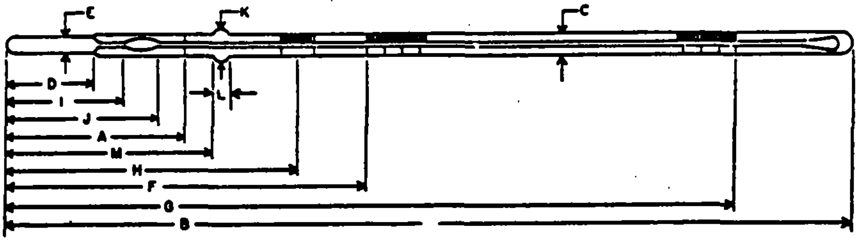
| ASTM No. and Name | Range | For Test at | Immersion | Graduations | | | Scale Error max | Special Inscription | Expansion Chamber |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Subdivisions | Long Lines at Each | Number at Each | | | Permit Heating to |
| 9C - 62<br>Pensky-Martens, Low Range Tag Closed Tester<br>9F - 62 | −5 to +110 C<br>20 to 230 F | | 57 | 0.5 C<br>1 F | 1 C<br>5 F | 5 C<br>10 F | 0.5 C<br>1 F | ASTM<br>9C or 9F<br>57 MM IMM | 160 C<br>320 F |
| 10C - 62<br>Pensky-Martens, High Range<br>10F - 62 | 90 to 370 C<br>200 to 700 F | | 57 | 2 C<br>5 F | 10 C<br>25 F | 20 C<br>50 F | [a]<br>[b] | ASTM<br>10C or 10F<br>57 MM IMM | |

[a] Scale error: 1 C up to 260 C; 2 C over 260 C.

[b] Scale error: 2.5 F up to 500 F; 3.5 F over 500 F.

[c] An expansion chamber is provided for relief of gas pressure to avoid distortion of the bulb at higher temperatures. It is not for the purpose of joining mercury separations; and under no circumstances should the thermometer be heated above the highest temperature reading.

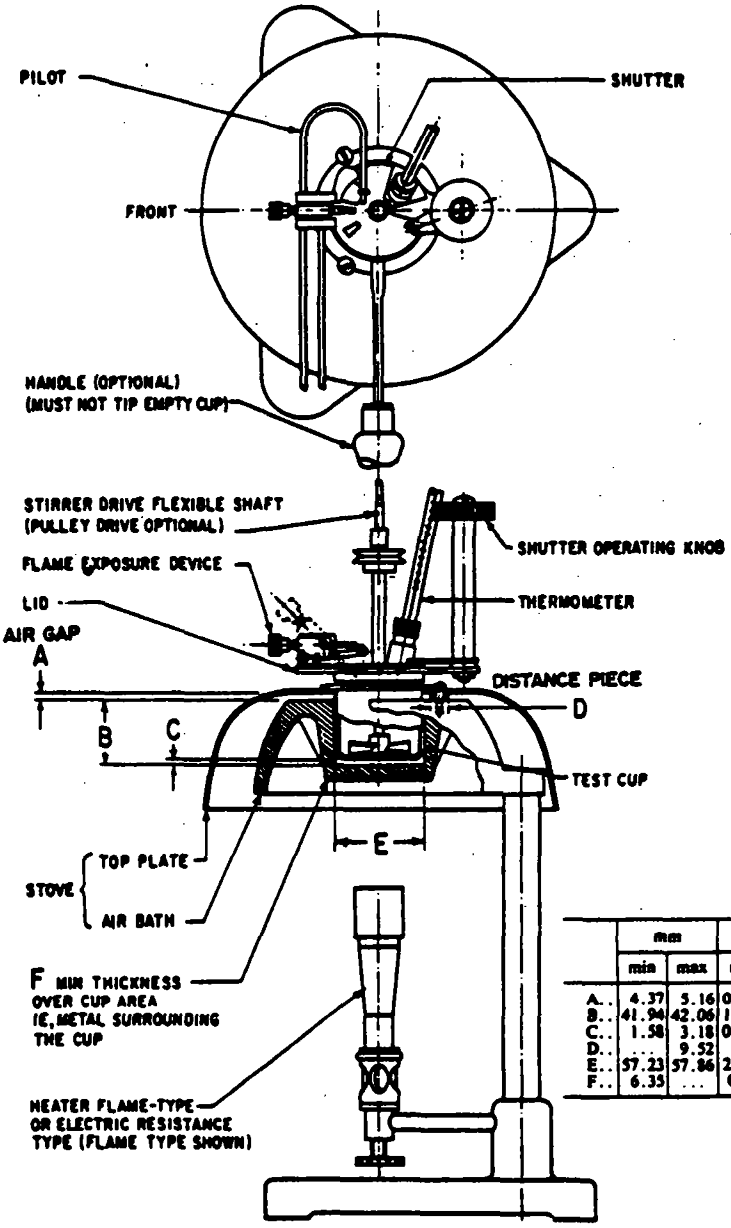
D 93 — 34



| | | Bulb | | Scale Location | | | | Ice Point Scale | | Contraction Chamber | | Stem Enlargement | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Total Length, ±5 | Stem OD | Length | OD | Bottom of Bulb to Line at | Distance | Bottom of Bulb to Line at | Distance | Range | Bottom of Bulb to Ice Point | Distance to Bottom, min | Distance to Top, max | OD | Length | Distance to Bottom |
| B | C | D | E | | F | | G | H | | I | J | K | L | M |
| 287 | 6.0 to 7.0 | 9.0 to 13 | > stem | 0 C<br>32 F | 85 to 98 | 100 C<br>212 F | 221 to 237 | | | | | 7.5 to 8.5 | 2.5 to 5.0[d] | 64 to 66 |
| 287 | 6.0 to 7.0 | 8.0 to 10 | 4.5 to 6.0 | 110 C<br>230 F | 86 to 99 | 360 C<br>680 F | 227 to 245 | | | | | 7.5 to 8.5 | 2.5 to 5.0[d] | 64 to 66 |

[d] The length of the enlargement, and the distance from the bottom of the enlargement to the bottom of the bulb shall be measured with the test gage shown in Fig. A6.

D 93 — 34

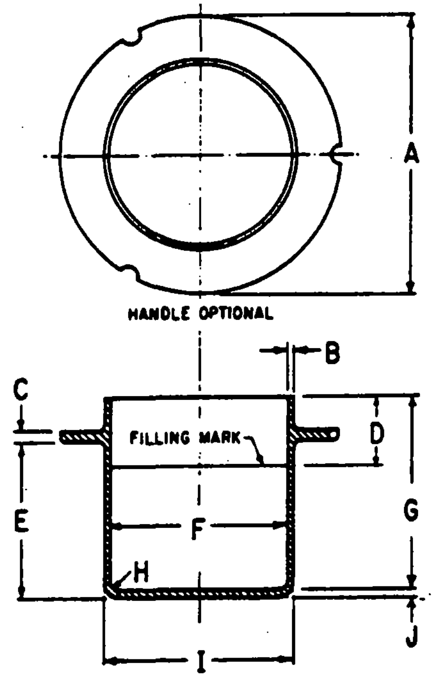


| | mm | | in. | |
|---|---|---|---|---|
| | min | max | min | max |
| A.. | 4.37 | 5.16 | 0.172 | 0.203 |
| B.. | 41.94 | 42.06 | 1.651 | 1.656 |
| C.. | 1.58 | 3.18 | 0.062 | 0.125 |
| D.. | ... | 9.52 | ... | 0.375 |
| E.. | 57.23 | 57.86 | 2.253 | 2.278 |
| F.. | 6.35 | ... | 0.25 | ... |

NOTE—Lid assembly may be positioned either right or left-handed.

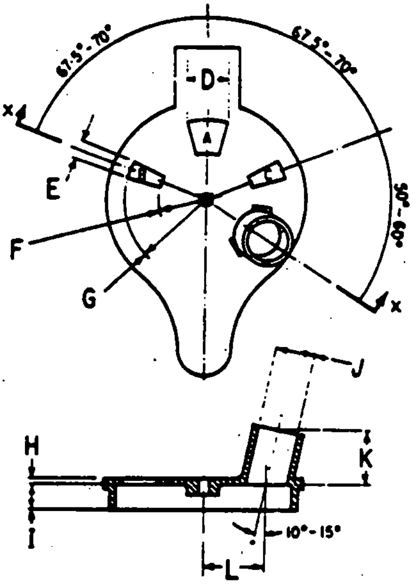
FIG. A1 Pensky-Martens Closed Flash Tester.

D 93 — IP 34



| | mm | | in. | |
|---|---|---|---|---|
| | min | max | min | max |
| A | 79.0 | 79.8 | 3.11 | 3.14 |
| B | 1.0 | ... | 0.04 | ... |
| C | 2.8 | 3.6 | 0.11 | 0.14 |
| D | 21.72 | 21.84 | 0.855 | 0.860 |
| E | 45.47 | 45.72 | 1.790 | 1.800 |
| F | 50.72 | 50.85 | 1.997 | 2.002 |
| G | 55.75 | 56.00 | 2.195 | 2.205 |
| H | 3.8 | 4.0 | 0.15 | 0.16 |
| I | 53.90 | 54.02 | 2.122 | 2.127 |
| J | 2.29 | 2.54 | 0.090 | 0.100 |

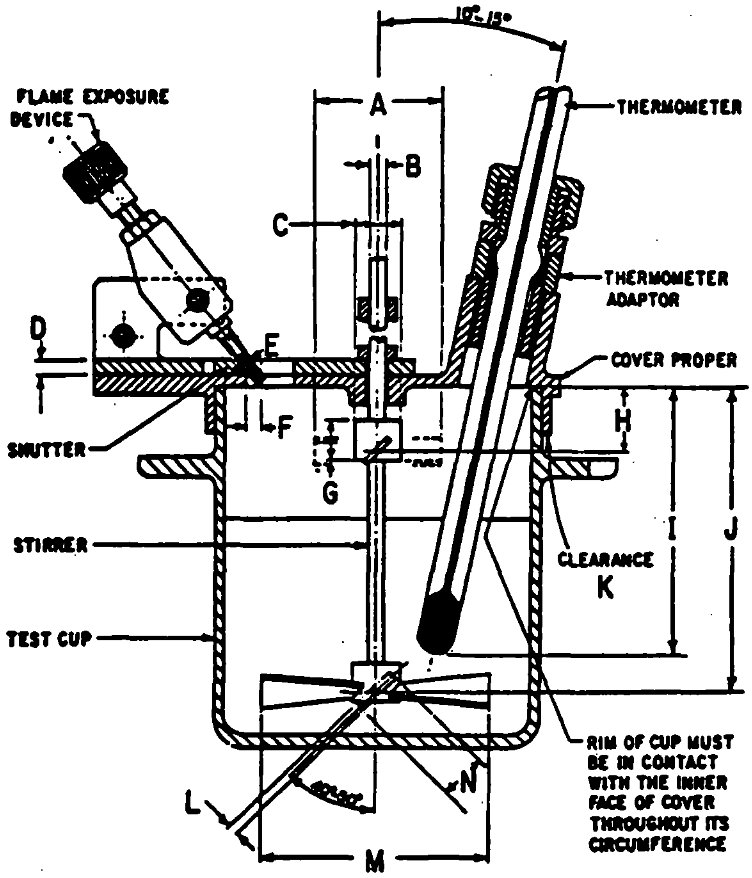
FIG. A2 Test Cup.



| | mm | | in. | |
|---|---|---|---|---|
| | min | max | min | max |
| D | 12.7 | 13.5 | 0.50 | 0.53 |
| E | 4.8 | 5.6 | 0.19 | 0.22 |
| F | 13.5 | 14.3 | 0.53 | 0.56 |
| G | 23.8 | 24.6 | 0.94 | 0.97 |
| H | 1.2 | 2.0 | 0.05 | 0.08 |
| I | 7.9 | ... | 0.31 | ... |
| J | 12.27 | 12.32 | 0.483 | 0.485 |
| K | 16.38 | 16.64 | 0.645 | 0.655 |
| L | 18.65 | 19.45 | 0.734 | 0.766 |

FIG. A3 Cover Proper.

D 93 — 34

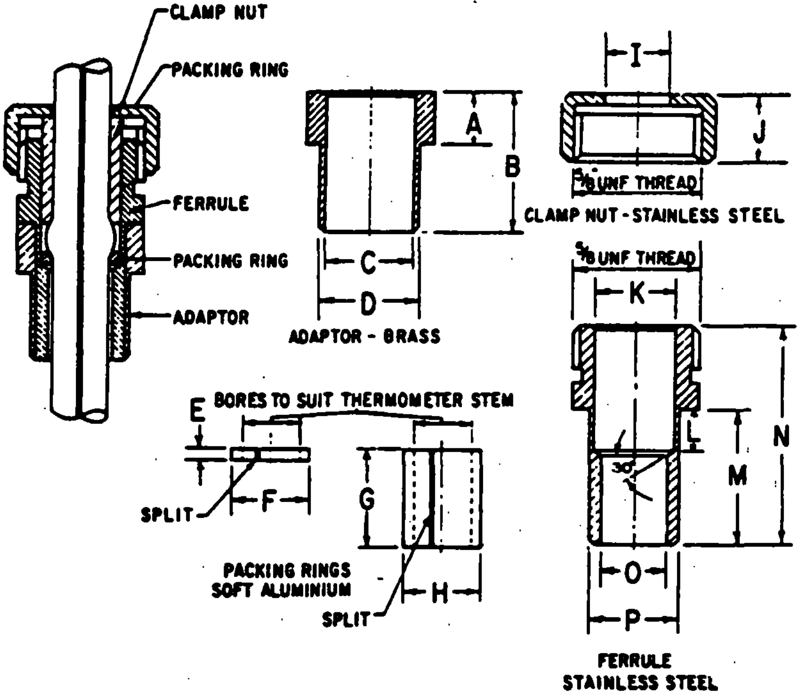


| | mm | | in. | |
|---|---|---|---|---|
| | min | max | min | max |
| A | 18.3 | 19.8 | 0.72 | 0.78 |
| B | 2.38 | 3.18 | 0.094 | 0.125 |
| C | 7.6 | 8.4 | 0.30 | 0.33 |
| D | 2.0 | 2.8 | 0.08 | 0.11 |
| E | 0.69 | 0.79 | 0.027 | 0.031 |
| F | 2.0 | 2.8 | 0.08 | 0.11 |
| G | 6.4 | 10.4 | 0.25 | 0.41 |
| H | 9.6 | 11.2 | 0.38 | 0.44 |
| I* | 43.0 | 46.0 | 1.69 | 1.81 |
| J | 50.0 | 51.6 | 1.97 | 2.03 |
| K | ... | 0.36 | ... | 0.014 |
| L | 1.22 | 2.06 | 0.048 | 0.08 |
| M | 31.8 | 44.4 | 1.25 | 1.75 |
| N | 7.6 | 8.4 | 0.30 | 0.33 |

* Includes tolerance for length of thermometer given in ASTM Specification E 1,[4] ASTM Thermometers.

FIG. A4 Test Cup and Cover Assembly.

D 93 — 34



| | mm | | in. | |
|---|---|---|---|---|
| | min | max | in. | max |
| A | 6.20 | 6.50 | 0.244 | 0.256 |
| B | 17.0 | 18.0 | 0.67 | 0.71 |
| C | 9.80 | 9.85 | 0.386 | 0.388 |
| D | 12.19 | 12.24 | 0.480 | 0.482 |
| E | 1.40 | 1.65 | 0.055 | 0.065 |
| F | 8.56 | 8.61 | 0.337 | 0.339 |
| G | 12.4 | 13.0 | 0.49 | 0.57 |
| H | 8.56 | 8.61 | 0.337 | 0.339 |
| I | 8.1 | 8.6 | 0.32 | 0.34 |
| J | 9.9 | 10.7 | 0.39 | 0.42 |
| K | 8.64 | 8.69 | 0.340 | 0.342 |
| L | 5.1 | 5.6 | 0.20 | 0.22 |
| M | 17.0 | 17.5 | 0.67 | 0.69 |
| N | 27.4 | 28.2 | 1.08 | 1.11 |
| O | 7.11 | 7.16 | 0.280 | 0.282 |
| P | 9.73 | 9.78 | 0.383 | 0.385 |

FIG. A5 Dimensions for Thermometer Adapter, Ferrule, and Packing Ring.

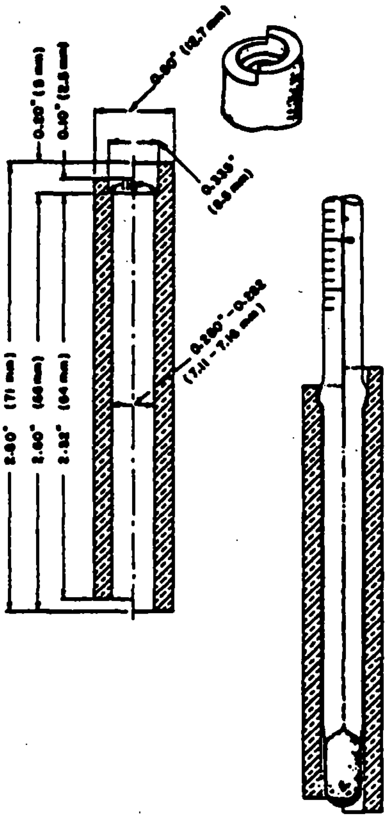


**FIG. A6 Test Gage for Checking Enlargements on Thermometers.**

*By publication of this standard no position is taken with respect to the validity of any patent rights in connection therewith, and the American Society for Testing and Materials does not undertake to insure anyone utilizing the standard against liability for infringement of any Letters Patent nor assume any such liability.*

**Statutory Authority:** *MS s 116.07 subd 4*

**7045.1180 APPENDIX E.**

# AMERICAN SOCIETY FOR TESTING AND MATERIALS

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THIS

## *Standard Method for*

## CLASSIFICATION OF SOILS FOR ENGINEERING PURPOSES[1]

**ASTM Designation: D 2487 – 69**

This Standard of the American Society for Testing and Materials is issued under the fixed designation D 2487; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

### 1. Scope

1.1 This method describes a system for classifying mineral and organo-mineral soils for engineering purposes based on laboratory determination of particle-size characteristics, liquid limit, and plasticity index, and shall be used when precise classification is required.

NOTE 1—This method provides qualitative data only. When quantitative information is required for detailed designs of important structures, this method must be supplemented by laboratory tests or other quantitative data to determine performance characteristics of the soil under expected field conditions.

NOTE 2—This method may also be used as an aid in training personnel in the use of ASTM Recommended Practice D 2488, for Description of Soils (Visual-Manual Procedure).[2]

### 2. Apparatus

2.1 *Apparatus for Preparation of Samples*—See Method D 421, for Dry Preparation of Soil Samples for Grain-Size Analysis and Determination of Soil Constants[2] or ASTM Method D 2217, for Wet Preparation of Soil Samples for Grain Size Analysis and Determination of Soil Constants.[2]

2.2 *Apparatus for Liquid Limit Test*—See ASTM Method D 423, Test for Liquid Limit of Soils.[2]

2.3 *Apparatus for Plastic Limit Test*—See ASTM Method D 424, Test for Plastic Limit and Plasticity Index of Soils.[2]

2.4 *Apparatus for Particle Size Analysis*—See 2. Apparatus, of ASTM Method D 422, for Grain-Size Analysis of Soils,[2] ASTM Method D 1140, Test for Amount of Materials in Soils Finer than the No. 200 Sieve,[2] and the classification chart (Fig. 1).

### 3. Sampling

3.1 Sampling shall be conducted in accordance with ASTM Method D 1452, for Soil Investigation and Sampling by Auger Borings,[2] ASTM Method D 1586, for Penetration Test and Split-Barrel Sampling of Soils,[2] ASTM Method D 1587, for Thin-Walled Tube Sampling of Soils,[2] or another standard accepted procedure.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the ASTM Committee D-18 on Soil and Rock for Engineering Purposes. A list of members may be found in the ASTM Yearbook.

Current edition effective Nov. 14, 1969. Originally issued 1966. Replaces D 2487 – 66 T.

[2] *Annual Book of ASTM Standards*, Part 11.

3.2 The sample shall be carefully identified as to origin by a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline.

3.3 The sample should also be described in accordance with ASTM Recommended Practice D 2488.

NOTE 3—A soil which is composed primarily of undecayed or partially decayed organic matter and has a fibrous texture, dark brown to black color, and organic odor should be designated as a *highly organic soil*, PT, and not subjected to the classification procedures described hereafter.

**4. Test Sample**

4.1 Test samples shall represent that portion of the field sample finer than the 3-in. (76-mm) sieve and shall be obtained as follows:

4.1.1 Air dry the field sample,

4.1.2 Weigh the field sample,

4.1.3 Separate the field sample into two fractions on a 3-in. (76-mm) sieve, and

4.1.4 Weigh the fraction retained on the 3-in. (76-mm) sieve. Compute the percentage of plus 3-in. (76-mm) material in the field sample, and note this percentage as auxiliary information.

4.1.5 Thoroughly mix the fraction passing the 3-in. (76-mm) sieve and select test samples.

**5. Preliminary Classification Procedure**

5.1 Procedure for the determination of percentage finer than the No. 200 sieve.

5.1.1 From the material passing the 3-in. (76-mm) sieve select a test sample and determine the percentage of the test sample finer than the No. 200 sieve in accordance with Method D 1140.

NOTE 4—Step 5.1.1 may be omitted if the soil can obviously be classified as fine-grained by visual inspection (see 5.3).

5.2 Classify the soil as *coarse-grained* if more than 50 per cent of the test sample is retained on the No. 200 sieve and follow 6, Procedure for Classification of Coarse-Grained Soils.

5.3 Classify the soil as *fine-grained* if 50 per cent or more of the test sample passes the No. 200 sieve and follow 7, Procedure for Classification of Fine-Grained Soils.

**6. Procedure for Classification of Coarse-Grained Soils** (More than 50 per cent retained on No. 200 Sieve).

6.1 Select test samples from the material passing the 3-in. (76-mm) sieve for the determination of particle-size characteristics, liquid limit, and plasticity index in accordance with ASTM Method D 421 or ASTM Method D 2217.

6.2 Determine the cumulative particle-size distribution of the fraction coarser than the No. 200 sieve in accordance with ASTM Method D 422.

6.3 Classify the sample as *gravel*, G, if 50 per cent or more of the coarse fraction (plus No. 200 sieve) is retained on the No. 4 sieve.

6.4 Classify the sample as *sand*, S, if more than 50 per cent of the coarse fraction (plus No. 200 sieve) passes the No. 4 sieve.

6.5 If less than 5 per cent of the test sample passed the No. 200 sieve as determined in 5.1.1, compute the coefficient of uniformity, $C_u$, and coefficient of curvature, $C_z$ as given in Equations 1 and 2.

$$C_u = \frac{D_{60}}{D_{10}} \qquad (1)$$

$$C_z = \frac{(D_{30})^2}{D_{10} \times D_{60}} \qquad (2)$$

| MAJOR DIVISIONS | | | GROUP SYMBOLS | TYPICAL NAMES |
|---|---|---|---|---|
| COARSE-GRAINED SOILS More than 50% retained on No. 200 sieve* | GRAVELS 50% or more of coarse fraction retained on No. 4 sieve | CLEAN GRAVELS | GW | Well-graded gravels and gravel-sand mixtures, little or no fines |
| | | | GP | Poorly graded gravels and gravel-sand mixtures, little or no fines |
| | | GRAVELS WITH FINES | GM | Silty gravels, gravel-sand-silt mixtures |
| | | | GC | Clayey gravels, gravel-sand-clay mixtures |
| | SANDS More than 50% of coarse fraction passes No. 4 sieve | CLEAN SANDS | SW | Well-graded sands and gravelly sands, little or no fines |
| | | | SP | Poorly graded sands and gravelly sands, little or no fines |
| | | SANDS WITH FINES | SM | Silty sands, sand-silt mixtures |
| | | | SC | Clayey sands, sand-clay mixtures |
| FINE-GRAINED SOILS 50% or more passes No. 200 sieve* | SILTS AND CLAYS Liquid limit 50% or less | | ML | Inorganic silts, very fine sands, rock flour, silty or clayey fine sands |
| | | | CL | Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays |
| | | | OL | Organic silts and organic silty clays of low plasticity |
| | SILTS AND CLAYS Liquid limit greater than 50% | | MH | Inorganic silts, micaceous or diatomaceous fine sands or silts, elastic silts |
| | | | CH | Inorganic clays of high plasticity, fat clays |
| | | | OH | Organic clays of medium to high plasticity |
| Highly Organic Soils | | | PT | Peat, muck and other highly organic soils |

* Based on the material passing the 3-in. (76-mm) sieve.

FIG. 1—Soil Classification Chart.

CLASSIFICATION OF SOILS FOR ENGINEERING PURPOSES (D 2487)

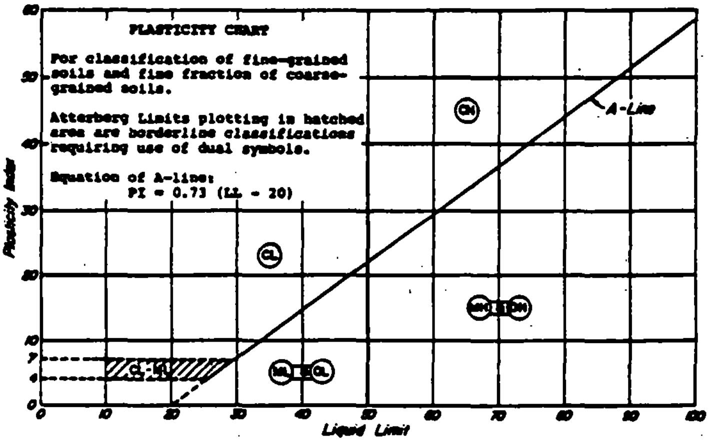
<table>
<tr><th colspan="3">CLASSIFICATION CRITERIA</th></tr>
<tr><td rowspan="8">Classification on basis of percentage of fines<br>Less than 5% Pass No. 200 sieve GW, GP, SW, SP<br>More than 12% Pass No. 200 sieve GM, GC, SM, SC<br>5% to 12% Pass No. 200 sieve Borderline Classification requiring use of dual symbols</td><td colspan="2">$C_u = D_{60}/D_{10}$ Greater than 4<br>$C_z = \frac{(D_{30})^2}{D_{10} \times D_{60}}$ Between 1 and 3</td></tr>
<tr><td colspan="2">Not meeting both criteria for GW</td></tr>
<tr><td>Atterberg limits plot below "A" line or plasticity index less than 4</td><td rowspan="2">Atterberg limits plotting in hatched area are borderline classifications requiring use of dual symbols</td></tr>
<tr><td>Atterberg limits plot above "A" line and plasticity index greater than 7</td></tr>
<tr><td colspan="2">$C_u = D_{60}/D_{10}$ Greater than 6<br>$C_z = \frac{(D_{30})^2}{D_{10} \times D_{60}}$ Between 1 and 3</td></tr>
<tr><td colspan="2">Not meeting both criteria for SW</td></tr>
<tr><td>Atterberg limits plot below "A" line or plasticity index less than 4</td><td rowspan="2">Atterberg limits plotting in hatched area are borderline classifications requiring use of dual symbols</td></tr>
<tr><td>Atterberg limits plot above "A" line and plasticity index greater than 7</td></tr>
<tr><td colspan="3"> </td></tr>
<tr><td colspan="3">Visual-Manual Identification, See ASTM Designation D 2488.</td></tr>
</table>

FIG. 1—*Continued.*

in which $D_{10}$, $D_{30}$, and $D_{60}$ are the particle-size diameters corresponding respectively to 10, 30, and 60 per cent passing on the cumulative particle-size distribution curve.

6.5.1 Classify the sample as *well-graded gravel*, GW or *well-graded sand*, SW, if $C_u$ is greater than 4 for gravel and 6 for sand, and $C_z$ is between 1 and 3.

6.5.2 Classify the sample as *poorly graded gravel*, GP, or *poorly graded sand*, SP, if either the $C_u$ or the $C_z$ criteria for well-graded soils are not satisfied.

6.6 If more than 12 per cent of the test sample passed the No. 200 sieve as determined in 5.1.1 determine the liquid limit and the plasticity index of a portion of the test sample passing the No. 40 sieve in accordance with ASTM Method D 423 and ASTM Method D 424.

6.6.1 Classify the sample as *silty gravel*, GM, or *silty sand*, SM if the results of the limits tests show that the fines are silty, that is, the plot of the liquid limit versus plasticity index falls below the "A" line (see Plasticity Chart, Fig. 1) or the plasticity index is less than 4.

6.6.2 Classify the sample as *clayey gravel*, GC, or *clayey sand*, SC, if the fines are clayey, that is, the plot of liquid limit versus plasticity index falls above the "A" line and the plasticity index is greater than 7.

6.6.3 If the fines are intermediate between silt and clay, that is, the plot of liquid limit versus plasticity index falls on or practically on the "A" line or falls above the "A" line but the plasticity index is in the range of 4 to 7, the soil should be given a borderline classification, such as GM-GC or SM-SC.

6.7 If 5 to 12 per cent of the test sample passed the No. 200 sieve, the soil should be given a borderline classification based on both its gradation and limit test characteristics, (see 6.6) such as GW-GC or SP-SM.

NOTE 5—In doubtful cases, the rule is to favor the less plastic classification. Example: a gravel with 10 per cent fines, a $C_u$ of 20, a $C_z$ of 2.0, and a plasticity index of 6 would be classified as GW-GM rather than GW-GC.

**7. Procedure for Classification of Fine-Grained Soils (50 per cent or more passing No. 200 sieve)**

7.1 From the material passing the 3-in. (76-mm) sieve, select a test sample for the determination of the liquid limit and plasticity index in accordance with ASTM Method D 421 or ASTM Method D 2217.

NOTE 6—It is recommended that the method for wet preparation be used for soils containing organic matter or irreversible mineral colloids.

7.2 Determine the liquid limit and the plasticity index of a portion of the test sample passing the No. 40 sieve in accordance with ASTM Method D 423, and ASTM Method D424.

7.3 Classify the soil as *inorganic clay*, C, if the plot of liquid limit versus plasticity index falls above the "A" line and the plasticity index is greater than 7.

7.3.1 Classify the soil as *inorganic clay of low to medium plasticity*, CL, if the liquid limit is less than 50 and the plot of liquid limit versus plasticity index falls above the "A"-line and the plasticity index is greater than 7. See area identified as CL on the Plasticity Chart, Fig. 1.

7.3.2 Classify the soil as *inorganic clay of high plasticity*, CH, if the liquid limit is greater than 50 and the plot of liquid limit versus plasticity index falls above the "A"-line. See area identified as CH on the Plasticity Chart, Fig. 1.

NOTE 7—In cases where the liquid limit exceeds 100 or the plasticity index exceeds 60, the plasticity chart may be expanded by maintaining the same scales on both axes and extending the A-line at the indicated slope.

7.4 Classify the soil as *inorganic silt*, M, if the plot of liquid limit versus plas-

CLASSIFICATION OF SOILS FOR ENGINEERING PURPOSES (D 2487)

ticity index falls below the "A" line or if the plasticity index is less than 4, unless it is suspected that organic matter is present in sufficient amounts to influence the soil properties, then tentatively classify the soil as *organic silt or clay*, O.

7.4.1 If the soil has a dark color and an organic odor when moist and warm, a second liquid limit test should be performed on a test sample which has been oven-dried at 110 ± 5 deg C for 24 hr.

7.4.2 Classify the soil as *organic silt or clay*, O, if the liquid limit after oven drying is less than three-fourths of the liquid limit of the original sample determined before drying. (See ASTM Designation D 2217, Procedure B).

7.4.3 Classify the soil as *inorganic silt of low plasticity*, ML, or as *organic silt or silt-clay of low plasticity*, OL, if the liquid limit is less than 50 and the plot of liquid limit versus plasticity index falls below the "A"-line or the plasticity index is less than 4. See area identified as ML and OL on the Plasticity Chart, Fig. 1.

7.4.4 Classify the soil as *inorganic silt of medium to high plasticity*, MH, or as *organic clay or silt-clay of medium to high plasticity*, OH, if the liquid limit is more than 50 and the plot of liquid limit versus plasticity index falls below the "A"-line. See area identified as MH and OH on the Plasticity Chart, Fig. 1.

7.5 In order to indicate their borderline characteristics, some fine-grained soils should be classified by dual symbols.

7.5.1 If the plot of liquid limit versus plasticity index falls on or practically on the "A" line or above the "A" line where the plasticity index is in the range of 4 to 7, the soil should be given an appropriate borderline classification such as CL-ML or CH-OH.

7.5.2 If the plot of liquid limit versus plasticity index falls on or practically on the line liquid limit = 50, the soil should be given an appropriate borderline classification such as CL-CH or ML-MH.

NOTE 8—In doubtful cases, the rule for classification is to favor the more plastic classification. Example: a fine-grained soil with a liquid limit of 50 and a plasticity index of 22 would be classified as CH-MH rather than CL-ML.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.1190 APPENDIX F.

# AMERICAN SOCIETY FOR TESTING AND MATERIALS

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*Recommended Practice for*

THIS

# DESCRIPTION OF SOILS[1]
# (VISUAL-MANUAL PROCEDURE)

**ASTM Designation: D 2488 – 69**

This Recommended Practice of the American Society for Testing and Materials is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

## 1. Scope

1.1 This recommended practice describes a procedure for the identification and description of soils for engineering purposes based on visual examination and simple manual tests.

NOTE 1—It does not conflict with other methods of soil identification or classification and in fact the user is encouraged to supplement the descriptions recommended herein with geologic, pedologic or local terms of description. On the other hand, when precise classification of soils for engineering purposes are required ASTM Method D 2487, for Classification of Soils for Engineering Purposes,[2] should be employed.

1.2 This recommended practice is intended to be used not only for identification of soils in the field but also in the office or in the laboratory or wherever soil samples are inspected and described.

1.2.1 The practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 2—The ability to identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics while performing the identification procedures.

## 2. Definitions and Description of Terms

2.1 The definitions of the soil components, boulders, cobbles, gravel, sand fines (silt and clay), organic soil, and peat are in accordance with ASTM Definitions D 653, Terms and Symbols Relating to Soil Mechanics.[2]

## 3. Equipment

3.1 *Required Equipment:*

3.1.1 Small supply of water and

3.1.2 Pocket knife or small spatula.

3.2 *Useful Auxiliary Equipment:*

3.2.1 Small bottle of dilute hydrochloric acid,

3.2.2 Small test tube and stopper,

3.2.3 Munsell Soil Color Chart or Rock Color Chart,

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[1] Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the ASTM Committee D-18 on Soil and Rock for Engineering Purposes. A list of members may be found in the ASTM Year Book.

Current edition effective Dec. 19, 1969. Originally issued 1966. Replaces D 2488 – 66 T.

[2] *Annual Book of ASTM Standards*, Part 11.

DESCRIPTION OF SOILS (VISUAL-MANUAL PROCEDURE) (D 2488)

3.2.4 Small hand lens, and

3.2.5 Pocket penetrometer or shear gauge.

**4. Sampling**

4.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate accepted or standard procedure.

NOTE 3—Preferably the sampling procedure should be identified as having been conducted in accordance with ASTM Method D 1452, for Soil Investigation and Sampling by Auger Borings,[3] ASTM Method D 1586, for Penetration Test and Split-Barrel Sampling of Soils,[3] ASTM Method D 1587 for Thin-Walled Tube Sampling of Soils,[3] etc.

4.2 The sample shall be carefully identified as to origin.

NOTE 4—The sample identification may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline.

**5. General Procedure for Identification**

5.1 On the basis of an examination of the characteristics of the particles which make up a soil sample it is possible to assign it to one of three primary groups. Although most soils have components representative of two or more groups it is usually possible to discern the most important component and assign the sample to that group. A most important distinction is made on the basis of size. Individual particles visible to the naked eye make up the *coarse* fraction and those too small to be seen individually make up the *fine* fraction or the *fines*. The *organic* component of soils may consist of undecayed or partially decayed twigs, leaves, needles, stems, roots, etc. which impart a woody or fibrous texture to the soil or it may also be so finely divided that it can only be identified by its dark brown, dark gray or black color and distinctive organic odor.

5.1.1 *Coarse-grained soils* are those in which more than half (by weight) of the particles are visible to the naked eye. In making this estimate, particles coarser than 3 in. (76 mm) in diameter should be excluded. However, where such very coarse particles can be observed in surface soils or in exposure in the walls of test pits an estimate of the percentage of a large volume of soil which is occupied by cobbles and boulders should be made. This percentage should be recorded independently of the description of the material smaller than 3 in. (76 mm) in diameter.

5.1.2 *Fine-grained soils* are those in which more than half (by weight) of the particles are so fine that they cannot be seen by the naked eye. They are partly subdivided on the basis of simple manual tests. They are also subidivded as *inorganic* or *organic* on the basis of the presence or absence of a significant quantity of organic matter. Inorganic soils are usually characterized by their bright or light colors.

5.1.3 *Organic soils* are those which contain significant quantities of organic matter. *Highly organic soils* can readily be recognized by the presence of decayed roots, leaves, grasses and other fibrous vegetable matter in various stages of decay. When moist, they have a dark brown, very dark gray or black color and a soft spongy feel. If the samples are fresh, a distinctive odor of rotting organic matter can usually be noted. Many soils are only *partly organic* and are in fact composed predominantly of inorganic material. Such soils, however, behave differently from typical inorganic soils and the presence of relatively small amounts of organic matter should be noted wherever possible. Any soil which has a dark brown, dark gray or black color probably contains some finely divided organic material. The identification as an organic soil can usually be completed by carefully noting the organic odor of fresh samples. If the sample is dry it should be moistened and warmed in the hand which may help to bring out the distinctive odor.

5.1.4 *Mixed-grained soils* are those inorganic or partly organic soils which contain materials representative of both the coarse and fine soil fractions. A high percentage of natural soils are mixed-grained. In many of these, however, one fraction predominates to such an extent that for practical purposes the soil may be identified as that fraction and the presence of the other noted as an appropriate fraction. As nearly as possible, the relative proportion of coarse and fine fraction should be estimated for all mixed-grained soils.

TABLE 1—IDENTIFICATION OF CONSISTENCY OF FINE-GRAINED SOILS FROM MANUAL TESTS.

| Consistency | Identification Procedure | Shear Strength, tons/ft² or kg/cm² |
|---|---|---|
| Soft | Easily penetrated several inches by thumb | Less than 0.25 |
| Firm (medium) | Penetrated several inches by thumb with moderate effort | 0.25 to 0.50 |
| Stiff | Readily indented by thumb, but penetrated only with great effort | 0.50 to 1.00 |
| Very stiff | Readily indented by thumb nail | 1.00 to 2.00 |
| Hard | Indented with difficulty by thumbnail | over 2.00 |

5.2 *Color* is an important property in identifying organic soils and within a given locality it may also be useful in identifying materials of similar geologic origin. Although qualitative color names are somewhat helpful, positive color identifications obtained by comparison with a standard color chart are even more useful. If the sample contains layers or patches of varying colors, this should be noted and all representative colors should be described. If possible, color should be described for moist samples.

NOTE 5—Charts especially prepared for describing the colors of soil and rock are available respectively. Such charts give typical descriptive names for the color chips and the correct Munsell color notation in terms of hue, value and chroma. Example: *Pink* (Moderate orange pink), *5 YR 8/4*.

5.3 Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation This is especially apparent in fresh samples, but if the samples are dried the odor may often be revived by heating a moistened sample. If the soil is dark colored, the odor should be described as *organic*, *earthy*, or *none*.

5.4 Whenever intact samples are described an estimate of the *moisture condition* should be noted. *Dry* materials require the addition of considerable moisture to attain optimum for compaction. *Moist* materials are near the optimum moisture content. *Wet* soils require drying to attain optimum moisture content and *saturated* (very wet) soils come from below the water table.

5.5 The *structural* characteristics of intact soil samples provide important clues to their performance as foundation materials. Whenever such samples are available or when the soil profile may be inspected during sampling from a pit, the structural characteristics should be described. *Stratified* materials consist of alternating layers of varying types (or color). If the layers are less than about ¼ in. (6 mm) thick, it may be described as *laminated* (or *varved*, if mostly fine-grained). *Fissured* materials break along definite planes of fracture with little resistance to fracturing. If the fracture planes appear polished or glossy, they should be described as *slickensided*. If a cohesive soil can be easily broken into small angular lumps which resist further breakdown, the structure may be described as *blocky*. A *lensed* structure is indicated by the inclusion of small pockets of different texture, such as small lenses of sand scattered through a mass of clay. The presence of special structural characteristics such as *root holes*, or *porous* openings should also be noted. If no structural characteristics are apparent, the soil may be described as *nonstratified* or *homogeneous*.

**(a) Rounded** **(b) Angular**

**(c) Subrounded** **(d) Subangular**

FIG. 1—Typical Shapes of Bulky Grains.

5.6 Some soils show definite evidence of *cementation* in the intact state. Where this is noted, the degree of cementation may be described as *weak* or *strong*. Since calcium carbonate is the most common cementing agent a report of its presence on the basis of the reaction with dilute hydrochloric acid is important. The intensity of the HCl reaction should be described as *none*, *weak*, or *strong*.

5.7 The degree of compactness or natural density of cohesionless soils and the consistency of cohesive soils are also important in foundation problems. Whenever undisturbed materials are examined either in field pits or from sealed samples, estimates of these properties should be noted. Usually the process of sampling disturbs cohesionless soils to such an extent that the natural density may only be determined in place. In *dense* cohesionless soil it is difficult to drive a 2 by 2 in. (5 by 5 cm) wooden stake more than a few inches; however, such a stake can easily be driven into *loose* material. Obviously, this simple method cannot be used to determine the relative density of *cemented* soils.

5.7.1 The consistency of cohesive soils may be determined in place or on

DESCRIPTION OF SOILS (VISUAL-MANUAL PROCEDURE) (D 2488)

undisturbed samples in accordance with the identification procedure given in Table 1. The quantitative measure of the shear strength is given as a basis for correlation with values obtained from pocket penetrometers or shear gauges, which are often used to estimate consistency.

5.8 It is often desirable to add an estimate of the classification of the soil in accordance with the groups used for engineering classification (Fig. 1 of ASTM Method D 2487[2]). The group symbol should be placed in parentheses at the end of the description in order to indicate that the classification has been estimated.

TABLE 2—CHECK LIST FOR DESCRIPTION OF COARSE-GRAINED SOILS.

1. *Typical Name* Boulders Cobbles Gravel Sand
   Add descriptive adjectives for minor constituents.
2. *Gradation* Well graded Poorly graded (Uniformly graded or Gap-graded)
   Describe range of particle sizes or predominant size or sizes as coarse, medium, or fine sand or gravel.
3. *Maximum Particle Size* Note per cent boulders and cobbles
4. *Size Distribution* Approximate per cent gravel, sand and fines in fraction finer than 3 in. (76 mm). Indicate plasticity of fines (See 7.5).
5. *Grain Shape* Angular Subangular Subrounded Rounded
6. *Mineralogy* Rock type for gravel, predominant minerals in sand.
   Note especially presence of mica flakes, shaly particles and organic material.
7. *Color* Use Munsell notation, if possible
8. *Odor* None Earthy Organic
   May be neglected except for dark colored soils.
9. *Moisture content* Dry Moist Wet Saturated
10. *Natural Density* Loose Dense
11. *Structure* Stratified Lensed Nonstratified
12. *Cementation* Weak Strong
    Note reaction with HCl as none, weak or strong.
13. *Local or Geologic Name*
14. *Group Symbol* Estimate if desired. See Classification Chart, Fig. 1, ASTM Designation D 2487.

**6. Procedure for Coarse-Grained Soils**

6.1 Select a representative sample of the soil material finer than 3 in. (76 mm) sieve, spread it out for examination and follow identification procedures.

6.1.1 For accurate identification, the minimum amounts of sample should be in accordance with the following schedule:

| Nominal Maximum Diameter | Minimum Sample Size |
|---|---|
| 2–2½ in. (50–65 mm) | 8 lb (3.6 kg) |
| 1–1½ in. (25–38 mm) | 4 lb (1.8 kg) |
| ¼–½ in. (6–12 mm) | 1 lb (0.45 kg) |
| less than ⅛ in. (3 mm) | ¼ lb (0.11 kg) |

6.2 Estimate the percentage of the coarse fraction larger than the No. 4 sieve (about ¼ in. or 5 mm).

6.2.1 Identify the soil as *gravel* if 50 per cent or more is larger.

6.2.2 Identify the soil as *sand* if less than 50 per cent is larger.

6.3 Estimate the percentage of fines.

6.3.1 Identify the soil as *clean gravel* or *clean sand* if the fines content is about 5 per cent or less.

6.3.1.1 Identify the soil as *well graded* if it has a wide range in grain size and substantial amounts of most intermediate particle sizes.

6.3.1.2 Identify the soil as *poorly graded* if it consists predominantly of one size (*uniformly graded*) or has a wide range of sizes with some intermediate sizes obviously missing (*gap-graded*).

6.3.2 Identify the soil as *gravel with fines* or *sand with fines* if the fines content is more than about 12 per cent.

6.3.2.1 Identify the soil as borderline *clean* to *with fines* if the fines content is between about 5 and 12 per cent.

6.3.2.2 Describe the fines as *silty* or *clayey* in accordance with identification procedures given under fine-grained soils.

6.4 Describe the grain shape of the sand and gravel portions of the coarse fraction as *angular*, *subangular*, *subrounded*, or *rounded*, (see Fig. 1).

6.4.1 *Angular* particles have sharp edges and relatively plane sides with unpolished surfaces.

6.4.2 *Subangular* particles are similar to angular but have somewhat rounded edges.

**TABLE 3—IDENTIFICATION OF FINE-GRAINED SOIL FRACTIONS FROM MANUAL TESTS.**

| Typical Name | Dry Strength | Dilatancy Reaction | Toughness of Plastic Thread | Plasticity[a] Description |
|---|---|---|---|---|
| Sandy Silt........ | None—Very Low | Rapid | Weak—Soft | None—Slight |
| Silt.............. | Very Low—Low | Rapid | Weak—Soft | None—Slight |
| Clayey Silt........ | Low—Medium | Rapid—Slow | Medium Stiff | Slight—Medium |
| Sandy Clay | Low—High | Slow—None | Medium Stiff | Slight—Medium |
| Silty Clay........ | Medium—High | Slow—None | Medium Stiff | Slight—Medium |
| Clay.............. | High—Very High | None | Very Stiff | High |
| Organic Silt...... | Low—Medium | Slow | Weak—Soft | Slight |
| Organic Clay...... | Medium—Very High | None | Medium Stiff | Medium—High |

[a] The term low may be substituted for slight in the description of plasticity.

6.4.3 *Subrounded* particles exhibit nearly plane sides but have well-rounded corners and edges.

6.4.4 *Rounded* particles have smoothly curved sides and no edges.

6.5 Add appropriate descriptive notes regarding maximum size, size distribution, per cent cobbles and boulders, mineralogy, color, odor, moisture condition, natural density, structure, cementation, local or geologic name, and group symbol. Follow check list, Table 2.

NOTE 6—A complete description of a river valley sample estimated to contain about 20 per cent gravel, 65 per cent sand and 15 per cent silt could take the form of this example: *Silty Sand* well-graded gravelly. Maximum size, 8 in. (203 mm), about 5 per cent cobbles. About 20 per cent subrounded igneous gravel, 65 per cent subrounded to subangular quartz sand, and 15 per cent low plasticity fines. Light brown (7.5 YR 6/4). Moist. Dense. Stratified. No reaction to HCl. Alluvial sand (SM).

**7. Procedure for Fine-Grained and Organic Soils**

7.1 Select a representative sample of the material for examination. See 6.1.1.

7.2 Describe the color of the moist soil.

7.3 Describe the odor of the moist soil (warming if necessary to intensify the odor).

7.4 Identify the soil as *organic* if it has a black, dark brown or dark gray color (Munsell value 4 or less, chroma 3 or less) and a distinctive organic odor.

7.4.1 Identify the soil as *highly organic* if it has predominantly a woody or fibrous texture resulting from a composition of partially decayed leaves, twigs, needles, stems, roots, etc. Further identification is unnecessary.

7.4.2 Identify the soil as *partly organic* if it does not have a fibrous texture and appears to be predominantly mineral in character. Proceed with identification procedure for fine-grained soils.

7.5 From the representative sample, select enough material to provide two cubes approximately ½ in. (13 mm) in size after the gravel and coarse sand fraction has been removed. Use these samples to perform the dry strength, dilatancy and plasticity tests.

7.5.1 *Dry Strength*—Mold one of the samples until it has the consistency of putty, adding a small amount of water if necessary, and form into a cube or ball. Allow the sample to dry completely in the sun, air or oven at a temperature not exceeding 110 C. Test the strength of the dry sample by crushing between the fingers.

NOTE 7—If the soil sample contains dry lumps, an experienced operator can determine the dry strength without preparing a pat for this particular purpose. The process of molding and drying usually produces higher strengths than are found in natural aggregates of soil. The presence of high-strength water-soluble cementing materials, such as calcium carbonates, may cause exceptionally high dry strengths but this can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 5.6).

7.5.1.1 Describe as *very low* or *none* if the dry sample crumbles with the mere pressure of handling.

7.5.1.2 Describe as *low* if the dry sample crumbles to powder with little finger pressure.

7.5.1.3 Describe as *medium* if considerable finger pressure is required to powder the sample. Usually, when the soil has medium dry strength a smear of powder can be easily rubbed off the smooth surface of the sample.

7.5.1.4 Describe as *high* if the sample cannot be crushed to powder by finger pressure, even though it may be broken. Usually, when the sample has high dry strength it is not even possible to rub off a smear of powder from a smooth surface of the dry sample.

7.5.1.5 Describe as *very high* if the sample cannot be broken between the thumb and a hard surface.

7.5.2 *Dilatancy*—Add sufficient water, if necessary, to the other one of the samples to produce a soft, but not sticky, consistency. Smooth the soil pat in the palm of one hand with the blade of a knife or small spatula, shake horizontally, and strike the back of the hand vigorously against the other hand several times. Note reaction. Squeeze the sample by closing the hand and note reaction.

7.5.2.1 Describe the reaction as *rapid* if water appears on the surface during shaking and disappears quickly upon squeezing. The presence or absence of the free water can be noted by the shiny or dull appearance of the surface.

7.5.2.2 Describe the reaction as *slow* if vigorous tapping is required to bring water to the surface and squeezing causes little change in appearance.

7.5.2.3 Describe the reaction as *none* if the test produces no visible change in the sample.

7.5.3 *Plastic Thread*—Following the completion of the dilatancy test the sample is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily it should be spread out into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. (3 mm). The thread will crumble near the *plastic limit*. Note the pressure required to roll out the thread especially near the plastic limit; also note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

7.5.3.1 Describe the thread as *weak and soft* if, near the plastic limit, only slight pressure is required to roll it, the thread has little or no strength and after crumbling the thread pieces cannot be formed into a coherent mass.

7.5.3.2 Describe the thread as *medium stiff* if, near the plastic limit, medium pressure is required to roll it, the thread will support its own weight when a few inches long, and after crumbling the thread pieces can be molded into a lump which crumbles with slight kneading.

7.5.3.3 Describe the thread as *very stiff* if, near the plastic limit, considerable pressure is required to roll it, the thread will easily support its own weight when several inches long, and after crumbling the thread pieces can be molded into a lump which is coherent and tough.

7.6.1 *Sandy silt* has very low dry strength or none, a reaction to the dilatancy test of rapid, a plastic thread which is weak and soft, and a significant sand content which can be noted by a gritty feel. It can be described as having slight plasticity or none.

7.6.2 *Silt* has very low to low dry strength, a reaction to the dilatancy test of rapid, and a plastic thread which is weak and soft. It can be described as having slight plasticity or none.

TABLE 4—CHECK LIST FOR DESCRIPTION OF FINE-GRAINED AND PARTLY-ORGANIC SOILS.

| | | | | | | |
|---|---|---|---|---|---|---|
| 1. *Typical Name* | Sandy Silt | Silt | Clayey Silt | Sandy Clay | | |
| | Silty Clay | Clay | Organic Silt | Organic Clay | | |
| 2. *Maximum Particle Size* | Note percentage of boulders and cobbles | | | | | |
| 3. *Size Distribution* | Approximate per cent gravel, sand and fines in fraction finer than 3 in. (76 mm) | | | | | |
| 4. *Dry Strength* | None | Very Low | Low | Medium | High | Very High |
| 5. *Dilatancy* | None | Slow | Rapid | | | |
| 6. *Plastic Thread* | Weak and Soft | Medium Stiff | Very Stiff | | | |
| 7. *Plasticity of Fines* | None | Slight (low) | Medium | High | | |
| 8. *Color* | Use Munsell notation, if possible. Note presence of mottling or banding. | | | | | |
| 9. *Odor* | None | Earthy | Organic | | | |
| | May be neglected except *for* dark-colored soils. | | | | | |
| 10. *Moisture Content* | Dry | Moist | Wet | Saturated | | |
| 11. *Consistency* | Soft | Firm (Medium) | Stiff | Very Stiff | Hard | |
| 12. *Structure* | Stratified | Laminated (Varved) | Fissured | | | |
| | Slickensided | Blocky | Lensed | Homogeneous (Nonstratified) | | |
| 13. *Cementation* | Weak | Strong | | | | |
| | Note reaction with dilute hydrochloric acid as none, weak or strong. | | | | | |
| 14. *Local or Geologic Name* | | | | | | |
| 15. *Group Symbol* | Estimate if desired. See Classification Chart, Fig. 1, ASTM Method D 2487 | | | | | |

7.5.4 *Plasticity*—On the basis of its dry strength, dilatancy and toughness describe the overall plasticity as shown in Table 3.

7.6 Identify the soil as *silt* or *clay* with appropriate adjectives. See Table 3.

NOTE 8—The relative percentage of coarse and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 sec. The relative proportions can be estimated from the relative volume of each size separate.

7.6.3 *Clayey silt* has low to medium dry strength, a reaction to the dilatancy test of rapid to slow, and a medium stiff plastic thread. It can be described as having slight or medium plasticity.

7.6.4. *Sandy clay* has low to high dry strength, a reaction to the dilatancy test of slow to none, and a medium stiff plastic thread which may break prematurely because of the presence of sand grains. It can be described as having slight or medium plasticity.

7.6.5 *Silty clay* has medium to high dry strength, a reaction to the dilatancy test of very slow to none, and a medium

DESCRIPTION OF SOILS (VISUAL-MANUAL PROCEDURE) (D 2488)

stiff plastic thread. It can be described as having slight or medium plasticity.

7.6.6 *Clay* has a high to very high dry strength, no reaction to the dilatancy test and a very stiff plastic thread. It can be described as having high plasticity.

7.6.7 *Organic silt* has low to medium dry strength, a slow reaction to dilatancy test, and a weak and soft plastic thread It can be described as having slight plasticity.

7.6.8 *Organic clay* has medium to very high dry strength, a reaction to the dilatancy test of very slow to none, and a medium stiff plastic thread. It can be described as having medium or high plasticity.

7.7 Add appropriate descriptive notes regarding maximum size, size distribution, per cent cobbles and boulders, plasticity of fines, color, odor, moisture condition, consistency, structure, cementation, local or geologic name and group symbol. Follow check list, Table 4.

NOTE 9—A complete description of an undisturbed sample of a windblown silt could take the form of this example: *Clayey silt*, some fine sand. Maximum size about 0.1 mm. About 10 per cent fine sand, 90 per cent slightly plastic fines. Yellowish brown (10 YR 5/6 dry). Dry. Firm. Nonstratified, but with numerous vertical root holes. Strong reaction to HCl. Loess (ML).

**Statutory Authority:** *MS s 116.07 subd 4*

**7045.1200 APPENDIX G.**

technical practices committees

NACE Standard TM-01-69

# Test Method

# Laboratory Corrosion Testing of Metals for the Process Industries

Approved March, 1969
National Association of Corrosion Engineers
2400 West Loop South
Houston, Texas 77027
713/622-8980

The National Association of Corrosion Engineers issues this Standard in conformity to the best current technology regarding the specific subject. This Standard represents minimum requirements and should in no way be interpreted as a restriction on the use of better procedures or materials. Neither is this Standard intended to apply in any and all cases relating to the subject. Numerous external factors may negate the usefulness of this Standard in specific instances.

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## Foreword

Unit Committee T-5A ("Corrosion in Chemical Processes") of the National Association of Corrosion Engineers issues this Standard with a dual purpose.

The first purpose is to standardize, as much as possible, *simple immersion corrosion studies. In this sense*, this Standard is reasonable and effective without imposing inflexible requirements as to apparatus, conditions, or techniques. The actual conditions of test will be determined by the problem at hand and limited only by the ingenuity of the individual investigator.

The second *purpose* of this Standard is to present to the user a consensus on the best current technology in this field of laboratory corrosion testing. As such, this Standard enumerates and discusses the many factors which must be considered, controlled, and reported in order to aid in correlation or reproducibility of such studies.

The techniques described permit the investigator to reproduce to a considerable extent in the laboratory, through *judicious experimental design, the process conditions* which govern corrosion mechanisms. The tests are not to be construed as "accelerated" tests, which are generally unreliable. The methods described are also applicable to materials qualification tests for quality control. However, the latter require more rigid definition of apparatus, conditions, and technique.

The ultimate purpose is better correlation of results in the future and the reduction of conflicting reports through a more detailed recording of meaningful factors and conditions.

## TEST METHOD

## Laboratory Corrosion Testing of Metals for the Process Industries

CONTENTS

## 1. General

1.1 This Standard describes the factors which influence laboratory tests. These factors include specimen preparation, apparatus, test conditions (solution composition, temperature, velocity, aeration, volume, method of supporting specimens, duration of test), methods of cleaning specimens, evaluation of results, and calculation of corrosion rates. This Standard also emphasizes the importance of recording all pertinent data and provides a check list for reporting test data.

1.2 Experience has shown that all metals and alloys do not respond alike to the many factors that control corrosion and that "accelerated" corrosion tests give indicative results only. Consequently, it is impractical to propose an inflexible standard laboratory corrosion testing procedure for general use except for material qualification tests, where standardization is obviously required.

1.3 In designing any corrosion test, consideration must be given to the various factors discussed in this test method because these factors have been found to affect greatly the results obtained.

## 2. Specimen Preparation

2.1 In laboratory tests, corrosion rates of duplicate specimens are usually within ± 10% under the same test conditions. Occasional exceptions, in which a large difference is observed, can occur under conditions of borderline passivity of metals or alloys that depend on a passive film for their resistance to corrosion. Therefore, at least duplicate specimens should be exposed in each test.

2.2 If the effects of corrosion are to be determined by changes in mechanical properties, untested duplicate specimens should be preserved in a non-corrosive environment for comparison with the corroded specimens. The mechanical property commonly used for comparison is the tensile strength. The procedure for determining this value is shown in detail in ASTM Standard E-8-66.

2.3 The size and the shape of specimens will vary with the purpose of the test, nature of the materials, and apparatus used. A large surface-to-mass ratio and a small ratio of edge area to total area are desirable. These ratios can be achieved through the use of square or circular specimens of minimum thickness. Circular specimens should be cut preferably from sheet and not bar stock to minimize the exposed end grain.

2.3.1 A circular specimen of about 1 1/2-inch diameter is a convenient shape for laboratory corrosion tests. With a thickness of approximately 1/8 inch and a 5/16 or 7/16-inch diameter hole for mounting, these specimens will readily pass through a 45/50 ground glass joint of a distillation kettle. The total surface area of a circular specimen is given by the following equation:

$$A = \frac{\pi}{2}(D^2 - d^2) + t\pi D + t\pi d$$

where t = thickness, D = diameter of the specimen, and d = diameter of the mounting hole. If the hole is completely covered by the mounting support, the last term ($t\pi d$) in the equation is omitted.

2.3.2 Strip coupons (2 x 1 x 1/16 or 1/8 inch) may be preferred as corrosion specimens, particularly if interface or liquid line effects are to be studied by the laboratory test, but such effects are beyond the scope of this Standard.

2.3.3 All specimens should be measured carefully to permit accurate calculation of the exposed areas. An area calculation accurate to plus or minus 1% is usually adequate.

2.4 More uniform results may be expected if a substantial layer of metal is removed from the specimens to eliminate variations in condition of the original metallic surface. This can be done either by chemical treatment (pickling), electrolytic removal, or by grinding with a coarse abrasive paper or cloth, such as No. 50, using care not to work harden the surface (see Section 2.7). At least 0.0001 inch or 10 to 15 milligrams per square inch should be removed. If clad alloy specimens are to be used, special attention must be given to insure that excessive metal is not removed. After final preparation of the specimen surface, the specimens should be stored in a desiccator until exposure if they are not used immediately.

2.5 Exposure of sheared edges should be avoided unless *the purpose of the test is to study effects of the shearing* operation. It may be desirable to test a surface representative of the material and metallurgical condition used in practice.

2.6 The specimen can be stamped with an appropriate identifying mark.

2.6.1 The stamp, besides identifying the specimen, introduces stresses and cold work in the specimen, that could be responsible for localized corrosion and/or stress corrosion cracking.

2.6.2 Stress corrosion cracking at the identifying mark is a positive indication of susceptibility to such corrosion; however, the absence of cracking should not be interpreted as indicating resistance. Additional tests should be run to study specifically the effects of stress.

2.7 Final surface treatment of the specimens should include finishing with No. 120 abrasive paper or cloth, or the equivalent, unless the surface is to be used in the mill-finished condition. This resurfacing may cause some surface work-hardening to an extent which will be determined by the vigor of the surfacing operation but is not ordinarily significant.

2.7.1 Coupons of different alloy compositions should never be ground on the same cloth.

2.7.2 Wet grinding should be used on alloys which work harden quickly, such as the austenitic stainless steels.

2.8 The specimens should be finally degreased by scrubbing with bleach-free scouring powder, followed by thorough rinsing in water and in a suitable solvent (such as acetone, methanol, or a mixture of 50% methanol and 50% ether) and air dried. For relatively soft metals such as aluminum, magnesium, and copper, scrubbing with abrasive powder is not always needed and can mar the surface of the specimen. The use of towels for drying may introduce an error through contamination of the specimens with grease or lint.

2.9 The dried specimens should be weighed on an analytical balance to an accuracy of plus or minus 0.5 milligram.

2.10 The method of specimen preparation should be described when reporting tests results to facilitate interpretation of data by other persons.

2.10.1 Reports should include trade name or composition of specimens in the following order of preference: (a) chemical composition determined by analysis, (b) approximate or nominal chemical composition, and (c) trade name or grade and specification (if bought to MIL, ASTM, etc.).

2.10.2 Metallurgical condition of the specimens including the degree of hot or cold working and heat treatment, should be described as completely as possible.

2.11 The use of welded specimens is often desirable because some welds may be cathodic or anodic to the base metal and may affect the corrosion.

2.11.1 The heat-affected zone is also of importance but should be studied separately because welds on coupons do not faithfully reproduce heat input or size effects of full-size vessels.

2.11.2 Corrosion of a welded coupon is best reported by description and thickness measurements rather than a mils-per-year rate because the attack is normally localized and not representative of the entire surface.

2.11.3 A complete discussion of corrosion testing of welded coupons or the effect of heat treatment on the corrosion resistance of a metal is not within the scope of this Standard.

## 3. Equipment and Apparatus

3.1 A versatile and convenient apparatus should be used, consisting of a kettle or flask of suitable size (usually 500 to 5000 milliliters), a reflux condenser with atmospheric seal, a sparger for controlling atmosphere or aeration, a thermowell and temperature regulating device, a heating device (mantle, hot plate, or bath), and a specimen support system. If agitation is required, the apparatus can be modified to accept a suitable stirring mechanism such as a magnetic stirrer. A typical resin flask set up for this type test is shown in Figure 1.

3.2 These suggested components can be modified, simplified, or made more sophisticated to fit the needs of a particular investigation. The suggested apparatus is basic, and the apparatus is limited only by the judgment and ingenuity of the investigator.

3.2.1 A glass reaction kettle can be used where configuration and size of specimens will permit entry through the narrow kettle neck.

3.2.2 In some cases, a wide mouth jar with a suitable closure is sufficient when simple immersion tests at ambient temperatures are to be investigated.

3.2.3 Open beaker tests should not be used because of evaporation and contamination.

3.2.4 In more complex tests, provisions might be needed for continuous flow or replenishment of the corrosive liquid while simultaneously maintaining a controlled atmosphere.

## 4. *Test Conditions*

4.1 Selection of the conditions for a laboratory corrosion test will be determined by the purpose of the test.

4.1.1 If the test is to be a guide for the selection of a material for a particular purpose, the limits of controlling factors in service must be determined. These factors include oxygen concentration, temperature, rate of flow, pH value, and other important characteristics of the solution.

4.2 An effort should be made to duplicate all service conditions in the corrosion test.

4.3 It is important that test conditions be controlled throughout the test in order to ensure reproducible results.

4.4 The spread in corrosion rate values for duplicate specimens in a given test probably should not exceed ± 10% of the average when the attack is uniform.

4.5 Composition of solution.

4.5.1 Test solutions should be prepared accurately from chemicals conforming to the Standards of the Committee on Analytical Reagents of the American *Chemical Society,*[1] and *distilled* water, except in those cases where naturally occurring solutions or those taken directly from some plant process are used.

4.5.2 The composition of the test solution should be controlled to the fullest extent possible and should be described as completely and as accurately as possible when the results are reported.

4.5.2.1 Minor constituents should not be overlooked because they often affect corrosion rates.

4.5.2.2 Chemical content should be reported as percentage by weight of the solution. Molarity and normality are also helpful in defining the concentration of chemicals in the test solution.

4.5.3 The composition of the test solution should be checked by analysis at the end of the test to determine the extent of change in composition, such as might result from evaporation.

4.5.4 Evaporation losses should be controlled by a constant level device or by frequent additions of appropriate solution to maintain the original volume within ± 1%.

4.5.5 In some cases, composition of the test solution may change as a result of catalytic decomposition or by reaction with the test coupon. These changes should be determined if possible. Where required, the exhausted constituents should be added or a fresh solution provided, during the course of the test.

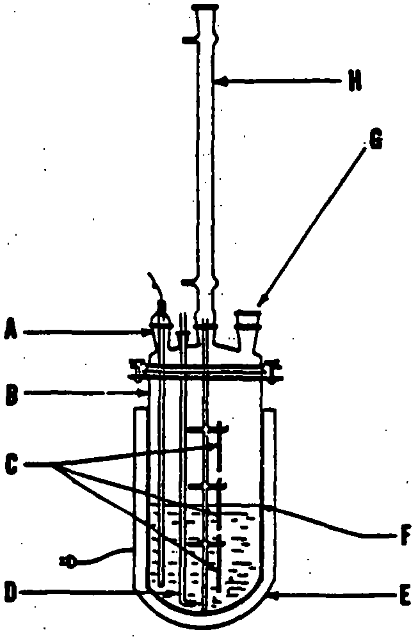


Figure 1 — Typical resin flask that can be used as a versatile and convenient apparatus to conduct simple immersion tests. Configuration of the flask top is such that more sophisticated apparatus can be added as required by the specific test being conducted. A = thermowell, B = resin flask, C = specimens hung on supporting device, D = gas inlet, E = heating mantle, F = liquid interface, G = opening in flask for additional apparatus that may be required, and H = reflux condenser.

4.5.6 When possible, only one type of metal should be exposed in a given test. If several different metals are exposed in the same solution, the corrosion products from one metal may affect the rate of attack on another metal. For example, copper corrosion products can reduce corrosion of stainless steel and titanium but can accelerate corrosion of aluminum.

4.6 *Temperature of solution.*

4.6.1 Temperature of the corroding solution should be controlled within ± 1 C (± 1.8 F) and must be *stated in the report of test results.*

4.6.2 If no specific temperature, such as boiling, is required or if a temperature range is to be investigated, the selected temperatures used in the test must be reported.

4.6.3 For tests at ambient temperatures, the tests should be conducted at the highest temperature anticipated for stagnant storage in summer months. This temperature may be as high as 40 to 45 C (104 to 113 F) in some areas. The variation in temperature should be reported also (e.g., 40 C ± 2 C).

4.7 Aeration of solution.

4.7.1 Unless specified, the solution should not be aerated. Most tests related to process equipment should be run with the natural atmosphere inherent in the process, such as the vapors of the boiling liquid.

4.7.2 If aeration is used, the specimens should not be located in the direct air stream from the sparger. Extraneous effects can be encountered if the air stream impinges on the specimens.

4.7.3 If complete exclusion of dissolved oxygen is necessary, specific techniques are required such as prior heating of the solution and sparging with an inert gas (usually nitrogen). A liquid atmospheric seal is required on the test vessel to prevent further contamination.

4.7.4 If oxygen saturation of the test solution is desired, this can best be achieved by sparging. For other degrees of aeration, the solution should be sparged with synthetic mixtures of air or oxygen with an inert gas.

4.8 Solution velocity.

4.8.1 The effect of velocity is not usually determined in normal laboratory tests although specific tests have been designed for this purpose. However, for the sake of reproducibility, some velocity control is desirable.

4.8.2 Tests at the boiling point should be conducted with minimum possible heat input, and boiling chips should be used to avoid excessive turbulence and bubble impingement.

4.8.3 In tests conducted below the boiling point, thermal convection generally is the only source of liquid velocity.

4.8.4 In test solutions with high viscosities, supplemental controlled stirring with a magnetic stirrer is recommended.

4.9 Volume of test solution.

4.9.1 The volume of the test solution should be large enough to avoid any appreciable change in its corrosiveness either through exhaustion of corrosive constituents or accumulation of corrosion products that might affect further corrosion.

4.9.2 A suitable volume-to-area ratio is 125 milliliters of solution per square inch of specimen surface. This corresponds to the recommendation of ASTM Standard A262-64T for the Huey Test.

4.9.3 The preferred volume-to-area ratio is 250 milliliters of solution per square inch of specimen surface as stipulated in ASTM Standard A-279-63 on "Total Immersion Corrosion Test of Stainless Steels."

4.9.4 When the test objective is to determine the effect of a metal or alloy on the characteristics of the test solution (for example, to determine the effects of metals on dyes), it is desirable to reproduce the ratio of solution volume to exposed metal surface that exists in practice. The actual time of contact of the metal with the solution also must be taken into account. Any necessary distortion of the test conditions must be considered when interpreting the results.

4.10 Method of supporting specimens.

4.10.1 The supporting device and container should not be affected by or cause contamination of the test solution.

4.10.2 The method of supporting specimens will vary with the apparatus used for conducting the test but should be designed to insulate the specimens from each other physically and electrically and to insulate the specimens from any metallic container or supporting device used with the apparatus.

4.10.3 Shape and form of the specimen support should assure free contact of the specimen with the corroding solution, the liquid line, or the vapor phase as shown in Figure 1. If clad alloys are exposed, special procedures will be required to insure that only the cladding is exposed unless the purpose is to test the ability of the cladding to protect cut edges in the test solution.

4.10.4 Some common supports are glass or ceramic rods, glass saddles, glass hooks, fluorocarbon plastic strings, and various insulated or coated metallic supports.

4.11 Duration of test.

4.11.1 Although duration of any test will be determined by the nature and purpose of the test an excellent procedure for evaluating the effect of time on corrosion of the metal and also on the corrosiveness of the environment in laboratory tests has been presented by Wachter and Treseder.[2] This technique is called the "Planned Interval Test," and the procedure and evaluation of results are given in Table 1. Other procedures that require the removal of solid corrosion products between exposure periods will not measure accurately the normal changes of corrosion with time.

4.11.2 Materials which experience severe corrosion generally do not need lengthy tests to obtain accurate corrosion rates. Although this assumption is valid in many cases, there are cases where it is not valid. For example, lead exposed to sulfuric acid corrodes at an extremely high rate at first while building a protective film, then the rates decrease considerably so that further corrosion is negligible. The phenomenon of forming a protective film is observed with many corrosion resistant materials, and therefore short tests on such materials would indicate a high corrosion rate and would be completely misleading.

4.11.3 Short time tests also can give misleading results on alloys that form passive films, such as stainless steels. With borderline conditions, a prolonged test may be needed to permit breakdown of the passive film and subsequently more rapid attack. Consequently, tests run for long periods are considerably more realistic than those conducted for short durations. This statement must be qualified by stating that corrosion should not proceed to the point where the original specimen size or the exposed area is drastically reduced or where the metal is perforated.

4.11.4 If anticipated corrosion rates are moderate or low, the following equation[2] gives a suggested test duration:

$$\text{Duration of test (hr)} = \frac{2000}{\text{corrosion rate (mpy)}}$$

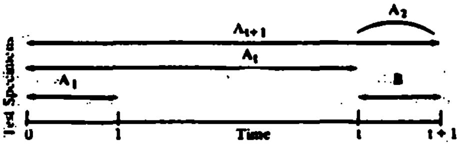
Examples: Where the corrosion rate is 10 mpy, the test should run for at least 200 hours. If the rate is 1 mpy, the duration should be at least 2000 hours.

4.11.4.1 This method of estimating test duration is useful only as an aid in deciding, after a test has been made, whether or not it is desirable to repeat the test for a longer period. The most common testing periods are 48 to 168 hours (2 to 7 days).

4.11.5 In some cases, it may be necessary to know the degree of contamination caused by the products of corrosion; this can be accomplished by analysis of

TABLE 1 – Planned Interval Test*

(Reprinted by permission from "Chemical Engineering Progress," June, 1947.)



Identical specimens all placed in the same corrosive fluid. Imposed conditions of the test kept constant for entire time t + 1. Letters, $A_1$, $A_t$, $A_{t+1}$, B, represent corrosion damage experienced by each test specimen. $A_2$ is calculated by subtracting $A_t$ from $A_{t+1}$.

| Occurrences During Corrosion Test | | Criteria |
|---|---|---|
| Liquid corrosiveness | unchanged | $A_1 = B$ |
| | decreased | $B < A_1$ |
| | increased | $A_1 < B$ |
| Metal corrodibility | unchanged | $A_2 = B$ |
| | decreased | $A_2 < B$ |
| | increased | $B < A_2$ |

Combinations of Situations

| Liquid Corrosiveness | Metal Corrodibility | Criteria |
|---|---|---|
| 1. unchanged | unchanged | $A_1 = A_2 = B$ |
| 2. unchanged | decreased | $A_2 < A_1 = B$ |
| 3. unchanged | increased | $A_1 = B < A_2$ |
| 4. decreased | unchanged | $A_2 = B < A_1$ |
| 5. decreased | decreased | $A_2 < B < A_1$ |
| 6. decreased | increased | $A_1 > B < A_2$ |
| 7. increased | unchanged | $A_1 < A_2 = B$ |
| 8. increased | decreased | $A_1 < B > A_2$ |
| 9. increased | increased | $A_1 < B < A_2$ |

Example of Planned Interval Corrosion Test

Conditions: Duplicate strips of low-carbon steel, each 3/4 by 3 in., immersed in 200 ml of 10% $AlCl_3$ - 90% $SbCl_3$ mixture through which dried HCl gas was slowly bubbled at atm. pressure. Temperature 90 C.

| | Interval, days | Wt. Loss, mg | Penetration, mils | Apparent Corrosion Rate, mils/yr |
|---|---|---|---|---|
| $A_1$ . . | 0-1 | 1080 | 1.69 | 620 |
| $A_t$ . . . | 0-3 | 1430 | 2.24 | 270 |
| $A_{t+1}$ . | 0-4 | 1460 | 2.29 | 210 |
| B . . . | 3-4 | 70 | 0.11 | 40 |
| $A_2$ . . | calc. 3-4 | 30 | 0.05 | 18 |

$A_2 < B < A_1$
$0.05 < 0.11 < 1.69$

Therefore, liquid markedly decreased in corrosiveness during test, and formation of partially protective scale on the steel was indicated.

the solution after corrosion has occurred. The corrosion rate can be calculated from the concentration of the matrix metal found in the solution, and it can be compared to that determined from the weight loss of the specimens. However, some of the corrosion products usually adhere to the specimen as a scale, and the corrosion rate calculated from the metal content in the solution is not always correct.

## 5. Methods of Cleaning Specimens After the Test

5.1 Before specimens are cleaned, their appearance should be observed and recorded. Location of deposits, variations in types of deposits, or variations in corrosion products are extremely important in evaluating localized corrosion, such as pitting and concentration cell attack.

5.2 Cleaning specimens after the test is a vital step in the corrosion test procedure and, if not done properly, can cause misleading results.

5.2.1 Generally, the cleaning procedure should

TABLE 2—Methods for Chemical Cleaning of Corrosion Test Specimens After Exposure

| Material | Chemical | Time | Temperature | Remarks |
|---|---|---|---|---|
| Aluminum and Aluminum Alloys | 70% $HNO_3$ | 2-3 min | Room | Follow by light scrub. |
| | or<br>2% $CrO_3$, 5% $H_3PO_4$, Soln. | 10 min | 175-185 F (79-85 C) | Used when oxide film resists $HNO_3$ treatment. Follow by 70% $HNO_3$ treatment previously described. |
| Copper and Copper Alloys | 15-20% HCl | 2-3 min | Room | Follow by light scrub. |
| | or<br>5-10% $H_2SO_4$ | 2-3 min | Room | Follow by light scrub. |
| Lead and Lead Alloys | 1% acetic acid | 10 min | Boiling | Follow by light scrub. Removes PbO. |
| | or<br>5% ammonium acetate | 5 min | Hot | Follow by light scrub. Removes PbO and/or $PbSO_4$. |
| | or<br>80 g/l NaOH,<br>50 g/l mannitol,<br>0.62 g/l hydrazine sulfate | 30 min, or until clean | Boiling | Follow by light scrub. |
| Iron and Steel | 20% NaOH, 200 g/l zinc dust | 5 min | Boiling | ... |
| | or<br>*conc. HCL 50 g/l*<br>$SnCl_2$ + 20 g/l $SbCl_3$ | Until clean | Cold | ... |
| Magnesium and Magnesium Alloys | 15% $CrO_3$, 1% $AgCrO_4$ Soln. | 15 min. | Boiling | ... |
| Nickel and Nickel Alloys | 15-20% HCl | Until clean | Room | ... |
| | or<br>10% $H_2SO_4$ | Until clean | Room | ... |
| Stainless Steel | 10% $HNO_3$ | Until clean | 140 F (60 C) | Avoid contamination with chlorides |
| Tin and Tin Alloys | 15% $Na_3PO_4$ | 10 min | Boiling | Follow by scrubbing. |
| Zinc | 10% $NH_4Cl$<br>followed by<br>5% $CrO_3$, 1% $AgNO_3$ Soln. | 5 min<br>20 sec | Room<br>Boiling | Follow by light scrubbing.<br>... |
| | or<br>*Saturated ammonium acetate* | Until clean | Room | Follow by light scrub. |
| | or<br>100 g/l NaCN | 15 min | Room | ... |

remove all corrosion products from specimens with a minimum removal of sound metal.

5.2.2 Set rules cannot be applied to specimen cleaning because procedures will vary depending on the type of metal being cleaned and on the degree of adherence of corrosion products.

5.3 Cleaning methods can be divided into three general categories: mechanical, chemical, and electrolytic.

5.3.1 Mechanical cleaning includes scrubbing, scraping, brushing, mechanical shocking, and ultrasonic procedures. Scrubbing with a bristle brush and mild abrasive is the most popular of these methods; the others are used principally as a supplement to remove heavily encrusted corrosion products before scrubbing. Care should be used to avoid the removal of sound metal.

5.3.2 Chemical cleaning implies the removal of material from the surface of the specimen by dissolution in an appropriate chemical solution. Solvents such as acetone, carbon tetrachloride, and alcohol, are used to remove oil, grease, or resin and are usually applied prior to other methods of cleaning. Chemicals are chosen for application to a specific material. Some of these treatments in general use are outlined in Table 2.

5.3.3 Electrolytic cleaning should be preceded by scrubbing to remove loosely adhering corrosion products. One method of electrolytic cleaning that has been found to be useful for many metals and alloys is as follows:

| | |
|---|---|
| Solution | 5% (by weight) $H_2SO_4$ |
| Anode | Carbon or lead |
| Cathode | Test specimen |
| Cathode C.D. | 20 amp/dm$^2$<br>(129 amp/sq in) |
| Inhibitor | 2 cc organic inhibitor per liter |
| Temperature | 74 C (165 F) |
| Exposure period | 3 minutes |

5.3.3.1 Precautions must be taken to insure good electrical contact with the specimen, to avoid contamination of the solution with easily reducible metal ions, and to insure that inhibitor decomposition has not occurred. Instead of using 2 milliliters of any proprietary inhibitor, 0.5 gram per liter of inhibitors such as diorthotolyl thiourea or quinoline ethiodide can be used.

5.4 Whatever treatment is used to clean specimens after a corrosion test, its effect in removing metal should be determined, and the weight loss should be corrected accordingly. A "blank" specimen should be weighed before and after exposure to the cleaning procedure to establish this weight loss.

5.4.1 Following removal of all scale, the specimen should be treated as discussed in Section 2.8.

5.4.2 A description of the cleaning method should be included with the data reported.

## 6. Evaluation of Results

6.1 After corroded specimens have been cleaned, they should be reweighed with an accuracy corresponding to that of the original weighing. The weight loss during the test period can be used as the principal measure of corrosion.

6.2 After the specimens have been reweighed, they should be examined carefully for the presence of pits. If there are pits, the average and maximum depths of pits are determined after measurement with a pit gauge or a calibrated microscope which can be focused first on the edge and then on the bottom of the pit. An excellent discussion of pitting corrosion has been published.[3]

6.2.1 Pit depths should be reported in millimeters or thousandths of an inch for the test period and not interpolated or extrapolated to millimeters per year or thousandths of an inch per year or any other arbitrary period because rarely, if ever, is the rate of initiation or propagation of pits uniform.

6.2.2 The size, shape, and distribution of pits should be noted. A distinction should be made between those occurring underneath the supporting devices (concentration cells) and those on surfaces that were freely exposed to the test solution.

6.3 If the material being tested is suspected of being subject to dealloying forms of corrosion such as dezincification, or to intergranular attack, a cross section of the specimen should be microscopically examined to determine the type and depth of such attack.

6.4 The specimen may be subjected to simple bending tests to determine whether any embrittlement has occurred.

6.5 It may be desirable to make quantitative mechanical tests to compare the exposed specimens with uncorroded specimens reserved for the purpose, as described in Section 2.2.

### 7. Calculating Corrosion Rates

7.1 The calculation of corrosion rates requires several pieces of information and several assumptions.

7.1.1 The use of corrosion rates implies that all weight loss has been due to general corrosion and not to localized corrosion, such as pitting or sensitized areas on welded coupons. Localized corrosion is reported separately.

7.1.2 The use of corrosion rates also implies that the material has not been internally attacked as by dezincification or intergranular corrosion.

7.1.3 Internal attack can be expressed as a corrosion rate if desired. However, the calculations must not be based on weight loss, which is usually small, but on microsections which show depth of attack.

7.2 Assuming that localized or internal corrosion is not present or are recorded separately in the report, the corrosion rate expressed as mils penetration per year (mpy) or millimeters per year (mmpy) can be calculated by the equations:

$$\text{mpy} = \frac{\text{wt loss} \times 534}{(\text{area})\,(\text{time})\,(\text{metal density})}$$

$$\text{mmpy} = \frac{\text{wt loss} \times 13.56}{(\text{area})\,(\text{time})\,(\text{metal density})}$$

where weight loss is in milligrams, area is square inches of metal surface exposed, and time is hours exposed.

Metal density of many common alloys (expressed in grams per cubic centimeter) is listed in Table 3. The density for new or unlisted alloys can be obtained from the producer or from various metal handbooks.

TABLE 3 - *Density of Common Metals for Use in Corrosion Rate Calculations*[a]

| Alloy | Density, g/cc |
|---|---|
| **Aluminum** | |
| 99.0 + Al | 2.71 |
| Al, 1.2 Mn | 2.73 |
| Al, 1.0 Mg, 0.6 Si, 0.25 Cr | 2.70 |
| **Brass** | |
| 85 Cu, 15 Zn | 8.75 |
| 71 Cu, 28 Zn, 1 Sn | 8.53 |
| 65 Cu, 35 Zn | 8.47 |
| 60 Cu, 39.25 Zn, 0.75 Sn | 8.41 |
| **Bronze** | |
| 95 Cu, 5 Sn | 8.86 |
| 90 Cu, 10 Sn | 8.78 |
| 85 Cu, 5 Sn, 5 Zn, 5 Pb | 8.80 |
| 94.8 Cu, 3 Si | 8.53 |
| 95 Cu, 5 Al | 8.17 |
| 85-90 Cu, 10 Al | 7.58 |
| **Copper** | |
| 99.90 Cu, 0.01 P | 8.91 |
| **Cupro-Nickel** | |
| 90 Cu, 10 Ni | 8.93 |
| 70 Cu, 30 Ni | 8.94 |
| **Iron** | |
| 94 Fe, 3.5 C, 2.5 Si | 7.00 |
| 96 Fe, 3.0 C | 7.60 |
| 99.94 Fe, 0.025 S, 0.017 Mn, 0.012 C, 0.005 P | 7.86 |
| 84.3 Fe, 14.5 Si, 0.35 Mn, 0.85 C | 7.00 |
| **Lead** | |
| 99.90 + Pb | 11.34 |
| **Nickel** | |
| 99.4 Ni + Co | 8.89 |
| 67 Ni, 30 Cu | 8.84 |
| 62 Ni, 30 Mo, 5 Fe | 9.24 |
| 58 Ni, 17 Mo, 15 Cr, 5 W, 5 Fe | 8.94 |
| 80 Ni, 14 Cr, 6 Fe | 8.51 |
| ***Steel*** | |
| 0.20 C, Mn, P, S | 7.85 |
| **Stainless Steel** | |
| 11.50-13.50 Cr, 0.15 C | 7.75 |
| 14.00-18.00 Cr, 0.12 C | 7.70 |
| 18.00-20.00 Cr, 8.00-12.00 Ni, 0.08 C | 7.93 |
| 16.00-18.00 Cr, 10.00-14.00 Ni, 2.00-3.00 Mo, 0.08 C | 7.98 |
| 17.00-19.00 Cr, 9.00-12.00 Ni, 0.08 C, Ti | 8.02 |
| 17.00-19.00 Cr, 9.00-12.00 Ni, 0.08 C, Cb | 8.02 |
| 19.00-21.00 Cr, 24.00-30.00 Ni, 2.00-3.00 Mo, 3.00-4.00 Cu | 8.00 |
| **Tantalum** | 16.60 |
| **Tin** | 7.30 |
| **Titanium** | 4.54 |
| **Zirconium** | 6.53 |

## 8. Reporting the Data

8.1 The importance of reporting all data as completely as possible cannot be overemphasized.

8.2 Expansion of the testing program in the future or correlating the results with tests of other investigators will be possible only if all pertinent information is properly recorded.

8.3 The following checklist is a recommended guide for reporting all important information and data:

8.3.1 Corrosive media and concentration (changes during test).

8.3.2 Volume of test solution.

8.3.3 Temperature (maximum, minimum, average).

8.3.4 Aeration (describe conditions or technique).

8.3.5 Agitation (describe conditions or technique).

8.3.6 Type of apparatus used for test.

8.3.7 Duration of each test.

8.3.8 Chemical composition or trade name of metals tested.

8.3.9 Form and metallurgical conditions of specimens.

8.3.10 Exact size, shape, and area of specimens.

8.3.11 Treatment used to prepare specimens for test.

8.3.12 Number of specimens of each material tested, and whether specimens were tested separately or which specimens were tested in the same container.

8.3.13 Method used to clean specimens after exposure and the extent of any error expected by this treatment.

8.3.14 Actual weight losses for each specimen.

8.3.15 Evaluation of attack if other than general, such as crevice corrosion under support rod, pit depth and distribution, and results of microscopic examination or bend tests.

8.3.16 Corrosion rates for each specimen expressed as mils per year.

8.4 Minor occurrences or deviations from the proposed test program often can have significant effects and should be reported if known.

8.5 Statistics can be a valuable tool for analyzing the results from test programs designed to generate adequate data and should be used wherever possible. Excellent references for the use of statistics in corrosion studies include References 4 through 8.

## References

1. Joseph Rosin. Reagent Chemicals and Standards. Fifth Edition. D. Van Nostrand Co., Inc., 120 Alexander St., Princeton, N. J., 1966.
2. A. Wachter and R. S. Treseder. Corrosion Testing Evaluation of Metals for Process Equipment. *Chemical Engineering Progress.* 43, 315-326 (1947) June.
3. N. D. Greene and M. G. Fontana. A Critical Analysis of Pitting Corrosion. *Corrosion*, 15, 25t (1959) January.
4. Harold S. Mickley, Thomas K. Sherwood, and Charles E. Reed (editors). Applied Mathematics in Chemical Engineering, (second edition). McGraw-Hill, New York, N. Y., 1957.
5. D. M. Douglas. Are You Using or Abusing Data? Paper presented at the Northeast-North Central Region Conference, National Association of Corrosion Engineers, October 4-6, 1965, Pittsburgh, Pa.
6. W. J. Youden. Experimentation and Measurement. National Science Teachers Association, Washington, D. C., 1962.
7. E. F. Booth and G. E. G. Tucker. Statistical Distribution of Endurance in Electrochemical Stress-Corrosion Tests. *Corrosion*, 21, 173 (1965) May.
8. Compilation of Examples of Applying Statistics to Corrosion Problems. Report of ASTM Committee G-1 on Corrosion of Metals, prepared by Subcommittee III on Statistics. ASTM, 1916 Race St., Philadelphia, Pa.
9. Metals Handbook, American Society for Metals, Metals Park, Novelty, Ohio, 8th Edition, Volume 1, 1961.

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.1210 APPENDIX H.

**Title 10 C.F.R. Waste Disposal**

**§ 20.301 General requirement.**

No licensee shall dispose of licensed material except:

(a) By transfer to an authorized recipient as provided in the regulations in Part 30, 40, or 70 of this chapter, whichever may be applicable; or

(b) As authorized pursuant to § 20.302; or

(c) As provided in § 20.303 or § 20.304, applicable respectively to the disposal of licensed material by release into sanitary sewerage systems or burial in soil, or in § 20.106 (Radioactivity in effluents to unrestricted areas).

[25 FR 10914, Nov. 17, 1960, as amended at 39 FR 27121, July 25, 1974]

**§ 20.302 Method for obtaining approval of proposed disposal procedures.**

(a) Any licensee or applicant for a license may apply to the Commission for approval of proposed procedures to dispose of licensed material in a manner not otherwise authorized in the regulations in this chapter. Each application should include a description of the licensed material and any other radioactive material involved, including the quantities and kinds of such material and the levels of radioactivity involved, and the proposed manner and conditions of disposal. The application should also include an analysis and evaluation of pertinent information as to the nature of the environment, including topographical, geological, meteorological, and hydrological characteristics; usage of ground and surface waters in the general area; the nature and location of other potentially affected facilities; and procedures to be observed to minimize the risk of unexpected or hazardous exposures.

(b) The Commission will not approve any application for a license to receive licensed material from other persons for disposal on land not owned by the Federal government or by a State government.

(c) The Commission will not approve any application for a license for disposal of licensed material at sea unless the applicant shows that sea disposal offers less harm to man or the environment than other practical alternative methods of disposal.

[25 F.R. 10914, Nov. 17, 1960, as amended at 26 F.R. 352, Jan. 18, 1961; 36 F.R. 23138, Dec. 4, 1971]

**§ 20.303 Disposal by release into sanitary sewage systems.**

No licensee shall discharge licensed material into a sanitary sewerage system unless:

(a) It is readily soluble or dispersable in water; and

(b) The quantity of any licensed or other radioactive material released into the system by the licensee in any one day does not exceed the larger of subparagraphs (1) or (2) of this paragraph:

(1) The quantity which, if diluted by the average daily quantity of sewage released into the sewer by the licensee, will result in an average concentration equal to the limits specified in Appendix B, Table I, Column 2 of this part; or

(2) Ten times the quantity of such material specified in Appendix C of this part; and

(c) The quantity of any licensed or other radioactive material released in any one month, if diluted by the average monthly quantity of water released by the licensee, will not result in an average concentration exceeding the limits specified in Appendix B, Table I, Column 2 of this part; and

(d) The gross quantity of licensed and other radioactive material released into the sewerage system by the licensee does not exceed one curie per year.

Excreta from individuals undergoing medical diagnosis or therapy with radioactive material shall be exempt from any limitations contained in this section.

**§ 20.304 Disposal by burial in soil.**

No licensee shall dispose of licensed material by burial in soil unless:

(a) The total quantity of licensed and other radioactive materials buried at any one location and time does not exceed, at the time of burial, 1,000 times the amount specified in Appendix C of this part; and

(b) Burial is at a minimum depth of four feet; and

(c) Successive burials are separated by distances of at least six feet and not more than 12 burials are made in any year.

**§ 20.305 Treatment or disposal by incineration.**

No licensee shall treat or dispose of licensed material by incineration except as specifically approved by the Commission pursuant to §§ 20.106(b) and 20.302.

[29 FR 14435, Oct. 21, 1964]

**§ 20.106 Radioactivity in effluents to unrestricted areas.**

(a) A licensee shall not possess, use, or transfer licensed material so as to release to an unrestricted area radioactive material in concentrations which exceed the limits specified in Appendix "B", Table II of this part, except as authorized pursuant to § 20.302 or paragraph (b) or this section. For purposes of this section concentrations may be averaged over a period not greater than one year.

(b) An application for a license or amendment may include proposed limits higher than those specified in paragraph (a) of this section. The Commission will approve the proposed limits if the applicant demonstrates:

(1) That the applicant has made a reasonable effort to minimize the radioactivity contained in effluents to unrestricted areas; and

(2) That it is not likely that radioactive material discharged in the effluent would result in the exposure of an individual to concentrations of radioactive material in air or water exceeding the limits specified in Appendix "B", Table II of this part.

(c) An application for higher limits pursuant to paragraph (b) of this section shall include information demonstrating that the applicant has made a reasonable effort to minimize the radioactivity discharged in effluents to unrestricted areas, and shall include, as pertinent:

(1) Information as to flow rates, total volume of effluent, peak concentration of each radionuclide in the effluent, and concentration of each radionuclide in the effluent averaged over a period of one year at the point where the effluent leaves a stack, tube, pipe, or similar conduit;

(2) A description of the properties of the effluents, including:

(i) chemical composition:

(ii) physical characteristics, including suspended solids content in liquid effluents, and nature of gas or aerosol for air effluents;

(iii) the hydrogen ion concentrations ($p^H$) of liquid effluents; and

(iv) the size range of particulates in effluents released into air.

(3) A description of the anticipated human occupancy in the unrestricted area where the highest concentration of radioactive material from the effluent is expected, and, in the case of a river or stream, a description of water uses downstream from the point of release of the effluent.

(4) Information as to the highest concentration of each radionuclide in an unrestricted area, including anticipated concentrations averaged over a period of one year:

(i) In air at any point of human occupancy; or

(ii) In water at points of use downstream from the point of release of the effluent.

(5) The background concentration of radionuclides in the receiving river or stream prior to the release of liquid effluent.

(6) A description of the environmental monitoring equipment, including sensitivity of the system, and procedures and calculations to determine concentrations of radionuclides in the unrestricted area and possible reconcentrations of radionuclides.

(7) A description of the waste treatment facilities and procedures used to reduce the concentration of radionuclides in effluents prior to their release.

(d) For the purpose of this section the concentration limits a Appendix "B", Table II of this part shall apply at the boundary of the restricted area. The concentration of radioactive material discharged through a stack, pipe or similar conduit may be determined wih respect to the point where the material leaves the conduit. If the conduit discharges within the restricted area, the concentration at the boundary may be determined by applying appropriate factors for dilution, dispersion, or decay between the point of discharge and the boundary.

(e) In addition to limiting concentrations in effluent streams, the Commission may limit quantities of radioactive materials released in air or water during a specified period of time if it appears that the daily intake of radioactive material from air, water, or food by a suitable sample of an exposed population group, averaged over a period not exceeding one year, would otherwise exceed the daily intake resulting from continuous exposure to air or water containing one-third the concentration of radioactive materials specified in Appendix "B", Table II of this part.

(f) The provisions of this section do not apply to disposal of radioactive material into sanitary sewerage systems, which is governed by § 20.303.

[29 F.R. 14434, Oct. 21, 1964]

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.1220 APPENDIX I.

**Title 16 C.F.R.**

**§ 1500.41 Method of testing primary irritant substances.**

Primary irritation to the skin is measured by a patch-test technique on the abraded and intact skin of the albino rabbit, clipped free of hair. A minimum of six subjects are used in abraded and intact skin tests. Introduce under a square patch, such as surgical gauze measuring 1 inch by 1 inch and two single layers thick, 0.5 milliliter (in the case of liquids) or 0.5 gram (in the case of solids and semisolids) of the test substance. Dissolve solids in an appropriate solvent and apply the solution as for liquids. The animals are immobilized with patches secured in place by adhesive tape. The entire trunk of the animal is then wrapped with an impervious material, such as rubberized cloth, for the 24-hour period of exposure. This material aids in maintaining the test patches in position and retards the evaporation of volatile substances. After 24 hours of exposure, the patches are removed and the resulting reactions are evaluated on the basis of the designated values in the following table:

| Skin reaction | Value[1] |
|---|---|
| Erythema and eschar formation: | |
| No erythema | 0 |
| Very slight erythema (barely perceptible) | 1 |
| Well-defined erythema | 2 |
| Moderate to severe erythema | 3 |
| Severe erythema (beet redness) to slight eschar formations (injuries in depth) | 4 |
| Edema formation: | |
| No edema | 0 |
| Very slight edema (barely perceptible) | 1 |
| Slight edema (edges of area well defined by by definite raising) | 2 |
| Moderate edema (raised approximately 1 millimeter) | 3 |
| Severe edema (raised more than 1 millimeter and extending beyond the area of exposure) | 4 |

Readings are again made at the end of a total of 72 hours (48 hours after the first reading). An equal number of exposures are made on areas of skin that have been previously abraded. The abrasions are minor incisions through the stratum corneum, but not sufficiently deep to disturb the derma or to produce bleeding. Evaluate the reactions of the abraded skin at 24 hours and 72 hours, as described in this paragraph. Add the values for erythema and eschar formation at 24 hours and at 72 hours for intact skin to the values on abraded skin at 24 hours and at 72 hours (four values). Similarly, add the values for edema formation at 24 hours and at 72 hours for intact and abraded skin (for values). The total of the eight values is divided by four to give the primary irritation score; for example:

| Skin reaction | Exposure time (hours) | Evaluation value |
|---|---|---|
| Erythema and eschar formation: | | |
| Intact skin | 24 | 2 |
| Do | 72 | 1 |
| Abraded skin | 24 | 3 |
| Do | 72 | 2 |
| Subtotal | | 8 |
| Edema formation: | | |
| Intact skin | 24 | 0 |
| Do | 72 | 1 |
| Abraded skin | 24 | 1 |
| Do | 72 | 2 |
| Subtotal | | 4 |
| Total | | 12 |

Thus, the primary irritation score is 12+4=3.

**Statutory Authority:** *MS s 116.07 subd 4*

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[1] The "value" recorded for each reading is the average *value of the six or more animals subject to the test.*

## 7045.1230 APPENDIX J.

**Title 49 C.F.R.**

**§ 173.50 An explosive.**

(a) For the purpose of Parts 170-189 of this subchapter an explosive is defined as any chemical compound, mixture, or device, the primary or common purpose of which is to function by explosion, i.e., with substantially instantaneous release of gas and heat, unless such compound, mixture, or device is otherwise specifically classified in Parts 170-189 of this subchapter.

**§ 173.300 Definitions.**

For the purpose of Parts 170-189 of this chapter, the following terminology is defined:

(a) *Compressed gas*. The term "compressed gas" shall designate any material or mixture having in the container an absolute pressure exceeding 40 p.s.i. at 70°F. or, regardless of the pressure at 70°F., having an absolute pressure exceeding 104 p.s.i. at 130°F.; or any liquid flammable material having a vapor pressure exceeding 40 p.s.i. absolute at 100°F. as determined by ASTM Test D-323.

(b) *Flammable compressed gas*. Any compressed gas as defined in paragraph (a) of this section shall be classified as "flammable compressed gas" if any one of the following occurs:

(1) Either a mixture of 13 percent or less (by volume) with air forms a flammable mixture or the flammable range with air is wider than 12 percent regardless of the lower limit. These limits shall be determined at atmospheric temperature and pressure. The method of sampling and test procedure shall be acceptable to the Bureau of Explosives.

(2) Using the Bureau of Explosives' Flame Projection Apparatus (see Note 1), the flame projects more than 18 inches beyond the ignition source with valve opened fully, or the flame flashes back and burns at the valve with any degree of valve opening.

(3) Using the Bureau of Explosives' Open Drum Apparatus (see Note 1) there is any significant propagation of flame away from the ignition source.

(4) Using the Bureau of Explosives' Closed Drum Apparatus (see Note 1), there is any explosion of the vapor-air mixture in the drum.

(c) *Non-liquefied compressed gas*. A "non-liquefied compressed gas" is a gas, other than gas in solution, which under the charged pressure is entirely gaseous at a temperature of 70°F.

(d) *Liquefied compressed gas*. A "liquefied compressed gas" is a gas which, under the charged pressure, is partially liquid at a temperature of 70°F.

(e) *Compressed gas in solution*. A "compressed gas in solution" is a non-liquefied compressed gas which is dissolved in a solvent.

(f) *Flammable range*. The term "flammable range" shall designate the difference between the minimum and maximum volume percentages of the material in air that forms a flammable compressed gas.

(g) *Filling density*. The term "filling density" shall designate the percent ratio of the weight of gas in a container to the weight of water that the container will hold at 60°F. (One pound of water equals 27.737 cubic inches at 60°F.) For example, for a liquefied petroleum gas of 0.504/0.510 specific gravity, a 100-pound cylinder holds 238.1 pounds of water and the filling density is 42 percent; therefore the amount of gas permitted is 0.42×238.1 or 100 pounds.

(h) *Service pressure*. The term "service pressure" shall designate the authorized pressure marking on the container. For example, for cylinders marked "DOT 3A1800", the service pressure is 1800 psig (pounds per square inch gauge).

[29 F.R. 18743, Dec. 29, 1964, as amended by Amdt. 173-16, 34 F.R. 18248, Nov. 14, 1969; Amdt. 173-54, 36 F.R. 18469, Sept. 15, 1971]

**§ 173.300 Definitions.**

(b) *Flammable compressed gas*. Any compressed gas as defined in paragraph (a) of this section shall be classed as "flammable gas" if any one of the following occurs:

**§ 177.824 Retesting and inspection of cargo tanks.**

(a) *General*. In accordance with § 173.33 of this chapter (cargo tank use authorization) every cargo tank and every compartment of a cargo tank authorized as a qualified container, except those cargo tanks having a capacity of 3,000 gallons or less used exclusively for the transportation of flammable liquids, must fulfill the applicable requirements as set forth in this section.

(1) Each cargo tank, except specifications MC 330 and MC 331 cargo tanks, must be in compliance with the testing requirements prescribed in paragraphs (a), (b),

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NOTE 1: A description of the Bureau of Explosives' Flame Projection Apparatus, Open Drum Apparatus, Closed Drum Apparatus, and method of tests may be procured from the Bureau of Explosives.

(c), and (d) of this section. Each cargo tank must be in accordance with the marking requirement of paragraph (h) of this section.

(2) Every cargo tank whether constructed in accordance with DOT specifications or being operated as a novel tank under special permit authorization shall not continue in service unless it has successfully fulfilled the testing requirements as set forth in this section.

(b) *Visual inspection requirements*. Every cargo tank shall receive an external visual inspection at least once in ever 2-year period. The first such inspection shall be required at the time the next hydrostatic retest is due or prior to January 31, 1969, whichever comes first. This inspection shall be made by a responsible and experienced inspector who shall record the condition of the items set forth below. The inspection record shall be approved and signed by an authorized representative of the owner or operator. A written report of each inspection shall be retained in carrier's or owner's files for a period of 2 years after the date of inspection. Where insulation precludes external visual inspection, the cargo tank shall receive a visual internal inspection for corroded areas, defects in welds or tank sheets. Where visual inspection is precluded by both internal coating and external insulation or when the cargo tank is not equipped with a manhole, the tank shall be hydrostatically tested at 5-year intervals except as otherwise provided in paragraph (c) of this section. The tank shall not be placed in or returned to service if evidence of any unsafe condition is discovered and until such condition has been corrected. Inspection shall consist of the following items:

(1) The tank shall be inspected for: Corroded areas, bad dents, and defects in welds; defects in piping, valves, and gaskets; and other conditions, including leakage, which indicate weakness in the tank that might render it unsafe for transportation service.

(2) Devices for tightening manhole covers must be operative and leakage at manhole covers and gaskets must be corrected.

(3) Spring-loaded safety-relief valves rated in excess of 7 p.s.i.g. shall be removed from the cargo tank and tested.

(4) All emergency devices and valves must be free from corrosion, distortion, and any damage which will prevent their normal operation.

(5) Missing or loose bolts or nuts on any flanged connection or blank flange must be replaced or tightened.

(6) Required markings on the tank shall be legible.

(7) The entire vehicle shall be inspected for and comply with the Motor Carrier Safety Regulations, Part 393, Chapter III, of this title.

(c) *Hydrostatic or pneumatic testing requirements*. In addition to the visual inspection requirements as contained in paragraph (b) of this section, hydrostatic or pneumatic testing of cargo tanks (or compartments) is required to be conducted in accordance with the provisions contained in paragraph (d) of this section to qualify as an authorized container if:

(1) The cargo tank has been out of service (transporting dangerous articles) 1 year or more, or

(2) The cargo tank has been involved in an accident in which it may have been dented, torn, or otherwise damaged so as to affect its product retention integrity, or

(3) The shell of the cargo tank as originally manufactured has been modified, or

(4) The cargo tank is operating under special permit authorization. Such tanks shall be hydrostatically tested once every calendar year unless otherwise provided for in the special permit, and shall successfully fulfill the requirements set forth in paragraph (d) of this section. No two such required tests shall be closer than 6 months.

(d) *Hydrostatic or pneumatic testing procedure*. The requalification as an authorized container of cargo tanks (or compartments) required to be hydrostatically or pneumatically tested in accordance with paragraph (a) of this section shall be based on successfully meeting the requirements of this paragraph.

(1) *General*. (i) *If a cargo tank is compartmented* each compartment shall be similarly tested with the adjacent compartment empty and at atmospheric pressure.

(ii) All closures shall be in place while the test is being made. During the test all relief devices shall be clamped, plugged or otherwise rendered inoperative. Relief devices shall be returned to their operative condition immediately after the tests are completed.

(iii) The tank or compartment must hold the prescribed pressure for at least 10 minutes. All tank valves, piping, and other accessories in communication with the lading must be pressure tested and proven tight at the tank design pressure.

(iv) All pressure bearing portions of the heating system of a cargo tank (or compartment) employing such media as steam or hot water for heating the lading shall be tested under hydrostatic pressure and proven to be tight at 14.06 kg./sq. cm (200 psig). Systems employing flues for heating the lading shall be suitably tested to insure against product leakage into the flues or into the atmosphere.

(2) *Hydrostatic test*. For Hydrostatic testing, the tank (including its domes, if any) must be completely filled with water or a liquid having a viscosity similar to water. Pressure must be gauged at the top of the tank applied in accordance with Table I following paragraph (d)(3) of this section.

(3) *Pneumatic test*. Pneumatic pressure must be applied in accordance with Table I of this paragraph. During the pneumatic test the entire surface of all joints under pressure must be coated with a solution of soap and water, heavy oil, or other materials suitable for the purpose of foaming or bubbling to indicate the presence of leaks. Other methods equally sensitive for determining leaks may be used.

TABLE I

| Container type: | *Test pressure KG/SQ. CM.* |
|---|---|
| MC 300, 301, 302, 303, 305, 306 | 0.2109 (3 psig) |
| MC 304, 307 | 1.76[1] (25[1] psig) |
| MC 310, 311, 312 | 0.2109[1] (3[1] psig) |

[1] Or 1½ times design pressure whichever is greater.

(4) *Required results*. A cargo tank (or compartment) required to be hydrostatically or pneumatically tested in accordance with paragraph (a) of this section may not be returned to service as a specification cargo tank unless it has successfully retained the applicable test pressure (see Table I in paragraph (d) (3) of this section) without leakage, undue distortion, excessive permanent expansion, or evidence of impending failure. The suitability of any repairs shall be determined by the same method of test.

(i) Cargo tanks (or compartments) with heating systems shall successfully withstand the hydrostatic pressure and examination specified in paragraph (d) (1) (iv) of this section.

(e) *Compressed gas cargo tanks, specifications MC 330 and MC 331*. Each cargo tank constructed in compliance with specification MC 330 or MC 331 (§ 178.337 of this subchapter) must be inspected and tested in accordance with § 173.33 of this subchapter.

(f) *Reporting requirements*. Each motor carrier shall file with the Director, Bureau of Motor Carrier Safety, Federal Highway Administration, Department of Transportation, Washington, D.C. 20590, a written listing of all MC 330 and MC 331 cargo tanks he has in service. Each motor carrier, upon placing in service or withdrawing from service any MC 330 and MC 331 cargo tank (other than a cargo tank used in interchange service which is reported upon by another carrier), shall file a supplemental report with the Bureau.

(1) The initial listing and each subsequent report must include the following information:

(i) The carrier's name, address, and telephone number.

(ii) One of the following statements: "Cargo tank placed in service" or "Cargo tank withdrawn from service;" as appropriate, followed by the date of placement or removal;

(iii) The carrier's equipment number, manufacturer's name, manufacturer's serial number, specification MC 330 or MC 331, and "QT" (quenched and tempered) or "NQT" (not quenched and tempered).

(2) A copy of each report required by this paragraph must be retained by the carrier at its principal place of business during the period the tank is in the carrier's service and for 1 year thereafter. However, upon a written request to, and with the approval of, the Director, Regional Motor Carrier Safety Office, for the region in which a motor carrier has his principal place of business, the carrier may maintain the reports at a regional or terminal office.

(g) *Special testing required by the Department*. Upon the showing of probable cause of the necessity for retest, the Department may require any cargo tank to be retested at any time in accordance with the requirements prescribed for its periodic retest.

(h) *Test date markings*. The month and year of the last test must be durably and legibly marked on the tank in letters not less than 1¼ inches high, on the right side near the front. These markings must be near the metal certification plate, except on any tank having the plate other than on the right side near the front.

(i) *Withdrawal of certification*. If, as the result of an accident or for any other reason a cargo tank no longer meets the applicable specification, the carrier shall remove the metal certification plate or make it illegible (see § 173.24(c)(1)(v) of this subchapter). The details of the conditions necessitating withdrawal of the certification must be recorded and signed on the written certificate for that cargo tank. The vehicle owner shall retain the certificate for at least 1 year after withdrawal of the certification.

## Subpart C—Loading and Storage Chart of Hazardous Materials

**§ 177.848 Loading and storage chart of hazardous materials.**

**(a)** Hazardous materials must not be loaded, transported or stored together, except as provided in the Loading and Storage Chart of Hazardous Materials shown in this section.

The following table shows the hazardous materials which must not be loaded or stored together.

The letter X at an intersection of horizontal and vertical columns shows that these articles must not be loaded or stored together, for example: Detonating fuzes class A, with or without radioactive components g horizontal column must not be loaded or stored with high explosives or propellant explosives, class A b vertical column.

| | | Low explosives or black powder | High explosives or propellant explosives, class A | Initiating or priming explosives, wet: Diazodinitrophenol, fulminate of mercury, guanyl nitrosamino guanylidene hydrazine, lead azide, lead styphnate, nitro mannite, nitrosoguanidine, pentaerythrite tetranitrate, tetrazene, lead mononitroresorcinate | Blasting caps, with or without safety fuse (including electric blasting caps), detonating primers | Ammunition for cannon with explosive projectiles, gas projectiles, smoke projectiles, incendiary projectiles, illuminating projectiles or shell; ammunition for small arms with incendiary projectiles; ammunition for small arms with explosive projectiles; rocket ammunition with explosive projectiles, gas projectiles, smoke projectiles, incendiary projectiles, illuminating projectiles; boosters (explosive); bursters (explosive); and supplementary charges (explosive) without detonators • • | Explosive projectiles; bombs; torpedoes; mines; rifle or hand grenades (explosive); jet thrust units (jato), class A; igniters, jet thrust, class A; rocket motors, class A; igniters, rocket motor, class A • | Detonating fuzes, class A, with or without radioactive components | Ammunition for cannon with empty, inert-loaded or solid projectiles, or without projectiles; rocket ammunition with empty, inert-loaded or solid projectiles | Propellant explosives, class B; jet thrust units (jato), class B; igniters, jet thrust, class B; rocket motors, class B; rocket engines (liquid), class B; igniters, rocket motor, class B; starter cartridges, jet engine, class B | Fireworks special or railway torpedoes | Small arms ammunition, or cartridges, practice ammunition. | Primers for cannon or small arms, empty cartridge bags—black powder igniters, empty cartridge cases, primed, empty grenades, primed, combination primers or percussion caps, toy caps, explosive cable cutters, explosive rivets | Percussion fuzes, tracer fuzes or tracers | Time, combination or detonating fuzes, class C | Cordeau detonant fuse, safety squibs, fuse lighters, fuse igniters, delay electric igniters, electric squibs, instantaneous fuse or igniter cord | Fireworks, common, | Flammable liquids or flammable gases; Flammable liquid or flammable gas label. | Flammable solids or oxidizing materials, Flammable solid, oxidizer, or organic peroxide label. | Corrosive liquids; Corrosive label. | Nonflammable gases; Nonflammable gas label. | Poisonous gases or liquids, in tank car tanks, cylinders, projectiles or bombs, poison gas label | Radioactive materials |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | a | b | c | d | e | f | g | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| CLASS A EXPLOSIVES | | | | | | | | | | | | | | | | | | | | | | |
| Low explosives or black powder | (¹) | | | X | | | | | | | X | | | | | | X | X | X | X | X | X | [1]X |
| High explosives or propellant explosives, class A | (¹) | | | X | •X | | | X | | | X | | | | | | X | X | X | X | X | X | [1]X |
| Initiating or priming explosives, wet: Diazodinitrophenol, fulminate of mercury, guanyl nitrosamino guanylidene hydrazine, lead azide, lead | | | | | | | | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| styphnate, nitro mannite, nitrosoguanidine pentaerythrite tetranitrate, tetrazene, lead mononitroresorcinate | (a) | X | X | ---- | X | X | X | X | X | X | X | X | X | X | X | X | X | X | X | X | X | X | 'X |
| Blasting caps, with or without safety fuse (including electric blasting caps), detonating primers | (d) | ---- | •X | X | ---- | X | X | ---- | ---- | ---- | •X | ---- | ---- | ---- | ---- | ---- | •X | •X | ••X | •X | •X | X | 'X |
| Ammunition for cannon with explosive projectiles, gas projectiles, smoke projectiles, incendiary projectiles, illuminating projectiles or shell ammunition for small arms with explosive bullets, or ammunition for small arms with explosive projectiles, or rocket ammunition with explosive projectiles, gas projectiles, smoke projectiles, incendiary projectiles, illuminating projectiles; and boosters (explosive), bursters (explosive), or supplementary charges (explosive) without detonators [a] [e] | (e) | ---- | ---- | X | X | ---- | ---- | X | ---- | ---- | X | ---- | ---- | ---- | ---- | ---- | X | X | X | X | X | X | 'X |
| Explosive projectiles, bombs, torpedoes, or mines, rifle or hand grenades (explosive), jet thrust units (jato), explosive, class A, or igniters, jet thrust (jato), explosive, class A [a] | (f) | ---- | ---- | X | X | ---- | ---- | X | ---- | ---- | X | ---- | ---- | ---- | ---- | ---- | X | X | X | X | X | X | 'X |
| Detonating fuses, class A, with or without radioactive components | (g) | ---- | X | X | ---- | X | X | ---- | ---- | ---- | X | ---- | ---- | ---- | ---- | ---- | X | X | X | X | X | X | 'X |
| CLASS B EXPLOSIVES | | | | | | | | | | | | | | | | | | | | | | | |
| Ammunition for cannon with empty, inert-loaded or solid projectiles, or without projectiles, or rocket ammunition with empty projectiles, inert-loaded or solid projectiles or without projectiles | 1 | ---- | ---- | X | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | •X | ---- | X | ---- |
| Propellant explosives, class B, jet thrust units (jato), class B, igniters, jet thrust (jato), class B, or starter cartridges, jet engine, class B | 2 | ---- | ---- | X | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | •X | ---- | X | ---- |
| Fireworks, special or railway torpedoes | 3 | X | X | X | •X | X | X | X | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | X | ---- |

See footnotes at end of table.

## Subpart C—Loading and Storage Chart of Hazardous Materials—Continued

**§ 177.848 Loading and storage chart of hazardous materials—Continued**

The following table shows the hazardous materials which must not be loaded or stored together.

The letter X at an intersection of horizontal and vertical columns shows that these articles must not be loaded or stored together, for example: Detonating fuzes class A, with or without radioactive components g horizontal column must not be loaded or stored with high explosives or propellant explosives, class A b vertical column.

| | | Low explosives or black powder | High explosives or propellant explosives, class A | Initiating or priming explosives, wet: Diazodinitrophenol, fulminate of mercury, guanyl nitrosamino guanylidene hydrazine, lead azide, lead styphnate, nitro mannite, nitrosoguanidine, pentaerythrite tetranitrate, tetrazene, lead mononitroresorcinate | Blasting caps, with or without safety fuse (including electric blasting caps), detonating primers | Ammunition for cannon with explosive projectiles, gas projectiles, smoke projectiles, incendiary projectiles, illuminating projectiles or shell, ammunition for small arms with explosive bullets, or ammunition for small arms with explosive projectiles, or rocket ammunition with explosive projectiles, gas projectiles, smoke projectiles, incendiary projectiles, illuminating projectiles; and boosters (explosive), bursters (explosive), or supplementary charges (explosive) without detonators [1] | Explosive projectiles, bombs, torpedoes, or mines, rifle or hand grenades (explosive), jet thrust units (jato), explosive, class A or igniters, jet thrust (jato), explosive, class A [1] | Detonating fuzes, class A, with or without radioactive components | Ammunition for cannon with empty, inert-loaded or solid projectiles, or without projectiles, or rocket ammunition with empty projectiles, inert-loaded or solid projectiles or without projectiles | Propellant explosives, class B, jet thrust units (jato), class B, igniters, jet thrust (jato), class B, or starter cartridges, jet engines, class B | Fireworks, special or railway torpedoes | Small arms ammunition, or cartridges, practice ammunition | Primers for cannon or small arms, empty cartridge bags—black powder igniters, empty cartridge cases, primed, empty grenades, primed, combination primers or percussion caps, toy caps, explosive cable cutters, explosive rivets | Percussion fuzes, tracer fuzes or tracers | Time, combination or detonating fuzes, class C | Cordeau detonant fuse, safety squibs, fuse lighters, fuse igniters, delay electric igniters, electric squibs, instantaneous fuse or igniter cord | Fireworks, common | Flammable liquids or compressed flammable gases, red label | Flammable solids or oxidizing materials, yellow label | Acids or corrosive liquids, white label | Compressed nonflammable gases, green label | Poisonous gases or liquids in tank car tanks, cylinders, projectiles or bombs, poison gas label | Radioactive materials |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | a | b | c | d | e | f | g | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| CLASS C EXPLOSIVES | | | | | | | | | | | | | | | | | | | | | | | |
| Small arms ammunition, or cartridges, practice ammunition | 4 | | | X | | | | | | | | | | | | | | | | | | | |
| Primers for cannon or small arms, empty cartridge bags—black powder igniters, empty cartridge cases, primed, empty grenades, primed, combination primers or percussion caps, toy caps, explosive cable cutters, explosive rivets | 5 | | | X | | | | | | | | | | | | | | | | | | | |
| Percussion fuzes, tracer fuzes or tracers | 6 | | | X | | | | | | | | | | | | | | | | | | | |

| | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Fuse, combination or detonating fuzes, class C | 7 | | | X | | | | | | | | | | | | | | | | | | | |
| Cordeau detonant fuse, safety squibs, fuse lighters, fuse igniters, delay electric igniters, electric squibs, instantaneous fuse or igniter cord | 8 | | | X | | | | | | | | | | | | | | | | | | | |
| Fireworks, common | 9 | X | X | X | [a]X | X | X | X | | | | | | | | | | | | | | X | |
| OTHER DANGEROUS ARTICLES | | | | | | | | | | | | | | | | | | | | | | | |
| Flammable liquids or flammable gases; Flammable liquid or flammable gas label | 10 | X | X | X | [a]X | X | X | X | | | | | | | | | | | | | | X | |
| Flammable solids or oxidizing materials; Flammable solid, oxidizer, or organic peroxide label | 11 | X | [e]X | X | [a][e]X | X | X | X | | | | | | | | | | | | [b]X | | X | |
| Corrosive liquids; Corrosive label | 12 | X | X | X | [a]X | X | X | X | [b]X | [b]X | | | | | | | | | [b]X | | | X | |
| Nonflammable gases; Nonflammable gas label | 13 | X | X | X | [a]X | X | X | X | | | | | | | | | | | | | | | |
| Poisonous gases or liquids in tank car tanks, cylinders, projectiles or bombs, poison gas label | 14 | X | X | X | X | X | X | X | X | X | X | | | | | | X | X | X | X | | | |
| Radioactive materials | 15 | [f]X | [f]X | [f]X | [f]X | [f]X | [f]X | [f]X | | | | | | | | | | | | | | | |

[a] Blasting caps or electric blasting caps in quantities not exceeding 1,000 caps may also be loaded and transported with articles named in vertical and horizontal columns 3, 9, 10, 11, 12, and 13. Loading and transportation of blasting caps or electric blasting caps except as prescribed in § 177.835, in any quantity, with articles named in vertical or horizontal columns b, c, e, or f is prohibited.

[b] Corrosive liquids must not be loaded above or adjacent to flammable solids, oxidizing materials, ammunition for cannon with or without projectiles, or propellant explosives, except that shippers loading truckload shipments of corrosive liquids and flammable solids or oxidizing materials packages and who have obtained prior approval from the Department may load such materials together when it is known that the mixture of contents would not cause a dangerous evolution of heat or gas.

[c] Explosives, class A and explosives, class B must not be loaded or stored with chemical ammunition containing incendiary charges or white phosphorus either with or without bursting charges.

[d] Bursters (explosive), boosters (explosive), or supplementary charges (explosive) without detonators when shipped by, to or for the Departments of the Army, Navy, and Air Force of the United States Government may be loaded with any of the articles named except those in columns c, d, 3, 9, 10, 11, 12, 13, 14, and 15.

[e] Does not include nitro carbo nitrate or ammonium nitrate, fertilizer grade, which may be loaded, transported or stored with high explosives or with blasting caps or electric blasting caps, and detonating primers.

[f] Normal uranium, depleted uranium, and thorium metal in solid form may also be loaded and transported with articles named in vertical and horizontal columns a, b, c, d, e, f, and g.

NOTE 1: Charged electric storage batteries must not be loaded in the same vehicle with explosives, class A.

NOTE 2: Cyanides or cyanide mixtures must not be loaded or stored with acids or corrosive liquids.

NOTE 3: Gas identification sets may be loaded and transported with all articles named except those in column e.

NOTE 4: Nitric acid, when loaded in the same motor vehicle with other acids or other corrosive liquids in carboys, must be separated from the other carboys. A 2 by 6 inch plank, set on edge, should be nailed across the motor vehicle floor at least 12 inches from the nitric acid carboys, and the space between the plank and the carboys of nitric acid should be filled with sand, sifted ashes, or other incombustible absorbent material.

NOTE 5: Smokeless powder for small arms in quantities not exceeding 100 pounds net weight in one motor vehicle shall be classed as a flammable solid for purposes of transportation when approved for such classification by the Bureau of Explosives.

**Statutory Authority:** *MS s 116.07 subd 4*

**7045.1240 APPENDIX K.**

TYPE 3 - INLAND SHALLOW FRESH MARSHES. The soil is usually waterlogged during the growing season: often it is covered with as much as six inches or more of water. Vegetation includes, grasses, bulrushes, spikerushes, and various other marsh plants such as cattails, arrowheads, pickerelweed, and smartweeds. Common representatives in the north are reed, whitetop, rice cutgrass, carex, and giant burreed. In the southeast, maidencane, sawgrass, arrowhead, and pickerelweed are characteristic. These marshes may nearly fill shallow lake basins or sloughs, or they may border deep marshes on the landward side. They are also common as seep areas on irrigated lands.

Marshes of this type are used extensively as nesting and feeding habitat in the pothole country of the north central states and elsewhere. In combination with deep fresh marshes (type 4), they constitute the principal production areas for waterfowl. Florida and Georgia are the only states where the majority of the shallow fresh marshes are considered to be of lesser importance to waterfowl. Florida alone contains more than two million acres of this type.

| Flyway area | Acres |
|---|---|
| 1. Pacific north | 33,700 |
| 2. Pacific south | 64,100 |
| 3. Central north | 817,600 |
| 4. Central south | 84,600 |
| 5. Mississippi north | 758,500 |
| 6. Mississippi south | 15,300 |
| 7. Atlantic north | 35,900 |
| 8. Atlantic south | 2,159,900 |

TYPE 4 - INLAND DEEP FRESH MARSHES. The soil is covered with six inches to three feet or more of water during the growing season. Vegetation includes cattails, reeds, bulrushes, spikerushes, and wildrice. In open areas, pondweeds, naiads, coontail, watermilfoills, waterweeds, duckweeds, waterlilies, or spatterdocks may occur. Water hyacinth and waterprimroses form surface mats in some localities in the southeast. These deep marshes may almost completely fill shallow lake basins, potholes, limestone sinks, and sloughs, or they may border open water in such depressions.

Deep fresh marshes constitute the best breeding habitat in the country, and they are also important feeding places. In the western states they are heavily used by migrating birds, especially diving ducks. Florida and Texas are the only states in which the vast majority of these marshes are not rated as being of primary importance to waterfowl.

| Flyway area | Acres |
|---|---|
| 1. Pacific north | 92,500 |
| 2. Pacific south | 62,500 |
| 3. Central north | 686,500 |
| 4. Central south | 46,800 |
| 5. Mississippi north | 427,700 |
| 6. Mississippi south | 21,500 |
| 7. Atlantic north | 25,700 |
| 8. Atlantic south | 984,100 |

TYPE 5 - INLAND OPEN FRESH WATER. Shallow ponds and reservoirs are included in this type. Water is usually less than ten feet deep and is fringed by a border of emergent vegetation. Vegetation (mainly at water depths of less than six feet) includes pondweeds, naiads, wildcelery, coontail, watermilfoils, muskgrasses, waterlilies, spatterdocks, and (in the south) water hyacinth.

In the pothole country of the north central states, type 5 areas are used extensively as brood areas when, in midsummer and late summer, the less permanent marshes begin to dry out. The borders of such areas are used for nesting throughout the northern states. Where vegetation is plentiful, they are used in all sections of the country as feeding and resting areas by ducks, geese, and coots, especially during the migration period.

| Flyway area | Acres |
|---|---|
| 1. Pacific north | 40,500 |
| 2. Pacific south | 51,900 |
| 3. Central north | 676,800 |
| 4. Central south | 87,100 |
| 5. Mississippi north | 1,000,200 |
| 6. Mississippi south | 186,500 |
| 7. Atlantic north | 12,000 |
| 8. Atlantic south | 541,000 |

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.1250 APPENDIX L.

### Appendix I—Representative Sampling Methods

The methods and equipment used for sampling waste materials will vary with the form and consistency of the waste materials to be sampled. Samples collected using the sampling protocols listed below, for sampling waste with properties similar to the indicated materials, will be considered by the Agency to be representative of the waste.

Extremely viscous liquid—ASTM Standard D140-70 Crushed or powdered material—ASTM Standard D346-75 Soil or rock-like material—ASTM Standard D420-69 Soil-like material—ASTM Standard D1452-65

Fly Ash-like material—ASTM Standard D2234-76 (ASTM Standards are available from ASTM, 1916 Race St., Philadelphia, PA 19103)

Containerized liquid wastes—"COLIWASA" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods."[1] U.S. Environmental Protection Agency, Office of Solid Waste, Washington, D.C. 20460. (Copies may be obtained from Solid Waste Information, U.S. Environmental Protection Agency, 26 W. St. Clair St., Cincinnati, Ohio 45268)

Liquid waste in pits, ponds, lagoons, and similar reservoirs.—"Pond Sampler" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods."[1]

This manual also contains additional information on application of these protocols.

### Appendix II—EP Toxicity Test Procedure

*A. Extraction Procedure (EP)*

1. A representative sample of the waste to be tested (minimum size 100 grams) should be obtained using the methods specified in Appendix I or any other methods capable of yielding a representative sample within the meaning of Part 260. (For detailed guidance on conducting the various aspects of the EP see "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods." SW-846, U.S. Environmental Protection Agency Office of Solid Waste, Washington, D.C. 20460.[2])

2. The sample should be separated into its component liquid and solid phases using the method described in "Separation Procedure" below. If the solid residue[3] obtained using this method totals less than 0.5% of the original weight of the waste, the residue can be discarded and the operator should treat the liquid phase as the extract and proceed immediately to Step 8.

3. The solid material obtained from the Separation Procedure should be evaluated for its particle size. If the solid material has a surface area per gram of material equal to, or greater than, 3.1 cm$^2$ or passes through a 9.5 mm (0.375 inch) standard sieve, the operator should proceed to Step 4. If the surface area is smaller or the particle size larger than specified above, the solid material *should be prepared for extraction by crushing, cutting or grinding the material so that it passes through a 9.5 mm (0.375 inch)* sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described below.

4. The solid material obtained in Step 3 should be weighed and placed in an extractor with 16 times its weight of deionized water. Do not allow the material to dry prior to weighing. For purposes of this test, an acceptable extractor is one which will

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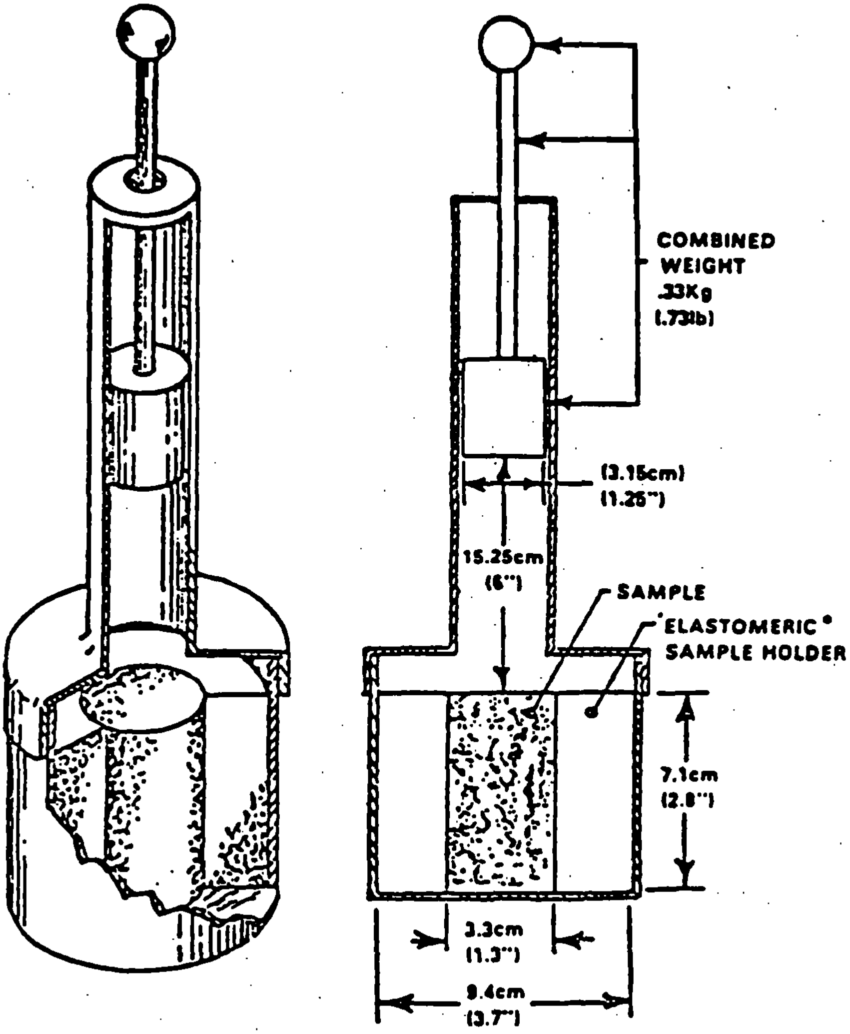
[1] These methods are also described in "Samplers and Sampling Procedures for Hazardous Waste Streams." EPA 600/2-80-018, January 1980.

[2] Copies may be obtained from Solid Waste Information, U.S. Environmental Protection Agcy, 26 W. St. Clair Street, Cincinnati, Ohio 45268.

[3] The percent solids is determined by drying the filter pad at 80°C until it reaches constant weight and then calculating the percent solids using the following equation:

$$\frac{\text{(weight of pad + solid)} - \text{(tare weight of pad)}}{\text{initial weight of sample}} \times 100 = \%\ \text{solids}$$



*ELASTOMERIC SAMPLE HOLDER FABRICATED OF MATERIAL FIRM ENOUGH TO SUPPORT THE SAMPLE

Figure 1

COMPACTION TESTER

[illegible]

impart sufficient agitation to the mixture to not only prevent stratification of the sample and extraction fluid but also insure that all sample surfaces are continuously brought into contact with well mixed extraction fluid.

5. After the solid material and deionized water are placed in the extractor, the operator should begin agitation and measure the pH of the solution in the extractor. If the pH is greater than 5.0, the pH of the solution should be decreased to 5.0 ± 0.2 by adding 0.5 N acetic acid. If the pH is equal to or less than 5.0, no acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction and if the pH rises above 5.2, 0.5N acetic acid should be added to bring the pH down to 5.0 ± 0.2. However, in no event shall the aggregate amount of acid added to the solution exceed 4 ml of acid per gram of solid. The mixture should be agitated for 24 hours and maintained at 20°-40° C (68°-104° F) during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45-A pH Controller manufactured by Chemtrix, Inc., Hillsboro, Oregon 97123 or its equivalent, in conjunction with a metering pump and reservoir of 0.5N acetic acid. If such a system is not available, the following manual procedure shall be employed:

(a) A pH meter should be calibrated in accordance with the manufacturer's specifications.

(b) the pH of the solution should be checked and, if necessary, 0.5N acetic acid should be manually added to the extractor until the pH reaches 5.0 ± 0.2. The pH of the solution should be adjusted at 15, 30 and 60 minute intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5N pH units.

(c) The adjustment procedure should be continued for at least 6 hours.

(d) If at the end of the 24-hour extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH should be adjusted to 5.0 ± 0.2 and the extraction continued for an additional four hours, during which the pH should be adjusted at one hour intervals.

6. At the end of the 24 hour extraction period, deionized water should be added to the extractor in an amount determined by the following equation:

$V = (20)(W) - 16(W) - A$
$V$ = ml deionized water to be added
$W$ = weight in grams of solid charged to extractor
$A$ = ml of 0.5N acetic acid added during extraction

7. The material in the extractor should be separated into its component liquid and solid phases as described under "Separation Procedure."

8. The liquids resulting from Steps 2 and 7 should be combined. This combined liquid (or the waste itself if it has less than ½ percent solids, as noted in Step 2) is the extract and should be analyzed for the presence of any of the contaminants specified in Table I of § 261.24 using the Analytical Procedures designated below.

*Separation Procedure*

Equipment: A filter holder, designed for filtration media having a nominal pore size of 0.45 micrometers and capable of applying a 5.3 kg/cm² (75 psi) hydrostatic pressure to the solution being filtered shall be used. For mixtures containing nonabsorptive solids, where separation can be affected without imposing a 5.3 kg/cm² pressure differential, vacuum filters employing a 0.45 micrometers filter media can be used. (For further guidance on filtration equipment or procedures see "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.")

Procedure:[3]

(i) Following manufacturer's directions, the filter unit should be assembled with a filter bed consisting of a 0.45 micrometer

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[3] This procedure is intended to result in separation of the "free" liquid portion of the waste from any solid matter having a particle size >0.45 µm. If the sample will not filter, various other separation techniques can be used to aid in the filtration. As described above, pressure filtration is employed to speed up the filtration process. This does not alter the nature of the separation. If liquid does not separate during filtration, the waste can be centrifuged. If separation occurs during centrifugation the liquid portion (centrifugate) is filtered through the 0.45 µm filter prior to becoming mixed with the liquid portion of the waste obtained from the initial filtration. Any material that will not pass through the filter after centrifugation is considered a solid and is extracted.

filter membrane. For difficult or slow to filter mixtures a prefilter bed consisting of the following prefilters in increasing pore size (0.65 micrometer membrane, fine glass fiber prefilter, and coarse glass fiber prefilter) can be used.

(ii) The waste should be poured into the filtration unit.

(iii) The reservoir should be slowly pressurized until liquid begins to flow from the filtrate outlet at which point the pressure in the filter should be immediately lowered to 10-15 psig. Filtration should be continued until liquid flow ceases.

(iv) The pressure should be increased stepwise in 10 psi increments to 75 psig and filtration continued until flow ceases or the pressurizing gas begins to exit from the filter outlet.

(v) The filter unit should be depressurized, the solid material removed and weighed and then transferred to the extraction apparatus, or, in the case of final filtration prior to analysis, discarded. Do not allow the material retained on the filter pad to dry prior to weighing.

*(vi) The liquid phase should be stored at 4°C for subsequent use in STEP —.*

B. Structural Integrity Procedure

Equipment: A Structural Integrity Tester having a 3.18 cm (1.25 in.) diameter hammer weighing 0.33 kg (0.73 lbs.) and having a free fall of 15.24 cm (6 in.) shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA. 22314, as Part No. 125, or it may be fabricated to meet the specifications shown in Figure 1.

*Procedure:*

1. The sample holder should be filled with the material to be tested. If the sample of waste is a large monolithic block, a portion should be cut from the block having the dimensions of a 3.3 cm (1.3 in.) diameter × 7.1 cm (2.8 in.) cylinder. For a fixated waste, samples may be cast in the form of a 3.3 cm (1.3 in.) diameter × 7.1 cm (2.8 in.) cylinder for purposes of conducting this test. In such cases, the waste may be allowed to cure for 30 days prior to further testing.

2. The sample holder should be placed into the Structural Integrity Tester, then the hammer should be raised to its maximum height and dropped. This should be repeecate fifteen times.

3. The material should be removed from the sample holder, weighed, and transferred to the extraction apparatus for extraction.

*Analytical Procedures for Analyzing Extract Contaminants*

The test methods for analyzing the extract are as follows:

(1) For arsenic, barium, cadmium, chromium, lead, mercury, selenium or silver: "Methods for Analysis of Water and Wastes," Environmental Monitoring and Support Laboratory, Office of Environmental Protection Agency, Cincinnati, Ohio 45268 (EPA-600/4-79-020, March 1979).

(2) For Endrin; Lindane; Methoxychlor; Toxaphene; 2,4-D; 2,4,5-TP Silver: in "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," September 1978, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 42568,

as standardized in "Test Methods for the Evaluation of Solid Waste, Physical Chemical Methods."

For all analyses, the method of standard addition shall be used for the quantification of species concentration. This method is described in "Test Methods for the Evaluation of Solid Waste." (It is also described in "Methods for Analysis of Water and Wastes.")

Billing Code 6580-01-M

[45 F.R. 33127, May 19, 1980]

**Statutory Authority:** *MS s 116.07 subd 4*

## 7045.1260 APPENDIX M.

Title 49 C.F.R.

**§ 172.202 Description of hazardous material on shipping papers.**

(a) Each description of a hazardous material on the shipping paper must include—

(1) The proper shipping name prescribed for the material as required by § 172.101.

(2) The class prescribed for the material as required by § 172.101. When the words of the proper shipping name are identical (excluding the entry "n.o.s.") with the words of the class, the inclusion of the class is not required.

(3) [Reserved]

(4) Except for empty packagings, the total quantity (by weight, volume, or as otherwise appropriate) of the hazardous material covered by the description.

(b) The basic description specified in paragraphs (a)(1) and (a)(2) of this section must be shown in sequence except that the technical name of the material may be entered between the proper shipping name and the class. For example: "Gasoline, Flammable liquid"; or "Flammable solid, n.o.s."; or "Corrosive liquid, n.o.s. (caprylyl chloride), corrosive material."

(c) The total quantity of the material covered by one description must appear before or after, or both before and after, the description required and authorized by this subpart.

(1) Abbreviations may be used to specify the type of packaging and weight or volume. For example: 40 cyl. Nitrogen, Non-flammable Gas-800 pounds; 1 box Cement, liquid n.o.s., Flammable liquid, 25 lbs.

(2) The type of packaging may be entered in any appropriate manner.

[Amdt. 172-29A, 41 FR 40677, Sept. 20, 1976]

**§ 172.203 Additional description requirements.**

(a) *Exemptions.* Each shipping paper issued in connection with a shipment made under an exemption must bear the notation "DOT-E" *followed by the exemption number assigned and so located that the notation is clearly associated with the description* to which the exemption applies.

(b) *Limited quantities.* The description for a material defined as "limited quantities" in this subchapter must include the words "Limited Quantities" or "Ltd. Qty." following the basic description.

(c) *Blasting caps.* The description for a shipment of blasting caps must have an entry stating the number of caps in the shipment, either before or after the basic description.

(d) *Radioactive material.* (1) The description for a shipment of radioactive material must include the following additional entries as appropriate:

(i) The name of each radionuclide in the radioactive material that is listed in § 173.390 of this subchapter. Abbreviations, e.g., "$^{99}$Mo" are authorized.

(ii) *A description of the physical and chemical form of the material, if the material is not in special form.*

(iii) The activity contained in each package of the shipment in terms of curies, millicuries, or microcuries. Abbreviations are authorized.

(iv) The category of label applied to each package in the shipment. For example: "RADIOACTIVE WHITE-I."

(v) The transport index assigned to each package in the shipment bearing RADIOACTIVE YELLOW-II or RADIOACTIVE YELLOW-III labels.

(vi) For a shipment of fissile radioactive materials—

(A) The words "Fissile Exempt." if the package is exempt pursuant to § 173.396(a) of this subchapter, or

(B) If not exempt, the fissile class of each package in the shipment, pursuant to § 173.389(a) of this subchapter; and

(C) For a Fissile Class III shipment, the additional notation: "Warning—Fissile Class III Shipment, Do not Load More than * * * Packages per Vehicle." (Asterisks to be replaced by appropriate number.) "In loading and Storage Areas, Keep at Least 20 Feet (6 Meters) from Other Packages Bearing Radioactive Labels."

(D) If a Fissile Class III shipment is to be transported by water, the supplementary notation must also include the following statement: *"For shipment by water, only one Fissile Class III shipment is permitted in each hold."*

(vii) For a package approved by the U.S. Energy Research and Development Administration (ERDA) or U.S. Nuclear Regulatory Commission (USNRC), a notation of the package identification marking as prescribed in the applicable ERDA or USNRC approval. (See § 173.393a of the subchapter.)

(viii) For an export shipment or a shipment in a foreign made package, a notation of the package identification marking as prescribed in the applicable International Atomic Energy Agency (IAEA) Certificate of Competent Authority which has been issued for the package. (See § 173.393b(a)(3) of the subchapter.)

(ix) For a shipment of radioactive materials being offered and accepted for transportation and transported within the United States under the provisions of § 171.12(e) of this subchapter, the shipping paper shall be annotated with the following entry:

This shipment contains packages of Type A/low specific activity radioactive materials limited in accordance with the 1973 IAEA Regulations, pursuant to the provisions of 49 CFR 171.12(e). (Non-applicable entry to be deleted.)

(e) *Empty packagings*. For other than a tank car, the description on the shipping paper for an empty packaging containing the residue of a hazardous material may contain the word(s) "EMPTY": or "EMPTY: Last contained * * *" followed by the name of the hazardous material last contained in the packaging. This entry may be before or after the basic description. For empty tank cars, see § 174.25(c) of this subchapter.

(f) *Transportation by air*. When a package containing a hazardous material is offered for transportation by air and this subchapter prohibits its transportation aboard passenger-carrying aircraft, the words "Cargo-only aircraft" must be entered after the basic description.

(g) *Transportation by rail*. (1) The shipping paper for a rail car containing a hazardous material must contain the notation "Placarded" followed by the name of the placard required for the rail car.

(2) The shipping paper for each specification DOT 112A or 114A tank car (without head shields) containing a flammable compressed gas must contain the notation, "DOT 112A" or "DOT 114A," as appropriate, and either "Must be handled in accordance with FRA E.O. No. 5" or "Shove to rest per E.O. No. 5."

(h) *Transportation by highway*. Following the basic description for a hazardous material in a specification MC 330 or MC 331 cargo tank made of quenched and tempered steel, there must be entered for—

(1) *Anhydrous ammonia*. (i) The words "0.2 per cent water" to indicate the suitability for shipping anhydrous ammonia in the cargo tank as authorized by § 177.817 of this subchapter, or

(ii) The words "NOT FOR Q AND T TANKS" when the anhydrous ammonia does not contain 0.2 per cent or more water by weight.

(2) *Liquefied petroleum gas*. The word "Non-corrosive" or "Non-cor" to indicate the suitability for shipment of the "Non-corrosive" liquefied petroleum gas offered for transportation by cargo tank as authorized by § 173.315(a)(1) Note 15 of this subchapter.

(i) *Transportation by water*. (1) Each shipment by water must have the following additional shipping paper entries:

(i) Identification of the type of packages such as barrels, drums, cylinders, and boxes,

(ii) The number of each type of package including those in a freight container or on a pallet, and

(iii) The gross weight of each type of package or the individual gross weight of each package.

(2) The shipping paper for a hazardous material offered for transportation by water to any country outside the United States must have in parenthesis the technical name of the material following the proper shipping name when the material is described by a "n.o.s." entry in § 172.101. For Example: Corrosive liquid, n.o.s. (caprylyl chloride), Corrosive material. However, for a mixture, only the technical name of any hazardous material giving the mixture its hazardous properties must be identified.

(3) The entry "Skin corrosive only" must be included to also authorize "under deck" stowage for corrosive liquid, n.o.s. and corrosive solid, n.o.s. that meet only the corrosion to skin criteria of § 173.240(a)(1).

(49 U.S.C. 1803, 1804, 1808)

[Amdt. 172-29A, 41 FR 40677, Sept. 20, 1976, as amended by Amdt. 172-29B, 41 FR 57067, Dec. 30, 1976; Amdt. 172-41, 43 FR 10918, Mar. 16, 1978; Amdt. 172-45, 43 FR 39791, Sept. 7, 1978]

**Statutory Authority:** *MS s 116.07 subd 4*