



## Standard Practice for Sampling and Handling Phenol, Cresols, and Cresylic Acid<sup>1</sup>

This standard is issued under the fixed designation D 3852; 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. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice is provided to ensure that phenol and cresylic acid are properly sampled to provide representative specimens for quality assurance analyses and that they are handled in a safe manner. In general, this practice also applies to cresols, xylenols, and some other alkylated phenolic materials; however, specific information regarding these materials should be sought and used if available.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Sections 5-8 and Note 1 and Note 2.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

E 300 Practice for Handling Industrial Chemicals<sup>2</sup>

#### 2.2 Other Documents:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200<sup>3</sup>

U.S. DOT Regulations, 49 CFR Transportation, Subchapter C, Parts 171 – 180<sup>3</sup>

NFPA No. 704-1996<sup>4</sup>

### 3. Significance and Use

3.1 This practice is issued to provide information useful in establishing sampling and handling procedures. It is expected that this information will only be utilized in conjunction with an existing health and safety program. The information provided cannot be used as a substitute for expert safety and medical advice, but rather as a supplement to such advice.

### 4. Description of Products (See Tables 1 and 2)

4.1 Phenol is a colorless to light pink crystalline material

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.08 on Handling and Sampling Aromatic and Cyclic Hydrocarbons.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.05.

<sup>3</sup> Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

<sup>4</sup> Available from National Fire Protection Assoc., 1 Battery March Park, Quincy, MA 02269-9101.

TABLE 1 Cresylic Properties

	Boiling Point, °C	Flash Point, °F	Flammability Limits, %		Specific Gravity
			Lower	Higher	
Phenol	181	175	1.7	8.6	1.06
Cresylic Acid <sup>A</sup>	220-250	200	1.1	...	1.05
Cresols	181-250	175	1.1	...	1.05

<sup>A</sup>A mixture of phenolic materials boiling above the cresal range.

which melts at 40 to 41°C (104 to 106°F). Technical and USP grades melt at lower temperatures.

4.2 Phenol is both extremely hygroscopic and sensitive to discoloration. Therefore, it cannot be overemphasized that proper precautions must be undertaken when unloading or sampling the product. Moisture must be excluded. The use of sampling devices that contain metals that may catalyze discoloration (iron, copper) must also be avoided.

4.3 Cresylic acid is a common chemical name applied to mixtures of alkyl-substituted phenols. Included are mixtures of cresols, xylenols, and higher alkylated phenols. Many cresylic acid mixtures contain measurable amounts of phenol.

4.4 Most cresylic acid mixtures are liquids at ambient temperatures. However, at low temperatures (<0°C) they sometimes become very viscous and difficult to pour. Some mixtures containing high concentrations of high melting isomers may form thick slurries or become solids at low temperatures.

4.5 While phenol or cresylic acid is highly dangerous when handled improperly, particularly at the elevated temperatures sometimes required to unload tank cars or tank trucks, handling and sampling need not be hazardous provided the dangers are recognized. Proper precautionary measures must be provided and scrupulously adhered to.

4.6 Department of Transportation (DOT) Hazardous Materials Regulations regarding the shipment of this chemical are specified in 49 CFR.

### 5. Hazards

5.1 For information on toxicity see Toxic Substances List, Appendix I,<sup>3</sup> 1975.

5.2 Consult current OSHA regulations suppliers' material safety data sheets (MSDSs), and local regulations for all materials utilized in this practice.

5.3 *Health*—Phenol is very corrosive to the skin and produces painful and dangerous burns in a very short time. Since phenol is a skin anesthetic, the first reaction is not pain, but a whitening of the exposed area. It is readily absorbed through

**TABLE 2 Bulk Packaging Regulatory Information**

NOTE 1—Single packagings are not permitted on passenger aircraft in accordance with the Internal Civil Aviation Organization Technical Instructions for the Safe Transport of Dangerous Goods By Air and the International Air Transport Association Dangerous Goods Regulations. Single packagings are permitted on cargo aircraft.

DOT Basic Description	RQ lb	Packaging Authorization 49 CFR, 173*				Label(s)	Placards	Remarks
		Listed Marine Pollutant	Exception	Non-bulk	Bulk			
Xylenols, 6.1, UN 2261, II	1000	yes	none	212	242	Poison	Poison	See Note 1
Cresols, 6.1, UN2076, II, (8)	100	yes	none	202	243	poison	poison	See Note 1.
Phenol, molten, 6.1, UN2312, II	1000	no	none	202	243	Poison	Poison	Forbidden for air transport
Phenol, solid 6.1, UN1671, II	1000	no	none	212	242	Poison	Poison	See Note 1.
Phenol solutions, 6.1, UN2821, II	1000	no	none	202	243	Poison	Poison	See Note 1.
Phenol solutions, 6.1, UN2821, III	1000	no	153	203	241	Poison or Keep Away From Food	Poison or Keep Away From Food	
Cresylic acid, 6.1, UN2022, II	100	yes	none	202	243	Primary: Poison Subsid- iary risk: Corrosive	Poison	See Note 1.

the skin and mucous membranes or lungs, and severe exposures may prove fatal unless prompt first aid and medical treatment are exercised. Since severe injury or death may result from excessive exposure to vapor or mist, adequate ventilation of working areas is imperative. It is therefore recommended that tank cars or tank trucks shall be unloaded in the open, rather than inside a closed building and workers shall wear appropriate protective clothing and personal protective equipment.

5.3.1 Qualitatively, cresylic acid is slightly less acute as a health hazard than phenol. However, contact of cresylic acid with the skin can produce painful and serious burns in a short time. It is readily absorbed through the skin and mucous membranes, through the gastro-intestinal tract, or through the lungs (either as a vapor or in droplet form), potentially resulting in systemic poisoning. Although no safety limits have been defined for cresylic acid vapor, severe injury or death can result from excessive exposure to high concentrations of the vapor or mist, or prolonged exposure to low concentrations. Therefore, adequate ventilation of work areas is imperative.

5.4 *Fire*—Cresylic acid parallels phenol in its fire hazard properties, being somewhat less hazardous due primarily to lower vapor pressure at any given temperature. Phenol is placed in Category 2 of the NFPA 704M fire hazard classification system (Note 1) while *m*- and *p*-cresols are in Category 1. Phenol and cresylic acid are combustible and flammable; toxic vapor will be given off at elevated temperature should this material become involved in a fire. Water (fog or spray), carbon dioxide extinguishers, foam, and dry chemical extinguishers are effective in fighting fires involving phenol and cresylic acid.

NOTE 1—For full description of NFPA categories, see NFPA publication No. 704-1996. Classification runs from 0 (no hazard) to 4 (very hazardous).

5.5 Molten phenol or cresylic acid dissolves carbon dioxide and releases it on solidification. Therefore, special precautions shall be observed if “inert gas” containing carbon dioxide is used to agitate or empty containers of phenol or cresylic acid to avoid pressure build-up (for example, leave vents open).

5.6 For chemical emergency (spill, leak, fire, exposure, or accident) call CHEMTREC, day or night, at 1-800-424-9300. For emergency calls outside the United States, call 703-527-3887. (Collect calls are accepted and all calls are recorded.)

## 6. Protective Equipment

6.1 No personal protection equipment is an adequate substitute for safe working conditions and intelligent conduct on the part of employees who work with phenol or cresylic acid. Employees who work with phenol or cresylic acid should be well trained and should maintain safe working conditions.

6.2 Persons engaged in the handling of phenol or cresylic acid shall use protective equipment as dictated by the extent of their exposure. The worker shall always wear chemical-type safety goggles and chemically impermeable gloves, as a minimum. Depending upon circumstances, additional personal protection may be advisable, including face shield, rubber shoes or boots, rubber aprons or acid-proof suits, and industrial gas masks or fresh air masks. Wearing full protective clothing is recommended when sampling tank cars, tank trucks, barges, ships, drums, process lines and process vessels. Working areas shall have immediately available deluge-type safety showers, easily accessible, plainly marked, and controlled by quick-opening valves. In addition, there shall be at hand a water hose that will deliver clean water at a moderate pressure. A small stream, such as from an eyewash fountain, is recommended for washing eyes (see 7.2).

## 7. First Aid

7.1 The establishment of first aid procedures must be done prior to sampling and handling of phenol and cresylic acid under the guidance of competent safety and medical advice.

7.2 The first aid procedures established should include, but not be limited to, the following considerations:

7.2.1 Speed in removing phenol or cresylic acid from the skin in case of accidental contact is of primary importance.

7.2.2 It is extremely important to immediately place under a physician’s care any person injured by skin contact, inhalation, or ingestion of phenol or cresylic acid.

## 8. Precautions

8.1 Any person sampling or handling these products should have specific first aid instructions and equipment available for use in the event of personal contact or exposure.

8.2 Conduct sampling and handling operations only by carefully instructed, experienced, reliable employees, under adequate supervision.

8.3 Accomplish loading, unloading, and sampling operations only when adequate lighting is provided.

8.4 Take extreme care to avoid spills and leaks. In case of a spill, wash contaminated areas thoroughly with large quantities of water and collect the liquid in the local chemical waste system. All spill-related activities should comply with applicable EPA, OSHA, and local regulations and laws.

8.5 Follow shipper's instructions always, and read and observe all caution markings on containers.

8.6 Although the vapor given off at elevated temperatures from phenol or cresylic acid will ignite, these materials can generally be handled with little direct danger of fire. The flash points of the liquids are higher than the temperatures at which they are normally handled. In spite of this, carefully restrict open flames and smoking in the vicinity of loading, unloading, and storage operations.

8.7 Do not permit any person ever to enter an empty phenol or cresylic acid tank, tank car, or tank truck until it has been thoroughly washed out with warm water, followed by a thorough steaming. Ensure that oxygen content is acceptable and vessel is free of organic vapors. Require the approval and observation by a supervisor in every case. Review Sections 6 and 7 in detail.

8.8 Allow no eating or drinking in close proximity to the phenol or cresylic acid handling or sampling operation.

8.9 Employees shall:

8.9.1 Know the hazards connected with the handling of phenol and cresylic acid;

8.9.2 Be completely acquainted with the purpose, use, and maintenance of personal protective equipment;

8.9.3 Be trained to report promptly to supervision all suspected leaks or equipment failures;

8.9.4 Be trained to recognize and report any symptoms of systemic poisoning or skin contact; be thoroughly trained in the proper procedures for administering first aid and for obtaining professional medical help;

8.9.5 Know and routinely practice the accepted methods of sampling and handling phenol or cresylic acid in order to avoid spilling or splashing, leaks, skin contact, vapor or mist inhalation, or ingestion;

8.9.6 Be completely familiar with the location and operation of safety showers, eye baths, hose lines, and all other first aid equipment; and

8.9.7 Know the importance of personal cleanliness and the necessity for immediate removal of clothing contaminated with phenol or cresylic acid.

## 9. Handling and Sampling of Drums

9.1 Before loading, unloading, or sampling of drums of phenol or cresylic acid, carefully read and proceed in accordance with Sections 4-8.

9.2 Handle drums carefully when being transported. Block drums in place during transportation to prevent movement and during unloading to prevent spilling.

9.3 Place the drum bung up, loosen the plug slowly to relieve internal pressure, and allow all internal pressure to vent prior to unloading or sampling.

9.4 Most cresylic acids are liquids at ambient temperatures. Phenol and some cresylic acids are solids at ambient temperatures. If melting is necessary to remove the contents of a drum, do the necessary heating in a special steam-heated melting chamber, hot-water bath, with steam coils, or with an approved electric drum heater. Never use a flame for melting the drum contents. Properly vent the drum during this operation in order to prevent pressuring.

NOTE 2—**Warning:** Do not overheat drums as the danger of spillage caused by thermal expansion and excessive vapors exists.

9.5 Prior to sampling, mix the contents of the drum thoroughly in order to ensure uniformity of the material. This may be accomplished by mechanical agitation or sparging with inert gas (nitrogen is recommended). Exercise extreme caution to prevent pressurization or splashing if inert gas sparging is used.

9.6 Obtain the sample in accordance with Practice E 300. In summary, accomplish sampling by using a clean, dry glass or polypropylene sample tube (see 12.5) and a clean, dry, glass or other appropriate container of appropriate size. The closure shall be a screw cap fitted with a polyethylene or other inert liner. Label the sample container to indicate, as a minimum, the date and time, source of sample, type of material, quantity, hazards, purpose of sample, and the name of the sampler and in accordance with OSHA regulations.

9.7 Unload by any convenient, safe method, including gravity flow and pumping. *Pressure unloading of drums is not recommended.* Take full precautions to protect personnel and equipment from the effects of a possible drum rupture.

## 10. Unloading of Tank Cars and Tank Trucks

10.1 Before unloading or sampling tank cars or tank trucks of phenol or cresylic acid, carefully read and proceed in accordance with Sections 4-8.

10.2 Always follow DOT Regulations and shipper's instructions, and place, read, and observe all caution markings on the sides of the tank or dome.

10.3 Brakes must be applied, warning signs in place, chocks in place, and ground or bonding wire, or both, attached before any operation begins. Follow normal derailment procedures.

10.4 In the event of a tank or fitting failure or leak, immediately notify your supervisor. Take whatever appropriate action is deemed necessary to prevent injury to personnel or damage to equipment (see 8.3).

10.5 *Top Unloading by Pumping:*

10.5.1 Verify that the contents of the tank car or tank truck are designated for storage in the tank you will be unloading to. Verify that adequate storage volume is available in the storage tank and that the tank is vented before connecting the unloading line.

10.5.2 Carefully open the vent on the tank car or tank truck and leave the vent open during loading and unloading. In case the dome is not equipped with an air inlet vent, vent by

carefully opening the manhole cover. Do not confuse the air inlet nozzle with the eduction or siphon pipe connection.

10.5.3 Verify that no solid has formed in the tank car or tank truck due to unusually low temperatures or a high concentration of a high melting isomer. In cases of solid formation or highly viscous materials, heating of the contents of the tank is recommended. This is accomplished by application of low-pressure steam to the heating coils of tank cars and trucks so equipped or through the use of insert or bayonet heaters. If a crust has formed, use insert or bayonet heaters to melt the crust prior to proceeding with heating the entire contents of the container. The use of mechanical agitation or inert gas (preferably nitrogen) sparging is recommended to facilitate this operation. Follow proper provision for venting during this operation. Hold heating and mixing to a minimum in order to avoid color degradation of the phenol or cresylic acid. Discontinue these operations prior to unloading or sampling.

10.5.4 After completing the recommendations in 10.5.1-10.5.3, connect the discharge side of the unloading pump to the line leading to the storage tank. Connect the suction side of the unloading pump to the eduction or siphon pipe on top of the dome. In the absence of such a permanent pipe, insert a clean stainless steel or nickel pipe of appropriate size through the manway opening to the bottom of the tank car or tank truck and connect the free end to the suction side of the pump.

NOTE 3—Transfer lines and associated valves should be preheated with low-pressure steam to facilitate unloading.

10.5.5 Open the appropriate valves in the transfer lines between the tank car (or tank truck) and the pump, and between the pump and the storage tank and start the pump.

10.5.6 After all the phenol or cresylic acid has been discharged, shut off the pump and close the unloading line valves at the receiving tank and at the tank car (or truck). Either drain the lines or blow them clear with compressed air or nitrogen to the tank or waste recovery drain designated by your supervisor. If compressed air (or nitrogen) is used, close the air supply when the unloading line is clear and vent the unloading line. Disconnect the compressed air line. Close all transfer line valves; disconnect the transfer lines from the tank car (or truck), pump, and storage tank; and thoroughly wash all traces of phenol or cresylic acid from the pipe ends and fittings.

10.5.7 Disconnect the steam and condensate piping to the tank car heating coils. Blow the coils out with compressed air in order to remove condensate and prevent freezing of pipes. Do not replace steam inlet and outlet caps; let them hang free by their safety chains so as to permit drainage of the pipes. Close all tank car dome openings, vents, and bottom outlet plugs.

#### 10.6 Top Unloading by Pressurization:

10.6.1 As a less desirable alternative to the use of a transfer pump for top unloading of material, the pressurization of the tank car or truck with compressed nitrogen to force the cresylic acid out may be used where the head pressure of the receiving vessel can be overcome within the recommended unloading pressure limits. The use of air for pressurization is not recommended because of the potential to form explosive mixtures. If this is done, place special emphasis on the inspection of the tank and its fittings for evidence of leaks or

defects and its maximum pressure rating. Unloading pressure shall never exceed a gage pressure of 25 psi (172 kPa) or the maximum pressure rating, whichever is less. The air or nitrogen line must be equipped with a check valve and a shut-off valve, a pressure-reducing-regulating valve set at 20 psi (138 kPa), a safety relief valve set at 20 to 25 psi (138 to 172 kPa), and a vent valve.

10.6.2 Vent the tank car or truck and inspect for the presence of solids (see 10.5.3); if solids are present, heat and stir the material until the solids are melted.

10.6.3 Connect the discharge line to the eduction pipe.

10.6.4 Make sure the storage tank is vented and has sufficient empty volume.

10.6.5 Close the tank car or truck venting and attach the nitrogen supply line to the inlet nozzle.

10.6.6 Open the appropriate discharge line valves and slowly apply nitrogen pressure to the tank car or truck. Adjust the nitrogen pressure to 20 psi (138 kPa) and maintain it until the tank car and discharge lines are empty.

10.6.7 Close the receiving tank valve, shut off the nitrogen supply line valve, and open the relief valve.

10.6.8 Verify that the lines are depressurized, then disconnect the nitrogen supply line, the discharge line, and thoroughly wash all material from pipe ends and fittings.

10.6.9 Close the tank car or truck dome openings securely.

10.6.10 Disconnect the steam and condensate piping to the tank car or truck heating coils. Blow out the coils with compressed air in order to remove condensate and prevent freeze up during cold weather. Do not replace steam inlet and outlet pipe caps; let them hang by their safety chains so as to permit drainage of the pipes.

#### 10.7 Bottom Unloading by Pumping:

10.7.1 Bottom unloading of molten phenol or cresylic acid increases the possibilities of spillage or spraying the operator. Observe extra caution to prevent this occurrence.

10.7.2 If it is necessary to unload molten phenol or cresylic acid through the bottom outlet, install an auxiliary shut-off valve in the bottom outlet leg.

10.7.3 Verify that the unloading line is properly attached to the designated storage tank, that the storage tank is vented, and that sufficient storage volume is available to hold the contents of the car or truck being unloaded.

10.7.4 Carefully open the vent on the tank car (or truck) to release any pressure that may have built up.

10.7.5 Open the manway and visually inspect the contents for the presence of solids. If solids are noted, proceed as instructed in 10.5.3.

10.7.6 Be sure the bottom interior valve is closed tight by turning the top operated valve rod clockwise on top of the car. Verify this by placing a container under the plug on the bottom outlet leg and loosening the plug. Allow any trapped phenol or cresylic acid to drain into the container. *Do not remove the plug entirely until you are certain that the inside valve is holding.*

10.7.7 If the outlet leg is equipped with an auxiliary valve, open it carefully to allow any trapped phenol or cresylic acid to drain into the container. If there is no bottom auxiliary valve, connect one to the bottom outlet leg. Close the auxiliary valve.

10.7.8 Attach the unloading line to the auxiliary outlet



valve. This line must have a drain valve. Make sure the drain valve is closed.

10.7.9 Open the outside auxiliary shut-off valve.

10.7.10 Open the inside bottom outlet valve and pump or drain molten phenol or cresylic acid from the tank car (or truck).

10.7.11 Close the inside bottom outlet valve; open the drain valve and drain the bottom outlet leg into the container.

10.7.12 Close the outside auxiliary shut-off valve and the receiving-tank valve, drain the unloading lines or blow them clear of phenol or cresylic acid with compressed air or nitrogen to the tank or waste-recovery drain designated by your supervisor. If nitrogen is used, close the nitrogen supply when the unloading line is clear and vent the unloading line. Disconnect the nitrogen line.

10.7.13 Close all transfer-line valves; disconnect the transfer lines from the tank car (or truck), pump, and storage tank; and thoroughly wash all traces of phenol or cresylic acid from the pipe ends and fittings.

10.7.14 Disconnect the steam condensate piping to the tank-car heating coils. Blow the coils out with compressed air or nitrogen in order to remove condensate and prevent freezing of pipes. Do not replace steam inlet and outlet caps; let them hang free by their safety chains so as to permit drainage of the pipes securely. Close all tank-car dome openings, vents, and bottom-outlet plugs.

10.8 After unloading is completed, by whatever means, remove chocks, safety signs, and ground wire.

10.9 *Tank Cars*—Turn the “Poison” placards for empty phenol tank cars to “Residue Poison.” Turn the “Poison” placards for empty cresylic acid tank cars to “Residue Corrosive.” Return empty phenol cans with “Poison” placards; also, return empty cresylic acid cans with “Poison” placards as received.

10.10 *Tank Trucks*—Return tank trucks with “Poison” placards (for phenol) and for cresylic acid in place as received.

## 11. Sampling of Tank Cars and Tank Trucks

11.1 Sample tank cars or trucks in accordance with Practice E 300. Any method of sampling therein described that provides a representative sample is acceptable. Among these are “Average Sample,” “All-Level Sample,” “Continuous Sample,” and “Running Sample.”

11.1.1 Refer to 49 CFR 173 for specific packaging regulations for sample shipment.

11.2 A suitable clean, dry glass or other appropriate container, of appropriate size, must be used.

11.3 Sampling shall be done after heating or mixing, or both, or during unloading (in the case of a continuous sample).

11.4 Place emphasis on the use of clean and dry sampling equipment and sample containers.

11.5 Immediately after the sample container is filled, screw the cap on tightly before making any attempt to rinse off phenol or cresylic acid from the outside. Rinse bottle with water and follow approved procedures for disposal. Label the sample as with drum samples (see 9.6).

## 12. Storage

12.1 Refer to Sections 4-8 regarding the hazardous properties of phenol or cresylic acid before attempting any storage.

12.2 Heat tracing of tank vents and relief devices is recommended to keep vapors from solidifying and blocking those devices. Heat tracing of storage vessels is recommended to keep material in liquid form.

12.3 The choice of construction materials for storing phenol or cresylic acid depends on the color requirements in conjunction with the end use.

12.4 When color of the phenol or cresylic acid is not important, vessels of ordinary carbon steel serve satisfactorily. Phenol and cresylic acid have no appreciable corrosive activity on mild steel at the temperatures normally encountered in transportation and storage.

12.5 Hot phenol and cresylic acid readily attack metals such as copper, aluminum, magnesium, lead, and zinc. Therefore, these metals and their alloys are not recommended for use in phenol cresylic acid storage tanks.

12.6 Molten phenol and cresylic acid dissolve and soften many organic polymers. It is recommended that previous testing be done to determine the stability and corrosion resistance of any polymer prior to its use as a lining material.

## 13. Barges and Tankers

13.1 Barges and tankers are sampled and handled in a manner similar to top unloading of tank cars and tank trucks (10.5). The shipment of these products by barge is regulated by the U.S. Coast Guard. Barges used for transporting these materials are regulated by 46 CFR, Subchapter O.

## 14. Keywords

14.1 alkyl-substituted phenols; cresols; cresylic acid; handling; phenol; sampling; xylenols

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