

Safe Handling of Cresols, Xylenols & Cresylic Acids

I. Introduction

Cresols, xylenols and cresylic acids are hazardous substances and dangerous both to people and the environment if handled improperly. Cresols, xylenols and cresylic acid products produced by Sasol Chemicals (USA) LLC are highly versatile materials and are used as intermediates in the manufacture of a wide variety of industrial products such as resins, flame retardants, antioxidants, and coatings. In these and other applications, cresylic acids can be stored, transferred, processed and disposed of safely when proper procedures and safeguards are used.

"Cresol" refers to any of the three isomers of methylphenol (C_7H_8O) or combinations thereof. "Cresols" commonly refer to a mixture which is predominantly methylphenol but may also contain lesser amounts of other alkylphenols. "Xylenol" is a common name for any of the six isomers of dimethylphenol ($C_8H_{10}O$) or their various combinations. Material which is predominantly dimethylphenol but which also contains ethylphenols and other alkylphenols may be referred to as "Xylenols". "Cresylic acid" is a generic term referring to various combinations of cresols, xylenols, phenol or other alkylphenols (ethylphenols, propylphenols, trimethylphenols, etc.). See Table A for specific information on individual isomers.

Purpose & Scope

The purpose of this document is to provide information gathered through Sasol's long experience in the safe handling of cresylic acids. It focuses on basic and practical information about working safely with these substances. Additional references are provided and it is strongly recommended that these and others be consulted prior to working with cresylic acids. Please do not hesitate to contact your regional Sasol office if we can be of assistance in the safe storage, handling, processing and disposal of our products.

II. Hazards

Health Hazards

The primary dangers posed in handling cresylic acids are those resulting from physical exposure. Cresylic acids are highly corrosive and contact with exposed skin or mucous membranes causes severe burns. These burns progress from an initial whitening of the exposed skin to blackish-brown necroses within 24 hours after exposure. Cresylic acids also exhibit anesthetic properties. Therefore, victims frequently misjudge the extent of their exposure when the initial burning sensation rapidly subsides. This can result in prolonged contact, causing toxic effects in addition to the corrosive damage.

Cresylic acids are readily absorbed through the skin and mucous membranes in liquid or vapor form and act as systemic toxins for which there is no established treatment. Relatively small areas

of exposure (e.g. an arm or a hand) can allow sufficient absorption to cause severe poisoning. Progressive symptoms of such poisoning include headache, dizziness, ringing in the ears, nausea, vomiting, muscular twitching, mental confusion, loss of consciousness and, possibly, death from lethal paralysis of the central nervous system. Chronic exposure can lead to loss of appetite, vomiting, nervous disorders, headaches, dizziness, fainting and dermatitis.

The Occupational Health & Safety Administration (OSHA) has established 5 ppm or 22 mg/m³ permissible exposure limits (PEL's) for cresols on an 8-hour time-weighted average basis. OSHA guidelines also indicate that adequate personal protective equipment (PPE) should be employed to avoid skin contact with cresols. Cresylic acids are not listed as carcinogens by OSHA, the International Agency for Research on Cancer (IARC) or the National Toxicology Program (NTP).

Fire Hazards

Cresylic acids are classified as combustible liquids by the U.S. Department of Transportation (DOT). The primary fire hazard they pose is the accidental ignition of a vapor/air mixture at a concentration within their explosivity range (~1%-8%, by volume). This potential exists whenever cresylic acids are stored, handled or processed at temperatures above their respective flash points (see Table A).

Another hazard posed by a fire is the potential for significant amounts of cresylic acid vapor to be formed and carried with the smoke, creating the possibility of serious physical and environmental exposure down-wind from the fire.

Reactivity Hazards

In general, cresylic acids are stable compounds. However, exposure of cresylic acids to strong oxidizers can result in exothermic reactions and potentially hazardous by-products. Another hazard associated with cresylic acids is the corrosive effects they have on the materials used in their storage, handling and processing.

Environmental Hazards

Cresylic acids show high acute toxicity towards both fish and aquatic invertebrates and must be prevented from entering surface or ground waters. Depending upon the specific composition, the material may be classified as a marine pollutant. Please refer to the current label and safety data sheet.

III. Physical & Chemical Characteristics

Phenol, pure cresol or xylenol isomers and cresylic acids with high concentrations of any single isomer may be solid at ambient temperatures. Broader mixtures of cresylic acids are typically liquid at ambient temperature.

Cresylic acids are most conveniently handled as liquids and offer a fairly broad temperature range within which they can be safely handled in this form. In this state, cresylic acids range in appearance from colorless to dark amber with a viscosity similar to mineral oil and a sharp, antiseptic odor. Their viscosity and density will decrease significantly with increasing temperature. Note that this decrease in density often corresponds to a significant increase in volume. This is important to consider when heating cresylic acids in a fixed-volume system. As the temperature rises, the cresylic acids will also emit pungent vapors capable of forming an ignitable mixture with air at their respective flash points. Certain cresylic acid mixtures and pure isomers are capable of super-cooling – that is, they can remain in liquid form at temperatures well below their freeze point. Factors such as seeding and physical shock can cause rapid crystallization of the super-cooled material.

Cresylic acids are highly soluble in phenol and many other organic solvents, including aliphatic alcohols, ethers, chloroform, solvent naphtha and glycerol. Cresylic acids will readily absorb moisture from the air, although to a lesser degree than phenol. Their solubility in water increases in the presence of other water-soluble organic compounds (e.g. methanol) and decreases with increasing levels of dissolved inorganic salts. Cresylic acids tend to sink in fresh water; but will float in concentrated brine. In liquid form, cresylic acids will also readily dissolve carbon dioxide, which is rapidly released if and when the material crystallizes.

Cresylic acids are chemically similar to phenol. They are weak acids and react with aqueous alkali to form water-soluble salts known as cresylates. However, their low acidity allows even weak acids such as carbon dioxide to liberate them from cresylates. Cresylic acids are sensitive to oxidation, forming various hydroquinones, quinols, quinones, cyclic ketones, furans and toluic ethers when exposed to oxidizing agents. Strong oxidizers may break down the phenolic ring. Exposure to air over time will lead to formation of trace levels of oxidatively-coupled phenolic dimers and/or trimers with accompanying color development. This does not typically affect the resulting product performance unless color is an issue.

Cresylic acids readily attack aluminum and aluminum alloys. At temperatures exceeding 120°C (248°F), this attack can be severe and may proceed explosively when moisture levels in the cresylic acids are below 0.3%. Reaction with copper and brass alloys is similar, but less severe. Cresylic acids also attack unalloyed and chromium steels at elevated temperatures, increasing in corrosivity with decreasing water content. Corrosion rates for austenitic (chromium-nickel) stainless steels exposed to boiling cresylic acids range from 0.1 to 1 mm/annum, depending on their specific type. The corrosion rate for nickel in cresylic acids @ 190°C (374°F) is 0.001 mm/annum. Tantalum is considered to be entirely resistant to cresylic acid corrosion.

IV. Controls for Working with Cresols

Safe storage, handling, processing and disposal of cresylic acids begin long before they ever arrive on-site. Measures necessary to ensure the health and well-being of employees, customers, the community and the environment include the development of effective administrative and engineering controls designed to specifically address the hazards associated with cresylic acids. Personal protective equipment (PPE) is integral to safe handling and should be viewed as the last line of defense against an accidental failure of the administrative and/or engineering controls.

Administrative Controls

Administrative controls are the foundation of any program designed for safely handling cresylic acids. Every company is unique in how they run their business and establish administrative controls. Those specifically developed for working with cresylic acids should address comprehensive process planning, thorough communication of hazards to employees and extensive training of employees on the proper implementation of all safety measures.

Engineering Controls

Engineering controls for safely storing, transferring, processing and disposing of cresylic acids should be developed around maintaining the physical integrity of the processing equipment from receipt to disposal. Selection of materials of construction and mechanical equipment should address both the physical and chemical properties of cresylic acids. Table B lists suggested materials for piping and tanks used for handling cresylic acids, along with suggestions for pumps, gaskets and seals. Special consideration must be given to providing adequate emergency equipment in areas where cresylic acids are handled, including readily accessible, plainly marked deluge-type safety showers and eye-wash stations.

Personal Protective Equipment (PPE)

All personnel who work with or near cresylic acids must use adequate personal protective equipment (PPE). The extent of the potential exposure and consideration of established permissible exposure limits (PEL's) should dictate the level of protection necessary. Personnel working with or near lab-scale quantities should always wear safety glasses with side-shields or chemical mono-goggles, chemical-resistant or impermeable gloves, long-sleeved shirts and trousers as a minimum. Circumstances such as elevated temperature and pressure or vacuum conditions should dictate if more substantial protection is necessary, including face shields, chemically impermeable outerwear, and breathing protection. Personnel transferring larger quantities of cresylic acids, or working in areas where a line-break could result in similar exposure, should always wear full protective equipment. Table C lists suggested protection levels and types of PPE.

V. Handling

This section highlights some of the key points covered in **ASTM D3852-07**, **Standard Practice for Sampling and Handling Phenol**, **Cresols**, **and Cresylic Acid**. It is <u>strongly</u> urged that this document be thoroughly reviewed by anyone who will be working with cresylic acids.

Receipt & Unloading

Sasol delivers cresylic acids by several different transportation modes – drums, tank-trucks, ISO-tank containers, railcars and variable capacity barges and tankers.

A number of considerations are common to all the procedures for unloading cresylic acids, regardless of the particular mode in which they are received. First, any employee directly involved with making the unloading connections should wear Level I (maximum) PPE. Second, all vessels should be vented to a contained system routed through either a caustic scrubber or a flare to prevent release of vapors to the atmosphere. Finally, particular care should be taken when heating is required to prevent excessive vapors and overflow resulting from thermal expansion of the material.

Drums should always be secured in place when unloading to prevent spillage. With the drum bung in the up position, loosen the plug slowly to relieve internal pressure and then unload the drum by either gravity-flow or pumping. Drums are not designed for pressurization. Therefore, pressurized unloading is not recommended as it could result in rupturing of the drum. If the material in the drum has solidified, it should be melted gradually in a hot box using low-pressure (60 psig / 414 kPa) steam or hot water, or by use of an approved electric drum-heater according to manufacturer recommendations. Drums should be vented during melting and unloaded as described above. Any solidified product in a drum should be thoroughly melted before unloading to ensure consistent isomer composition and complete emptying of the drum. Overheating should be avoided as it may lead to spillage caused by thermal expansion, excessive vapors and discoloration of the material.

The preferred method for unloading tank-trucks, ISO-tank containers and railcars is by pumping from the top of the vessel. Special attention should be taken when using this method to provide an adequate air-vent into the vessel. Alternatively, vessels may be unloaded from the top by pressurizing with compressed nitrogen or from the bottom by pumping or nitrogen pressurization. Any solidified material in the vessel should be melted prior to unloading by applying low-pressure (60 psig / 414 kPa) steam or warm water to the vessel heating coils or through the use of insert or bayonet heaters. Again, overheating should be avoided to prevent spillage, reduce vapors and slow discoloration. Transfer lines and vents should also be heated when working with freezable materials to avoid the pressure differences resulting from blockages.

Barges and tankers should be unloaded using the same top unloading method for tank-trucks.

Transfer

Carbon steel piping is adequate for transferring cresylic acids within a facility at typical storage and transfer temperatures (~60°C / 140°F). See Table B. Corrosion of the piping and darkening of the cresylic acids increases with increasing temperature. Austenitic stainless steel (304L or 316L) offers better protection from corrosion at processing temperatures in excess of 175°C (~350°F). Aluminum, copper and brass should be avoided. As a general rule, the piping system should be welded and flanged to minimize threaded connections. A spiral wound metal gasket of stainless steel and graphite is recommended. Heat-tracing of the piping may be necessary to prevent plugging if the particular cresylic acid being transferred is subject to freezing. (See the product's safety data sheet.) A standard ANSI-type chemical process pump is recommended using a single rotating bellows seal with a carbon face running on a stationary silicon carbide seat. A steam quench is a good feature to have for a seal in this type of service.

Storage

Cresylic acids can be safely held in carbon steel vessels (drums, railcars, tanks, etc.) at typical storage and transfer temperatures (~60°C / 140°F). Long-term corrosion of the tank and darkening of the cresylic acids increases with increasing temperature. Exposure to oxygen and iron will also accelerate darkening of the material. Thus, the ideal storage condition for maintaining cresylic acids would be in an austenitic stainless steel vessel with nitrogen-blanketing and mild heat (if necessary). Teflon® or Dimetcote®-lined vessels are also a suitable option. With the exception of these, resin-lined storage vessels should generally be avoided. Cresylic acids may dissolve or otherwise adversely affect the lining. Prior testing of any polymer that may come in contact with cresylic acid is strongly recommended. Aluminum, copper and brass should be avoided

Heating may be necessary to maintain certain cresylic acid or xylenol products in liquid form. Mild heating using electrical tracing or low-pressure (60 psig / 414 kPa) steam is suggested to maintain the material's temperature 10-15°C (20-30°F) above its freeze point. The heat should be evenly distributed in larger tanks via mixing to prevent "hot spots" that may lead to accelerated color formation. Vents and relief devices should also be heated when freezable material is involved as they may become plugged if cresylic acid vapors condense and crystallize in them.

Polypropylene and polyethylene bottles are suitable for short-term storage of small, lab-scale samples. However, plastic bottles tend to become more brittle over time as the cresylic acids slowly leach the plasticizers from them, thus increasing the potential for a rupture during handling. Glass or compatible metal containers are suitable for longer-term storage of lab-scale samples. Of course, any sample storage must adhere to local regulatory requirements (ex. US CFR Title 29 Part 1910). Transport of cresylic acid samples must meet regulatory requirements in approved containers.

Processing

Austenitic stainless steels (304L or 316L) offer better protection from corrosion than carbon steel at processing temperatures in excess of 175°C (~350°F). High-nickel alloys, such as Alloy 20 and Hastelloy C-276 offer the best protection from corrosion. Aluminum, copper and brass must be avoided as their reaction with cresylic acids may proceed explosively at elevated temperatures. The capacity of the processing equipment should be designed to allow for the thermal expansion of the cresylic acids at the maximum processing temperature. Cresylic acid vapors produced during processing should either be vented and incinerated, neutralized via a caustic scrubber, or condensed and recycled.

Disposal

Ideally, all cresylic acids should be consumed or recycled in processing. Any unused or residual cresylic acids must be disposed of properly. They may be incinerated at a permitted disposal facility, subject to federal, state and local regulations or managed by a waste disposal company approved for handling cresylic acids.

Do not re-use empty containers. Empty containers should be incinerated at a permitted facility, in accordance with local regulations.

VI. Emergency Procedures

Physical Exposure - External

The primary dangers involved in working with cresylic acids are the corrosive and toxic effects resulting from a physical exposure. Studies suggest that the severity of the exposure depends more on the magnitude of the exposed skin area than the concentration of cresylic acid. Therefore, the critical factor in dealing with an external physical exposure to cresylic acids is to minimize the extent and duration of the contact. To this end, the immediate response must be thorough flushing of the exposed areas with copious amounts of running water to remove all the cresylic acid in contact with the skin or eyes. Any contaminated clothing should be removed as quickly and carefully as possible during this process to avoid any additional skin contact. Any exposed areas will have readily absorbed the cresylic acids and may be evidenced by a characteristic whitening of the skin. After thorough flushing with water, a solution consisting of 2 parts polyethylene glycol 400 to 1 part ethanol (PEG/EtOH) should be liberally applied to any affected skin (avoid contact with eyes), allowed to remain 15 to 30 seconds and then flushed away with fresh running water. Continue the cycling of PEG/EtOH and water for at least 15 minutes and then finish with thorough washing with soap and water. This decontamination procedure reduces the severity of the exposure, but does not completely eliminate damage to the skin or toxic effects. Medical attention should be sought as soon as possible.

Physical Exposure – Internal

Do NOT induce vomiting. Rinse mouth with water. Ingest immediately about 350 ml (5 ml/kg body weight) of activated charcoal slurry. Note: To prepare activated charcoal slurry, mix thoroughly 50 g of activated charcoal in 400 ml (about 2 cups) water. Never give anything by mouth to an unconscious person. Call a physician or poison control center immediately.

Fire-Fighting

Fires involving cresylic acids are best extinguished using water fog or AFFF (foam). Do not use a water jet. Dry chemical and CO₂ media are also effective. Approved self-contained breathing apparatus and protective clothing should be employed as needed to avoid inhalation of vapors or skin contact with the cresylic acids, smoke or run-off water while fighting the fire.

Spill Containment & Clean-Up

Spill containment and cleanup of cresylic acids should only be performed by properly trained personnel employing an appropriate level of protective equipment as dictated by the extent of the spill. Small to medium spills on land should be surrounded by and absorbed onto inert clay absorbent and transferred to a disposal container. Larger land-spills should be diverted away from waterways, contained with booms, dikes or trenches, and collected in a vacuum truck. Any residual cresylic acids remaining after vacuuming should be cleaned up using the clay absorbent. All soils affected by the spill should be removed and placed in approved disposal containers.

Water spills are of particular concern due to the acute toxicity of cresylic acids to marine life. Clean up efforts should focus on containing the spill and quickly removing the cresylic acids that settle in deeper areas of the waterway. This can be aided greatly if the flow of water can be slowed or stopped. Further efforts should focus on removing as much of the dissolved cresylic acids as possible from the water using activated charcoal.

The composition and extent of any spill should be evaluated against local guidelines (ex. SARA Title III and RCRA in the U.S.) and reported to the proper agencies, if necessary. Any non-disposable clean-up equipment should be thoroughly decontaminated with soap and water after use.

VII. Miscellaneous

Frequently Asked Questions

- **Q.** How do I clean a tank or vessel that has been used to store cresylic acids?
- **R.** Hot-water washing followed by thorough steaming should effectively clean the vessel. Dilute caustic solution, followed by a water wash will also remove trace cresols. The cresylic acids in waste water and steam condensate must be recovered and either recycled or properly disposed.
- Q. How do I control cresylic acid odors?
- **R.** Cresylic acids have an extremely low odor threshold (<1 ppm); thus their significant antiseptic/medicinal odor can be detected as a result of very minor releases. Odors may be minimized by venting all tanks and process equipment through a caustic scrubber or flare.
- **Q.** Can I order cresylic acids in plastic or plastic-lined drums?
- **R.** Sasol does not typically supply cresylic acid in plastic or plastic-lined drums. Cresylic acids are excellent solvents for many resins. In addition, cresylic acids may leach plasticizers from plastics, causing them to become increasingly brittle. Prior testing of any polymer that may come in contact with cresylic acid is strongly recommended.
- **Q.** What is the shelf-life of cresylic acids?
- **R.** Cresylic acids are stable and have an indefinite shelf-life with regard to composition. Depending upon storage conditions, they may absorb moisture from the air, darken in color over time, or leach low level metals from the storage vessel. Protection of drums from weather is advised to prevent accelerated external corrosion of drums.

Links to Useful Resources

<u>United States Code of Federal Regulations</u>
<u>American Society for Testing and Materials (ASTM)</u>
<u>American Chemistry Council</u>

References

Ullmann's Encyclopedia of Industrial Chemistry, 5th ed., v. A8, pp. 25-59.

ASTM D 3852-07, Standard Practice for Sampling and Handling Phenol, Cresols and Cresylic Acid.

Deichmann, W.B., and Witherup, S. "Phenol studies. VI. The acute and comparative toxicity of phenol and o-, m-, and p-cresols for experimental animals," J. Pharmacol. Exp. Ther. 82:377-390, 1944.

Sasol Safety Data Sheets

Monteiro-Riviere, Nancy A. et.al., "Efficacy of Topical Phenol Decontamination Strategies on Severity of Acute Phenol Chemical Burns and Dermal Absorption: In Vitro and In Vivo Studies in Pig Skin"

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TABLE A: ISOMER DATA

PHENOL & CRESOLS	PHENOL	o-CRESOL	OH ■-CRESOL	p-CRESOL OH
Melting point (°C @ 101.3 kPa)	40.9	31.0	12.2	34.8
Flash point (°C, closed cup)	79	81	94	94
Boiling point (°C @ 101.3 kPa)	181.7	191.0	202.2	201.9
Density [I] (g/cm ³ @ 50°C)	1.050	1.022	1.011	1.012
Thermal expansion [I] (% V / °C) [calc]	0.080	0.091	0.078	0.073
Heat capacity [s or I] (J mol ⁻¹ K ⁻¹ @ 25°C)	154.2	154.7	225.1	150.3
Solubility in water (wt.% @ 25°C)	8.3	2.6	2.3	1.9

XYLENOLS	HO 2,3-XYLENOL	2,4-XYLENOL	2,5-XYLENOL	2,6-XYLENOL	HO 3,4-XYLENOL	3,5-XYLENOL
Melting point (°C @ 101.3 kPa)	72.6	24.5	74.8	45.6	65.1	63.4
Flash point (°C, closed cup)	93	89	89	86	104	100
Boiling point (°C @ 101.3 kPa)	216.9	210.9	211.1	201.0	227.0	221.7
Density [I] (g/cm ³ @ [°C])	0.981 [85]	1.003 [40]	0.958 [85]	0.978 [65]	0.982 [80]	0.971 [75]
Thermal expansion [I] (% V / °C) [calc]	.095	0.083	0.095	0.098	0.083	0.089
Heat capacity [I] (J mol ⁻¹ K ⁻¹ @ 80°C)	274	281	285	246	280	296
Solubility in water (wt.% @ 25°C)	0.47	0.61	0.49	0.64	0.50	0.49

TABLE B: ENGINEERING SUGGESTIONS

METALLURGY	<u>Acceptable</u>	Suggested	<u>Optimal</u>
Tanks	Carbon steel	AISI Type 304L stainless steel	AISI Type 316L stainless steel
Piping	Carbon steel	AISI Type 304L stainless steel	AISI Type 316L stainless steel
Processing Equipment	AISI Type 304L stainless steel	AISI Type 316L stainless steel	Alloy 20 or Hastelloy® C-276

MATERIAL TRANSFER	<u>Suggested</u>	
Pumps	 ASME B73.1/2 horizontal or vertical centrifugal pumps (normal service) API 610 horizontal centrifugal pumps (severe service) 	
Seals	API 682 single, rotating, bellows mechanical seals with silicon carbide stationary faces, carbon rotating faces and Hastelloy® C- 276 bellows	
Gaskets	 Grafoil®-filled 304L stainless steel 316L stainless steel spiral-wound Flexitallic® (or equal) 	

SAFETY	<u>Suggested</u>	
Safety Showers	 Deluge-type emergency shower w/ heat-tracing for freeze protection (if necessary). Include storage for soap and PEG/EtOH. 	
Eyewash Stations	 Low-pressure, fresh-water eyewash w/ heat-tracing for freeze protection (if necessary). 	

TABLE C: PERSONAL PROTECTIVE EQUIPMENT (PPE)

PROTECTION LEVELS	<u>Activities</u>	Suggested PPE	
Level I (maximum)	 Line breaking Emergency maintenance Working with hot cresylic acids Tank cleaning Temporary connections Unplugging equipment bleeds. 	Chemical mono-goggles or safety glasses w/ side-shields, face shield, chemical resistant gloves, rubber boots, full slicker suit and respirator or SCBA (if necessary), hard hat if required.	
Level II	 Connecting/disconnecting hoses for transfers Dip sampling of storage or shipping vessels. Pulling line, tank or pump samples from a sampling spigot. Hazardous laboratory work.(high temperature, vacuum or pressure) 	Safety glasses w/ side-shields or chemical mono-goggles, face shield, chemical resistant gloves, slicker coat, and respirator (if necessary), hard hat if required.	
Level III	 Analysis and testing of lab-scale samples. Lab-scale process simulations (ambient temp and pressure). Walking through processing or handling areas. 	Safety glasses w/ side-shields, chemical-resistant gloves, lab coat or coveralls and respirator (if necessary), hard hat (if required).	
Level IV (minimum)	 Walking through laboratory areas. Non-chemical work in proximity to labscale samples of cresylic acids. 	Safety glasses w/ side-shields, long- sleeved shirts or lab coat, trousers, hard hat (if required).	

PPE MATERIALS	Suggested Materials
Chemical-resistant gloves (disposable contact protection)	 Nitrile rubber – for initial short-term contact protection Latex rubber – consider potential allergic reactions
Chemical-resistant gloves (heavy, re-usable)	Butyl rubber Viton®
Chemically-resistant coats/coveralls	Polyethylene-coated Tyvek®PVC-Nylon-PVC
Work uniform/lab coat	Heavy cotton
Slicker coats/suits	PVC-Nylon-PVCGoreTex®