
Chlorinated Phenols

SPECIFICATIONS

Identity by

Infrared spectroscopy Passes test

Gas chromatography/mass spectrometry Passes test

Assay by

Gas chromatography $\geq 98.0\%$

Acid–base titrimetry 98–102%

Thin-layer chromatography Passes test

TESTS

Identity by Infrared Spectroscopy. Analyze the sample using the general procedure cited on page 92. Identifying absorbances for the compound listed in Table 5-1 must be present. The spectrum is compared to the NIST standardized library of compounds, if available. Absorbances found in the NIST spectrum should be present in the test spectrum; however, absorbance frequency and intensity may be different due to different analysis techniques. The spectrum may also be compared to other published libraries of infrared spectra, if available.

Identity by Gas Chromatography/Mass Spectrometry. Analyze the sample by gas chromatography/mass spectrometry using method GCMS-CP1 described below.

Method GCMS-CP1

Ionization Mode: Electron ionization/70 eV

Column: 100% Dimethylpolysiloxane, 30 M \times 0.25 mm i.d., 0.25- μ m film thickness

Temperature Program: 80 °C for 12 min, then 5 °C/min to 250 °C

Carrier Gas: Helium at 0.8 mL/min

Injector: Split/splitless (20:1 split)

Injector Temperature: 250 °C

Scan Range: 45–500 amu

Sample Size: 1 μ L of a 400 μ g/mL solution

Identifying ions for the compound listed in Table 5-1 must be present. The spectrum is compared to the NIST standardized library of compounds, if available. Ions in the test spectrum must match those found in the NIST spectrum.

Table 5-1. Chlorinated Phenols Compound Data

Name	CAS No.	Chemical Formula	Formula Weight	IR (cm ⁻¹)			MS (m/z)		
				IR-1	IR-2	IR-3	MS-1	MS-2	MS-3
2-Chlorophenol	95-57-8	C ₆ H ₅ ClO	128.56	3584	1486	740	128	64	130
3-Chlorophenol ^a	108-43-0	C ₆ H ₅ ClO	128.56	3649	1600	885	128	65	130
4-Chlorophenol	106-48-9	C ₆ H ₅ ClO	128.56	3650	1495	825	128	65	130
2,3-Dichlorophenol	576-24-9	C ₆ H ₄ Cl ₂ O	163.00	3575	1450	770	162	164	126
2,4-Dichlorophenol	120-83-2	C ₆ H ₄ Cl ₂ O	163.00	3584	1480	720	162	164	63
2,5-Dichlorophenol	583-78-8	C ₆ H ₄ Cl ₂ O	163.00	3579	1478	906	162	164	63
2,6-Dichlorophenol ^a	87-65-0	C ₆ H ₄ Cl ₂ O	163.00	3575	1458	770	162	164	63
3,4-Dichlorophenol	95-77-2	C ₆ H ₄ Cl ₂ O	163.00	3450	1470	650	162	164	99
3,5-Dichlorophenol	591-35-5	C ₆ H ₄ Cl ₂ O	163.00	3640	1590	828	162	164	63
2,3,4,5,6-Pentachlorophenol	87-86-5	C ₆ HCl ₅ O	266.34	3557	1383	770	266	264	268
2,3,4,5-Tetrachlorophenol	4901-51-3	C ₆ H ₂ Cl ₄ O	231.89	3520	1410	725	232	230	234
2,3,4,6-Tetrachlorophenol	58-90-2	C ₆ H ₂ Cl ₄ O	231.89	3570	1445	750	131	232	230
2,3,5,6-Tetrachlorophenol	935-95-5	C ₆ H ₂ Cl ₄ O	231.89	3563	1404	700	232	230	234
2,3,4-Trichlorophenol	15950-66-0	C ₆ H ₃ Cl ₃ O	197.45	3575	1453	783	196	198	97
2,3,5-Trichlorophenol	933-78-8	C ₆ H ₃ Cl ₃ O	197.45	3571	1585	840	196	198	97
2,3,6-Trichlorophenol	933-75-5	C ₆ H ₃ Cl ₃ O	197.45	3567	1449	800	196	198	200
2,4,5-Trichlorophenol	95-95-4	C ₆ H ₃ Cl ₃ O	197.45	3584	1458	870	196	198	97
2,4,6-Trichlorophenol	88-06-2	C ₆ H ₃ Cl ₃ O	197.45	3575	1470	730	196	198	97
3,4,5-Trichlorophenol	609-19-8	C ₆ H ₃ Cl ₃ O	197.45	3653	1420	816	196	198	133

^a Use GC-CP2.

Assay by Gas Chromatography. Analyze the sample by gas chromatography using method GC-CP1 described below. Alternate method GC-CP2 should be used for specific compounds as noted in Table 5-1.

Method GC-CP1

Column: 5% Diphenyl-95% dimethylpolysiloxane, 30 M × 0.53 mm i.d., 1.5-μm film thickness

Detector: Thermal conductivity

Detector Temperature: 300 °C

Injector: Split/splitless (10:1 split)

Injector Temperature: 250 °C

Sample Size: 2 μL of a 1000 μg/mL solution

Carrier Gas: Helium at 3.0 mL/min

Temperature Program: 80 °C for 12 min, then 5 °C/min to 240 °C

Method GC-CP2

Column: 100% Dimethylpolysiloxane, 30 M \times 0.25 mm i.d., 0.25- μ m film thickness

Detector: Thermal conductivity

Detector Temperature: 300 °C

Injector: Split/splitless (10:1 split)

Injector Temperature: 250 °C

Sample Size: 2 μ L of a 1000 μ g/mL solution

Carrier Gas: Helium at 1.0 mL/min

Temperature Program: 80 °C for 12 min, then 5 °C/min to 240 °C

Measure the area under each peak (excluding the solvent peak), and calculate the analyte content in area percent.

Assay by Acid–Base Titrimetry. Follow the general procedure cited on page 27. The following specific conditions are also required.

Weigh enough sample so that a minimum of 25 mL of titrant will be used. Dissolve the sample in 2-propanol. Titrate the sample using a standardized solution of potassium hydroxide in methanol.

Assay by Thin-Layer Chromatography. Analyze the sample by thin-layer chromatography using the general procedure described on page 86. The following specific conditions are also required.

Stationary Phase: Silica

Mobile Phase: 1:1 (v/v) Ethyl acetate:hexane

Detection Methods: UV at 254 nm and iodine

Compound must exhibit a single spot.