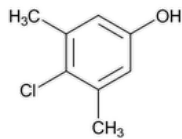


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Chloroxylenol



C_8H_9ClO 156.61
Phenol, 4-chloro-3,5-dimethyl-;
4-Chloro-3,5-xyleneol CAS RN[®]: 88-04-0; UNII: 0F32U78V2Q.

DEFINITION
Chloroxylenol contains NLT 98.5% of chloroxylenol (C_8H_9ClO).

- IDENTIFICATION**
- **A.** [SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), *Infrared Spectroscopy*: **197K**
 - **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay.

ASSAY

• **PROCEDURE**

Internal standard solution: 4 mg/mL of [USP Parachlorophenol RS](#) in toluene

Standard solution: 1 mg/mL of [USP Chloroxylenol RS](#) prepared as follows. Transfer 10 mg of [USP Chloroxylenol RS](#) to a 10.0-mL volumetric flask, add 2.0 mL of the *Internal standard solution*, and dilute with toluene to volume.

Sample solution: 1 mg/mL of Chloroxylenol prepared as follows. Transfer 10 mg of Chloroxylenol to a 10.0 mL volumetric flask, add 2.0 mL of the *Internal standard solution*, and dilute with toluene to volume.

Chromatographic system
(See [Chromatography \(621\)](#), *System Suitability*.)

Mode: GC

Detector: Flame ionization

Column: 0.32-mm × 30-m; coated with a 0.50-μm film of phase G42

Temperatures

Injection port: 250°

Detector: 250°

Column: See [Table 1](#).

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
105	0	105	1
105	6	190	8

Carrier gas: Helium
Flow rate: 2.4 mL/min
Injection volume: 2 μL

Split ratio: 25:1**Run time:** 23 min**System suitability****Sample:** *Standard solution***Suitability requirements****Resolution:** NLT 5.0 between the parachlorophenol peak and the chloroxylenol peak**Tailing factor:** NMT 1.5 for the chloroxylenol peak**Relative standard deviation:** NMT 1.5%**Analysis****Samples:** *Standard solution* and *Sample solution*Calculate the percentage of chloroxylenol (C_8H_9ClO) in the portion of Chloroxylenol taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times 100$$

 R_U = peak area ratio of the chloroxylenol peak to the parachlorophenol peak from the *Sample solution* R_S = peak area ratio of the chloroxylenol peak to the parachlorophenol peak from the *Standard solution* C_S = concentration of [USP Chloroxylenol RS](#) in the *Standard solution* (mg/mL) C_U = concentration of Chloroxylenol in the *Sample solution* (mg/mL)**Acceptance criteria:** NLT 98.5%**IMPURITIES**

- [RESIDUE ON IGNITION \(281\)](#): NMT 0.1%

Change to read:

- [▲Iron \(241\), Procedures, Procedure 1▲](#) (CN 1-JUN-2023)

Sample: 0.10 g

Analysis: Transfer the *Sample* to a suitable crucible, add 5 drops of sulfuric acid, and ignite at a low heat until thoroughly ashed. Add 10 drops of sulfuric acid to the carbonized mass, and heat cautiously until white fumes are no longer evolved. Ignite, preferably in a muffle furnace, at 500°–600°, until the carbon is completely burned off. Cool, add 4 mL of 6 N hydrochloric acid, cover, digest on a steam bath for 15 min, uncover, and slowly evaporate on a steam bath to dryness. Moisten the residue with 1 drop of hydrochloric acid, add 10 mL of hot water, and digest for 2 min. Dilute with water to 25 mL. Filter, if necessary. Rinse the crucible and the filter with 10 mL of water, combining the filtrate and rinsing in a 50-mL color-comparison tube; add 2 mL of hydrochloric acid; dilute with water to 47 mL; and mix.

Acceptance criteria: NMT 0.01%

- **LIMIT OF TETRACHLOROETHYLENE**

Internal standard stock solution: 20 µL/mL of 1-butanol in methanol**Internal standard solution:** 2 µL/mL of 1-butanol in methanol from *Internal standard stock solution***Tetrachloroethylene standard stock solution:** 20 µL/mL of tetrachloroethylene in methanol**Tetrachloroethylene standard solution:** 2 µL/mL of tetrachloroethylene in methanol from *Tetrachloroethylene standard stock solution*

Standard solution: 0.4 µL/mL each of 1-butanol and tetrachloroethylene in methanol from *Internal standard solution* and *Tetrachloroethylene standard solution*, respectively, prepared as follows. Combine 5 mL each of *Internal standard solution* and *Tetrachloroethylene standard solution* in a 25-mL volumetric flask, dilute with methanol to volume, and mix.

Sample solution: 160 mg/mL of chloroxylenol and 0.4 µL/mL of 1-butanol in methanol prepared as follows. Weigh 4 g of chloroxylenol in a 25-mL volumetric flask, combine with 5 mL of *Internal standard solution*, and dilute with methanol to volume.

Chromatographic system(See [Chromatography \(621\), System Suitability](#).)**Mode:** GC**Detector:** Flame ionization**Column:** 0.53-mm × 30-m; 1.0-µm film of phase G14 or G16**Carrier gas:** Hydrogen**Temperatures****Injector:** 240°**Detector:** 240°