

## **Parachlorophenol**

Type of Posting Revision Bulletin
Posting Date 30-Oct-2020
Official Date 01-Nov-2020
Expert Committee Small Molecules 1

In accordance with the Rules and Procedures of the Council of Experts, the Small Molecules 1 Expert Committee has indefinitely postponed the omission of the Parachlorophenol monograph. Comments were received that indicate the drug substance is referenced in the Camphorated Parachlorophenol monograph, which is needed to support public health.

The Parachlorophenol Revision Bulletin postponement will supersede the monograph omission becoming official on November 1, 2020.

Should you have any questions, please contact Morgan Puderbaugh, Senior Scientific Liaison (301-998-6833 or <a href="mailto:mxp@usp.org">mxp@usp.org</a>).

Revision Bulletin
Official: November 1, 2020

## Change to read:

# **Parachlorophenol**

<sup>▲</sup>The omission of this monograph has been postponed. As of November 1, 2020 this monograph remains official USP text. (RB 1-Nov-2020)

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 $C_6H_5CIO$  128.56

Phenol, 4-chloro-;

p-Chlorophenol [106-48-9]; UNII: 3DLC36A01X.

#### **DEFINITION**

Parachlorophenol contains NLT 99.0% and NMT 100.5% of parachlorophenol ( $C_6H_5CIO$ ).

#### **IDENTIFICATION**

• A.

Sample solution: 10 mg/mL of Parachlorophenol

**Analysis:** Add bromine TS dropwise to the *Sample solution*.

Acceptance criteria: A white precipitate is formed; at first it redissolves, but then it becomes permanent

as an excess of the reagent is added.

• B.

**Sample solution:** 10 mg/mL of Parachlorophenol

**Analysis:** Add 1 drop of ferric chloride TS to 10 mL of *Sample solution*.

**Acceptance criteria:** The solution acquires a violet-blue color.

• C.

**Analysis:** Heat a few crystals, held on a copper wire, in the edge of a nonluminous flame.

**Acceptance criteria:** A green color is imparted to the flame.

• D.

Sample: 1 g of Parachlorophenol

**Analysis:** Mix the *Sample* and 5 mL of sodium hydroxide solution (1 in 3), then add 1.5 g of monochloroacetic acid. Shake, and heat on a steam bath for 1 h. Cool, dilute with 15 mL of water, and acidify with hydrochloric acid. Extract with 50 mL of ether, wash the ether solution with 10 mL of cold water, then extract the ether solution with 25 mL of sodium carbonate solution (1 in 20). Acidify the solution with hydrochloric acid, collect the resulting precipitate on a filter, and recrystallize it from hot water.

Acceptance criteria: The resulting parachlorophenoxyacetic acid melts between 154° and 158°.

#### **ASSAY**

PROCEDURE

Sample: 1 g of Parachlorophenol

Titrimetric system
(See <u>Titrimetry (541)</u>.) **Mode:** Residual titration

Titrant: 0.1 N bromine VS

Back-titrant: 0.1 N sodium thiosulfate VS

Endpoint detection: Visual

**Analysis:** Transfer the *Sample* to a 500-mL volumetric flask, and dissolve and dilute with water to volume. Transfer a 25.0-mL portion of the solution to an iodine flask, cool in an ice bath to  $4^{\circ}$ , and add 20.0 mL of *Titrant*. Add 5 mL of hydrochloric acid, and immediately insert the stopper. Maintain the flask at a temperature of  $4^{\circ}$  for 30 min, shaking at frequent intervals. Allow it to stand for 15 min, remove the stopper just sufficiently to introduce quickly 5 mL of potassium iodide solution (1 in 5), taking care that no bromine vapor escapes, and at once insert the stopper in the flask. Shake thoroughly, remove the stopper, and rinse it and the neck of the flask with a small portion of water, allowing the washings to flow into the flask. Shake the mixture, and titrate the liberated iodine with *Back-titrant*, using 3 mL of starch TS as the indicator. Perform a blank determination. Each mL of 0.1 N bromine is equivalent to 3.214 mg of parachlorophenol ( $C_6H_5CIO$ ).

Acceptance criteria: 99.0%-100.5%

### **IMPURITIES**

## • LIMIT OF NONVOLATILE RESIDUE

**Sample:** 1 g of Parachlorophenol

**Analysis:** Heat the *Sample* in a tared container on a steam bath until it is volatilized, and dry at 105° for 1

h.

**Acceptance criteria:** NMT 0.1% of residue remains.

• LIMIT OF CHLORIDE

Sample solution: 10 mg/mL of Parachlorophenol

Analysis: Acidify 10 mL of Sample solution with 2 N nitric acid, and add a few drops of silver nitrate TS.

**Acceptance criteria:** No turbidity or opalescence is produced.

#### **SPECIFIC TESTS**

CLARITY AND REACTION OF SOLUTION

Sample solution: 10 mg/mL of Parachlorophenol

**Acceptance criteria:** Solution is clear and is acid to litmus.

• **Congeating Temperature** (651): Between 42° and 44°

# ADDITIONAL REQUIREMENTS

• PACKAGING AND STORAGE: Preserve in tight, light-resistant containers.

## **Page Information:**

Not Applicable

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