

cetylpyridinium chloride from Topical Solution to one-half of its original volume.

**Acceptance criteria:** The *Sample solution* meets the requirements of test A, (CN 1-May-2018) except that when silver nitrate TS is added, turbidity is produced rather than a curdy white precipitate.

## ASSAY

### PROCEDURE

**Sample solution:** Add a volume of Topical Solution nominally equivalent to 150 mg of cetylpyridinium chloride to a glass-stoppered, 500-mL graduated cylinder. Add 10 mL of chloroform, 0.4 mL of bromophenol blue solution (1 in 2000), and 5 mL of a freshly prepared solution of sodium bicarbonate (4.2 in 1000).

**Analysis:** Titrate the *Sample solution* with 0.02 M sodium tetraphenylboron VS until the blue color disappears from the chloroform layer. Add the last portions of the sodium tetraphenylboron solution dropwise, agitating vigorously after each addition. Each mL of 0.02 M sodium tetraphenylboron is equivalent to 7.160 mg of cetylpyridinium chloride ( $C_{21}H_{38}ClN \cdot H_2O$ ).

**Acceptance criteria:** 95.0%–105.0%

## ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers.
- **USP REFERENCE STANDARDS (11)**  
USP Cetylpyridinium Chloride RS

## Activated Charcoal

### DEFINITION

Activated Charcoal is the residue from the destructive distillation of various organic materials, treated to increase its adsorptive power.

### IMPURITIES

- **RESIDUE ON IGNITION (281)**  
**Sample:** 0.50 g  
**Acceptance criteria:** NMT 4.0%
- **ACID-SOLUBLE SUBSTANCES**  
**Sample:** 1.0 g  
**Analysis:** Boil the *Sample* with a mixture of 20 mL of water and 5 mL of hydrochloric acid for 5 min. Filter into a tared porcelain crucible, and wash the residue with 10 mL of hot water, adding the washing to the filtrate. To the combined filtrate and washing add 1 mL of sulfuric acid. Evaporate to dryness, and ignite to constant weight.  
**Acceptance criteria:** The residue weighs NMT 35 mg (NMT 3.5%).
- **CHLORIDE AND SULFATE, Chloride (221)**  
**Sample solution:** A 10-mL portion of the filtrate obtained in the test for *Reaction*  
**Acceptance criteria:** The *Sample solution* shows no more chloride than is contained in 1.5 mL of 0.020 N hydrochloric acid (NMT 0.2%).
- **CHLORIDE AND SULFATE, Sulfate (221)**  
**Sample solution:** A 10-mL portion of the filtrate obtained in the test for *Reaction*  
**Acceptance criteria:** The *Sample solution* shows no more sulfate than is contained in 1.0 mL of 0.020 N sulfuric acid (NMT 0.2%).
- **SULFIDE**  
**Sample:** 0.50 g  
**Analysis:** Place the *Sample* in a small conical flask. Add 20 mL of water and 5 mL of hydrochloric acid, and boil gently.  
**Acceptance criteria:** The escaping vapors do not darken paper moistened with lead acetate TS.

### CYANOGEN COMPOUNDS

**Sample:** 5 g

**Analysis:** Place the *Sample*, 50 mL of water, and 2 g of tartaric acid in a distilling flask connected to a condenser provided with a tightly fitting adapter, the end of which dips below the surface of a mixture of 2 mL of 1 N sodium hydroxide and 10 mL of water, contained in a small flask surrounded by ice. Heat the mixture in the distilling flask to boiling, and distill about 25 mL. Dilute the distillate with water to 50 mL, and mix. To 25 mL of the diluted distillate add 12 drops of ferrous sulfate TS, heat the mixture almost to boiling, cool, and add 1 mL of hydrochloric acid.

**Acceptance criteria:** No blue color is produced.

### Delete the following:

### HEAVY METALS (231)

**Sample:** 1.0 g

**Test preparation:** Boil the *Sample* with a mixture of 20 mL of 3 N hydrochloric acid and 5 mL of bromine TS for 5 min. Filter, and wash the charcoal and the filter with 50 mL of boiling water. Evaporate the filtrate and washing to dryness, and to the residue add 1 mL of 1 N hydrochloric acid, 20 mL of water, and 5 mL of sulfuric acid. Boil the solution until all of the sulfur dioxide is expelled. Filter if necessary, and dilute with water to 50 mL. To 20 mL of the solution add water to make 25 mL.

**Acceptance criteria:** NMT 50 ppm (Official 1-Jan-2018)

### UNCARBONIZED CONSTITUENTS

**Sample:** 0.25 g

**Analysis:** Boil the *Sample* with 10 mL of 1 N sodium hydroxide for 5 s, and filter.

**Acceptance criteria:** The filtrate is colorless.

### SPECIFIC TESTS

#### ADSORPTIVE POWER

**Alkaloids**

**Sample:** 1 g, previously dried at 120° for 4 h

**Analysis:** Shake the *Sample* with a solution of 100 mg of strychnine sulfate in 50 mL of water for 5 min, and filter through a dry filter, rejecting the first 10 mL of the filtrate. To a 10-mL portion of the subsequent filtrate add 1 drop of hydrochloric acid and 5 drops of mercuric-potassium iodide TS.

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

**Dyes**

**Sample:** 250 mg

**Analysis:** Pipet 50 mL of methylene blue solution (1 in 1000) into each of two glass-stoppered, 100-mL flasks. Add the *Sample* to one of the flasks, insert the stopper in the flask, and shake for 5 min. Filter the contents of each flask through a dry filter, rejecting the first 20 mL of each filtrate. Pipet 25-mL portions of the remaining filtrates into two 250-mL volumetric flasks. Add to each flask 50 mL of sodium acetate solution (1 in 10), mix, and add from a buret 35.0 mL of 0.1 N iodine VS, swirling the mixture during the addition. Insert the stoppers in the flasks, and allow them to stand for 50 min, shaking them vigorously at 10-min intervals. Dilute each mixture with water to volume, mix, allow to stand for 10 min, and filter through dry filters, rejecting the first 30 mL of each filtrate. Titrate the excess iodine in a 100-mL aliquot of each subsequent filtrate with 0.1 N sodium thiosulfate VS, adding 3 mL of starch TS as the endpoint is approached. Calculate the mL of 0.1 N iodine consumed in each titration.

**Acceptance criteria:** The difference between the two volumes is NLT 0.7 mL.

- **MICROBIAL ENUMERATION TESTS (61) and TESTS FOR SPECIFIED MICROORGANISMS (62):** It meets the requirements of the tests for absence of *Salmonella* species and *Escherichia coli*.