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Ichthammol

Ichthammol

CAS RN®: 8029-68-3; UNII: NQ14646378.

DEFINITION

Ichthammol is obtained by the destructive distillation of certain bituminous schists, sulfonation of the distillate, and neutralization of the product with ammonia. Ichthammol yields NLT 2.5% of ammonia (NH_3) and NLT 10.0% of total sulfur (S).

IDENTIFICATION

- **A.**

Sample solution: Ichthammol and water (10:90). Stir for 5 min with a magnetic stirrer.

Analysis: To the *Sample solution* add hydrochloric acid by 25% of the total volume. A heavy, resinous precipitate is formed. Remove the liquid by decantation, and wash the precipitate with 2 N hydrochloric acid until the last washing is nearly colorless. Transfer the precipitate to absorbent paper, allow it to stand for 10 min, and then transfer 10 mg of the precipitate to a 250-mL conical flask. To the flask add 100 mL of ether, attach an air condenser to the flask, and stir for 30 min with a magnetic stirrer.

Acceptance criteria: The precipitate does not dissolve completely.

- **B.**

Sample solution: 1 in 10

Analysis: To the *Sample solution* add 1 N sodium hydroxide, and heat to the boiling point.

Acceptance criteria: Ammonia is evolved.

ASSAY

- **Ammonia**

Sample solution: 50 mg/mL of Ichthammol in water

Titrimetric system

Mode: Residual titration

Titrant: 0.5 N sulfuric acid VS

Back-titrant: 0.5 N sodium hydroxide VS

Endpoint detection: Visual

Analysis: Transfer 100 mL of the *Sample solution* to a distillation flask, add 3 g of paraffin, and then add 20 mL of sodium hydroxide solution (4 in 10). Connect the flask to a condenser by means of a spray trap, and immerse the lower outlet tube of the condenser in 30.0 mL of *Titrant*. Distill slowly, collect 50 mL of distillate, and then titrate the excess acid with *Back-titrant*, using methyl red TS as the indicator. Perform a blank determination, and make any necessary correction. Each mL of 0.5 N sulfuric acid is equivalent to 8.515 mg of ammonia (NH_3).

Acceptance criteria: NLT 2.5% of ammonia (NH_3)

- **Total Sulfur**

Sample: 500–800 mg of Ichthammol

Analysis: Transfer the *Sample* to a Kjeldahl flask with the aid of 20 mL of water. Add 3 g of potassium chlorate, then slowly add 30 mL of nitric acid, and evaporate the mixture on a hot plate to 5 mL. Cool, repeat the oxidation with 3 g of potassium chlorate and 30 mL of nitric acid, and evaporate to 5 mL. Add 25 mL of hydrochloric acid, and again evaporate to 5 mL. Add 100 mL of water, heat to boiling, filter, and wash well. To the hot filtrate add 25 mL of barium chloride TS, and heat on a steam bath for 1 h. Collect the barium sulfate on a previously ignited and tared filtering crucible. Wash, dry, and ignite. Then cool, and weigh. Each g of barium sulfate is equivalent to 137.4 mg of total sulfur (S).

Acceptance criteria: NLT 10.0% of total sulfur (S)

IMPURITIES

- **Residue on Ignition ⟨281⟩:** NMT 0.5%
- **Limit of Ammonium Sulfate**

Sample solution: 40 mg/mL of Ichthammol in alcohol

Analysis: To 25 mL of *Sample solution* stir, filter, and wash the filter with a mixture of equal volumes of ether and alcohol until the last washing is clear and colorless. Air-dry the filter and residue, and pass 200 mL of warm water, slightly acidified with hydrochloric acid,

through the residue on the filter. Heat the filtrate to boiling, add barium chloride TS in excess, and heat for 1 h on a steam bath. Collect the precipitate of barium sulfate on a filter. Wash it well, dry, and ignite to constant weight. Each g of barium sulfate is equivalent to 566.1 mg of ammonium sulfate $[(NH_4)_2SO_4]$.

Acceptance criteria: NMT 8.0% of ammonium sulfate $[(NH_4)_2SO_4]$

SPECIFIC TESTS

• **Loss on Drying** ⟨731⟩

Analysis: Dry at 80° for 8 h, and continue the drying at 100° for constant weight.

Acceptance criteria: NMT 50.0%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
ICHTHAMMOL	Documentary Standards Support	SM12020 Small Molecules 1
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM12020 Small Molecules 1

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