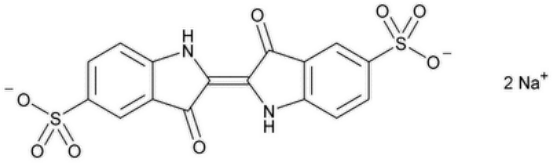
Status: Currently Official on 15-Feb-2025
Official Date: Official as of 01-Jun-2023
Document Type: USP Monographs
DocId: GUID-BE5D4F66-99C3-48A5-BDF9-FDABBEC261DB_2_en-US
DOI: https://doi.org/10.31003/USPNF_M40140_02_01
DOI Ref: 86ru2

Indigotindisulfonate Sodium



$C_{16}H_8N_2Na_2O_8S_2$ 466.35

1*H*-Indole-5-sulfonic acid,2-(1,3-dihydro-3-oxo-5-sulfo-2*H*-indol-2-ylidene)-2,3-dihydro-3-oxo-, disodium salt;

Disodium 3,3'-dioxo-[$\Delta^{2,2'}$-biindoline]-5,5'-disulfonate CAS RN®: 860-22-0; UNII: D3741U8K7L.

DEFINITION

Indigotindisulfonate Sodium contains NLT 96.0% and NMT 102.0% of sodium indigotinsulfonates, calculated on the dried basis as indigotindisulfonate sodium ($C_{16}H_8N_2Na_2O_8S_2$).

IDENTIFICATION

• **A.** **Identification Tests—General**, *Sodium*⟨191⟩and*Sulfate*⟨191⟩

Sample: Incinerate a portion of it.

Acceptance criteria: The residue meets the requirements.

• **B.** The addition of hydrochloric acid to a solution of it changes the color to bluish violet, and further dilution with water restores the original color.

• **C.** The addition of 1 N sodium hydroxide to a solution of it changes the color to yellow or olive-brown.

• **D.** The addition of sodium chloride to a solution of it produces a blue precipitate.

ASSAY

• **Procedure**

Standard solution: 10 µg/mL in USP Indigotindisulfonate Sodium RS in dilute hydrochloric acid (1 in 100)

Sample solution: 10 µg/mL of Indigotindisulfonate Sodium in dilute hydrochloric acid (1 in 100)

Blank: Dilute hydrochloric acid (1 in 100)

Instrumental conditions

Mode: Vis

Analytical wavelength: 610 nm

Cell: 1 cm

Analysis

Samples: *Standard solution*, *Sample solution*, and *Blank*

Calculate the percentage of indigotindisulfonate sodium ($C_{16}H_8N_2Na_2O_8S_2$) in the portion of Indigotindisulfonate Sodium taken:

$$\text{Result} = (A_U/A_S) \times (C_S/C_U) \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

C_S = concentration of USP Indigotindisulfonate Sodium RS in the *Standard solution* (µg/mL)

C_U = concentration of Indigotindisulfonate Sodium in the *Sample solution* (µg/mL)

Acceptance criteria: 96.0%–102.0% on the dried basis

OTHER COMPONENTS

• **Sulfur Content**

Sample solution: Place 25 mg in halide-free filter paper measuring 4 cm square, and fold the paper to enclose it.

Analysis: Proceed as directed in *Oxygen Flask Combustion ⟨471⟩*, using a 1-L flask and a mixture of 25 mL of water and 5 mL of hydrogen peroxide TS as the absorbing liquid. When the combustion is complete, place a few mL of water in the cup, loosen the stopper, and rinse the stopper, the specimen holder, and the sides of the flask with 20 mL of water. Add 2 mL of hydrochloric acid, dilute with water to 250 mL, heat to boiling, and slowly add 10 mL of barium chloride TS. Heat the mixture on a steam bath for 1 h, and collect the precipitate of barium sulfate on a filter. Wash until it is free from chloride, dry, ignite, and weigh. Each g of residue is equivalent to 137.4 mg of sulfur.

Acceptance criteria: 13.0%–14.0% on the dried basis

IMPURITIES

Change to read:

• ▲*Arsenic ⟨211⟩, Procedures, Procedure 2*▲ (CN 1-Jun-2023) : NMT 8 ppm

Change to read:

• ▲*Lead ⟨251⟩, Procedures, Procedure 1*▲ (CN 1-Jun-2023)

Sample solution: Place 4.0 g in a Kjeldahl flask, moisten with water, and add 10 mL of sulfuric acid and 5 mL of nitric acid. As soon as the first violent reaction subsides, heat until most of the brown fumes are expelled. Repeat the addition of nitric acid, 1–3 mL at a time, and heat until the Indigotindisulfonate Sodium is practically decomposed and most of the organic matter is in solution. Then add, cautiously and in small portions, 5 mL of perchloric acid. When the violent reaction subsides, continue the addition of small amounts of nitric acid, and heat as before until a colorless solution is obtained. (If the solution fails to become clear in 10–20 min after the addition of the perchloric acid, add 1–3 mL more of this acid, and continue the nitric acid treatment until the solution is colorless.) Boil for 10–15 min, cool, and neutralize with 1 N sodium hydroxide. Transfer to a 100-mL volumetric flask, and dilute with water to volume.

Analysis: Test 5 mL of the *Sample solution* according to the limit test for ▲*Lead ⟨251⟩, Procedures, Procedure 1*▲ (CN 1-Jun-2023) using 3 mL of Ammonium Citrate Solution, 1 mL of Potassium Cyanide Solution, and 0.5 mL of Hydroxylamine Hydrochloride Solution.

Acceptance criteria: 5 mL of the *Sample solution* contains NMT 2 µg of lead (corresponding to NMT 0.001%).

SPECIFIC TESTS

• **Loss on Drying ⟨731⟩**

Analysis: Dry at 105° for 3 h.

Acceptance criteria: NMT 5.0%

• **Water-Insoluble Substances**

Sample solution: 10.0 mg/mL in water

Analysis: Pass 100 mL of the *Sample solution* through a tared filtering crucible, wash with water until the filtrate is practically colorless, and dry the residue at 105° for 1 h.

Acceptance criteria: The weight of the residue does not exceed 5 mg.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers. Store at 25°, excursions permitted between 15° and 30°.

• **USP Reference Standards ⟨11⟩**

USP Indigotindisulfonate Sodium RS

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

Topic/Question	Contact	Expert Committee
INDIGOTINDISULFONATE SODIUM	Documentary Standards Support	SM42020 Small Molecules 4

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 29(6)

Current DocID: GUID-BE5D4F66-99C3-48A5-BDF9-FDABBEC261DB_2_en-US

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