

## UNITED STATES DEPARTMENT OF COMMERCE

WASHINGTON 25, D. C.

# National Bureau of Standards

## Certificate of Analysis

Standard Sample 83b

Arsenic Trioxide

Analysis<sup>a</sup>

Purity on basis of titration <sup>a</sup>	Nonvolatile matter	Lead	Chloride	Antimony	Iron	<sup>b</sup> Density <sup>20</sup> / <sub>4</sub>
Percent 100.00	Percent 0.005	Percent <0.001	Percent <0.005	Percent <0.002	Percent <0.0005	3.71

<sup>a</sup> By Keith M. Sappenfield, National Bureau of Standards.<sup>b</sup> International Critical Tables, I, p. 110; 1926.

*Purity on basis of titration.* In tests made at the National Bureau of Standards, Standard 83b showed a purity of 100.00 percent when standardized by means of purified iodine. The weights of arsenic trioxide and iodine were corrected to vacuum standard, weight burets were used, and all calculations were based on the 1953 International Table of Atomic Weights of the chemical elements.

*Drying.* Sample 83b when analyzed at the National Bureau of Standards showed no loss in weight when dried at 105°C.

## DIRECTIONS FOR USE

*Indicator.* Triturate 2.5 g of soluble starch with a little water and pour the suspension into 500 ml of boiling water. Cool the solution, add 0.5 ml of *N* HCl, mix, transfer to a glass-stoppered bottle and keep in a refrigerator.

*Preparation of approximately 0.1 N iodine solution.* Dissolve 12.7 g of resublimed iodine and 60 g of potassium iodide, reagent quality, in 75 ml of water. When the iodine has dissolved, transfer the solution to a glass-stoppered liter flask, dilute to the mark with water, and mix thoroughly.

*Standardization of the iodine solution.* Transfer approximately 0.2 g of arsenic trioxide to a clean, accurately weighed cylindrical weighing bottle and weigh accurately. Place the bottle with contents in a 200-ml round flat-bottomed Pyrex flask. Add 5 g of sodium carbonate and mix as thoroughly as possible with the arsenic trioxide. Insert a short-stemmed funnel in the neck of the flask and add 25 ml of water in 5-ml portions, shaking the flask after each addition. Heat gently until complete solution is effected. Cautiously add 15 ml of dilute sulfuric acid (1:5). Mix thoroughly and cautiously add 50 ml of a solution of sodium bicarbonate (40 g per liter). Rinse and remove the funnel. Titrate slowly with the iodine solution during constant agitation, until most of the iodine has been added. (0.2 g of As<sub>2</sub>O<sub>3</sub> requires approximately 40.4 ml of 0.1 *N* iodine.) Add 5 ml of starch solution and continue the titration until the initial pink coloration just passes to a clear blue. Deduct from the volume of iodine solution consumed the amount required to produce the same color in a solution composed of the added reagents and 40 to 50 ml of freshly boiled and cooled water in which 5 g of potassium iodide has been dissolved.

Calculate the titre of the iodine solution on the basis of the following equation:



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A. V. ASTIN, Director.

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