U. S. DEPARTMENT OF COMMERCE

# National Bureau of Standards

## Certificate of Analysis

OF

## STANDARD SAMPLE 83a

## ARSENIC TRIOXIDE

ANALYSIS[a]

| Purity on basis of titration | Nonvolatile matter | Sulfides | Chlorides | Antimony | Iron | Other foreign metals | [b] Density $\frac{20}{4}$ |
|---|---|---|---|---|---|---|---|
| Percent | Percent | Percent | Percent | Percent | Percent | | |
| 99.99 | 0.002 | $<0.00005$ | $<0.0005$ | 0.01 | $<0.0005$ | None found | 3.71 |

[a] By Howard B. Knowles, National Bureau of Standards.
[b] International Critical Tables, 1, p. 110 ; 1926.

*Purity on basis of titration.* In tests made at the National Bureau of Standards, Standard 83a showed a purity of 99.99 percent when standardized by means of purified iodine. The weights of arsenic trioxide and iodine were corrected to vacuum standard, weight burettes were used, and all calculations based on the 1941 International Table of Atomic Weights of the chemical elements.

*Drying.* Sample 83a when analyzed at the National Bureau of Standards showed no loss in weight when dried at 105° C. Drying is necessary only if the sample has been exposed to a humid atmosphere, or if an accuracy exceeding 1 part in 2000 is desired.

### 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 purified iodine and 60 g of pure potassium iodide 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 arsenious oxide 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 10 ml of normal NaOH, insert a short-stemmed funnel in the neck of the flask and agitate gently until complete solution is effected. Add 15 ml of normal $H_2SO_4$, mix thoroughly and cautiously, add 50 ml of a solution of $NaHCO_3$ (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 $As_2O_3$ 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 KI has been dissolved.

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

$$As_2O_3+2I_2+2H_2O \rightarrow As_2O_5+4HI$$

For a full discussion of this titration see "The Theory and Practice of the Iodometric Determination of Arsenious Acid," by Edward W. Washburn, J. Am. Chem. Soc., **30**, 31 (1908).

E. U. CONDON, *Director.*

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