DEPARTMENT OF COMMERCE

Bureau of Standards

Certificate of Analysis

OF

STANDARD SAMPLE No. 83

ARSENIC TRIOXIDE (As_2O_3)

ANALYSIS [a]

Purity on basis of titration	Nonvolatile matter	Sulphides	Chlorides	Antimony	Iron	Other foreign metals	[b] Density $\frac{20}{4}$
Per cent 99. 97	*Per cent* 0. 014	*Per cent* <0. 001	*Per cent* <0. 002	*Per cent* <0. 005	*Per cent* <0. 003	None found	3. 71

[a] By James I. Hoffman, Bureau of Standards. [b] International Critical Tables, 1, p. 110; 1926.

All calculations on this certificate are based on the 1925 International Table of Atomic Weights of the Chemical Elements.

Purity on Basis of Titration.—In tests made at the Bureau of Standards sample No. 83 showed a purity of 99.98 per cent when standardized by means of purified iodine, and 99.96 per cent when standardized by means of an iodine solution that had been standardized against sodium oxalate through potassium permanganate and sodium thiosulphate. The iodine used in the first titration was originally 99.97 per cent pure, and this was further purified by grinding with potassium iodide, and subliming three times. In the standardization by means of sodium oxalate, the Bureau of Standards Standard sample No. 40a was used on the basis of 99.96 per cent purity. The weights of sodium oxalate, arsenic trioxide and iodine were corrected to vacuo, weight burettes were used throughout the work, and approximately the same volumes of solution were used.

Drying.—Sample No. 83 is not appreciably hygroscopic, and it is unnecessary to dry it before use unless an accuracy better than 1 part in 2,000 is desired. In such case, the sample should be dried for 1 hour at 105° C. immediately before use. No appreciable error is introduced through weighing in an open container. When the sample was analyzed at the Bureau of Standards, it showed no loss on drying for 1 hour at 105° C., but gained 0.014 per cent when exposed for two weeks to an atmosphere having a relative humidity of 90 per cent.

Indicator.—The starch solution used in most of the work at the Bureau of Standards was prepared by grinding 5 g of soluble starch to a paste in an agate mortar, washing the paste into a flask with 400 ml of boiling water, and then cooling to approximately 20° C. The solution was then treated with 50 ml of a 25 per cent solution of potassium iodide and 50 ml of a cool 10 per cent solution of sodium hydroxide, and saturated with carbon dioxide. Solutions made directly from arrow root or potato starch are also satisfactory and can be prepared as directed by Alsberg, Griffing & Field, J. Am. Chem. Soc., **48**, pp. 1299–1300; 1926.

1. **Directions for use.**—*Preparation of approximately 0.1 N arsenite solution.*—The sample for use is preferably weighed by difference in a small weighing bottle owing to the difficulty of completely brushing arsenious oxide from metallic or glass surfaces. Accurately weigh a stoppered weighing bottle containing approximately 4.95 g of arsenious oxide. Transfer without loss to a graduated liter flask and again weigh the bottle. Do not attempt to brush out the adhering oxide. Moisten the sample with water, add 15 g of pure sodium hydroxide and 100 ml of distilled water. Swirl the contents of the flask gently until the arsenious oxide is in solution. Dilute to 250 ml with water and saturate the solution with carbon dioxide, thus converting all of the sodium hydroxide to sodium bicarbonate. Dilute to the mark, mix thoroughly, and stopper the flask. A solution thus prepared will preserve its titre almost indefinitely. If the solution is made up on a volume basis corrections must afterwards be made for temperature changes.

2. *Preparation of approximately 0.1 N iodine solution.*—Dissolve 12.7 g of resublimed iodine and 20 g of pure potassium iodide in 50 ml of water. When the iodine has dissolved transfer the solution to a glass-stoppered litre flask, dilute to the mark with water, mix thoroughly, and stopper the flask.

3. *Standardization of the iodine solution.*—Transfer an accurately measured portion (40 to 50 ml) of the arsenite solution to a flask and titrate with the 0.1 *N* iodine solution, using starch solution as indicator. *In order to obtain accurate results it is absolutely necessary that the solution be saturated with carbon dioxide at the end of the titration.* A current of carbon dioxide may be passed through the solution for a few minutes just before the endpoint is reached, or a few drops of hydrochloric acid may be added to liberate sufficient carbon dioxide to saturate the solution. If the flask is stoppered immediately after the completion of the titration, the pink or rose-colored endpoint is stable for days.

From the quantities of iodine and arsenite solutions used 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**, p. 31; 1908.

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Director.

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