DEPARTMENT OF COMMERCE

Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE No. 84

ACID POTASSIUM PHTHALATE ($HKC_8H_4O_4$)

ANALYSIS[1]

Purity on basis of titration	Chlorides	Sulphates	Heavy Metals	Iron	*Density $\frac{20}{4}$
Per cent 99.97	*Per cent* <0.001	None found	None found	*Per cent* <0.001	1.636

[1] By James I. Hoffman, Bureau of Standards.

*International Critical Tables 1926, Vol. 1, p. 155.

The observations on this certificate are based on the 1925 International Table of Atomic Weights of the Chemical Elements.

EFFECTIVE NEUTRALIZING POWER.—When sample No. 84 was dried at 120° C. and weighed in air against brass weights results agreed within 1 part in 3,500 with those obtained with the Bureau of Standards Standard Benzoic Acid No. 39c, and within part in 5,000 with those obtained with redistilled and diluted hydrochloric acid that had been standardized by precipitation as silver chloride. When the weights of silver chloride, benzoic acid, and acid potassium phthalate were corrected to vacuo, agreement to better than 1 part in 4,000 was obtained in comparison with sample No. 39c and 1 part in 3,500 in comparison with hydrochloric acid. Sample No. 84, therefore, has a neutralizing power equivalent to an acid potassium phthalate having a purity of 99.97 per cent.

DRYING.—This material is not appreciably hygroscopic, and it is unnecessary to dry it before using unless an accuracy better than 1 part in 2,000 is desired. If such accuracy is desired, the sample should be dried for 1 to 2 hours at 120° C. immediately before use. No appreciable error is introduced by weighing it directly in an open container. When the sample was analyzed at the Bureau of Standards it lost 0.020 per cent when dried for 2 hours at 120° C., and both dried and undried samples gained 0.020 per cent when exposed for 8 days to an atmosphere having a relative humidity of 90 per cent.

INDICATORS.—Phenolphthalein is a satisfactory indicator for use with this sample. If other indicators are used, they must show a color change at approximately pH 9.

Serious errors may be introduced if the alkaline solution is standardized with phenolphthalein as indicator and subsequently used with an indicator, such as methyl orange. This error is especially significant if only small volumes of the titrating solutions are used, or if the titration is made in a large final volume.

Alkaline solutions standardized against acid potassium phthalate with phenolphthalein as indicator can afterwards be used for the standardization of acid solutions with methyl orange as indicator provided titrations are made in the same volume in both cases and a correction is applied for the volume of acid required to pass from the phenolphthalein end point to that of methyl orange. Theoretically this correction is approximately 0.10 cc of 0.1 *N* solution if the final volume of the titrated solution is 100 cc. An accuracy to at least 1 part in 1,000 can be attained if the correction is determined as follows: Add three drops of a 1 per cent solution of phenolphthalein to 100 cc of recently boiled water, and then add sufficient alkali solution to give an end point with phenolphthalein. Disregard the quantity of alkali solution added, and take the burette readings from this point. Now add 3 drops of a 0.02 per cent solution of methyl orange and sufficient 0.1 *N* acid to produce the pink color of methyl orange. Titrate back with 0.1 *N* alkali solution to the same end point as is taken in the usual titration (preferably pH=4.2).

Buffered solutions of 3.8, 4.0, and 4.2 pH are useful in accurately determining the methyl orange end point. Such solutions can be purchased or can be easily prepared according to the formulas given in "The Determination of Hydrogen Ions," by W. Mansfield Clark, 2d ed., p. 106, 1922; and in International Critical Tables, Vol. 1, p. 81, 1926.

If the acid and alkali solutions are equivalent, the quantity of acid minus the quantity of alkali solution represents the quantity of acid required to pass from the phenolphthalein end point to that of methyl orange. In actual tests the corrections in a final volume of 100 cc amounted to an average of 0.13 cc of 0.1 *N* acid, and in a volume of 500 cc the corrections amounted to 0.52 cc of 0.1 *N* acid.

DIRECTIONS FOR USE IN ACIDIMETRY.—Dry the sample for 1 hour at 120° C., and cool in a desiccator containing a good desiccant, such as concentrated sulphuric acid. Accurately weigh about 1 g of the dried acid potassium phthalate and transfer to a 300 cc flask which has been swept free from carbon dioxide. Add 50 cc of cool water that is free from carbon dioxide, stopper the flask, and shake gently until the sample is dissolved. When the sample is in solution, add 3 drops of a 1 per cent solution of phenolphthalein and titrate with an approximately 0.1 *N* solution of sodium hydroxide that is free from carbon dioxide, taking precautions to exclude the latter.

Determine the quantity of sodium hydroxide required to produce the end point by matching the color in another flask containing the indicator and the same volume of solution free from carbon dioxide. Subtract the amount required from that used in the first titration and calculate the normality of the alkali solution on the basis of the following equation:

$$HKC_8H_4O_4 + NaOH = NaKC_8H_4O_4 + H_2O$$

In acidimetry, 204.136 g of acid potassium phthalate is equivalent to 1.008 g of hydrogen and 1.0207 g is equivalent to 50 cc of 0.1 *N* solution.

George K. Burgess

Director.

Washington, D. C.
May 27, 1927

U. S. GOVERNMENT PRINTING OFFICE: 1927