U. S. DEPARTMENT OF COMMERCE

National Bureau of Standards

Certificate of Analysis

OF

STANDARD SAMPLE 84a

ACID POTASSIUM PHTHALATE ($HKC_8H_4O_4$)

ANALYSIS[a]

Indicated purity on basis of titration	Chlorides	Sulphates	Heavy metals	Iron	[b] Density at 20° C.
Percent 100.00	*Percent* <0.001	None found	None found	*Percent* <0.0005	g/cm³ 1.636

[a] By John L. Hague, National Bureau of Standards.

[b] International Critical Tables, 1, 155 (1926)

All calculations on this certificate are based on the 1937 International Table of Atomic Weights.

DRYING.—(*a*) *Sample as issued:* The diameter of the crystals of the material as issued ranges from about 0.18 mm (U. S. Standard Sieve No. 80) to approximately 1.7 mm (U. S. Standard Sieve No. 12). The particle size of about 70 percent of the sample lies between the No. 20 and No. 60 sieves. When the sample is dried at 120° C. the entrapped water is removed rather slowly. The loss in weight is about 0.02 percent when the material is dried at 120° C. for 2 hours, approximately 0.08 percent for 120 hours, and about 0.11 percent for 500 hours. After 500 hours the change in weight is less than 0.00005 percent per hr.

(*b*) *Sample ground to pass a U. S. Standard Sieve No. 100:* When the sample is lightly crushed to a fineness of approximately 100 mesh, much of the entrapped water is lost and most of what remains can be driven off by heating for 2 hours at 120° C. For example, the loss on drying for 2 hours at 120° C. then approximates 0.015 percent and for 120 hours about 0.02 percent. The crushed sample shows no significant change in weight after drying for 2 hours at 120° C.

EFFECTIVE NEUTRALIZING POWER.—When the crushed sample was dried for 2 hours at 120° C. and titrated with standard 0.1 *N* sodium hydroxide, as described in the "Directions for Use in Acidimetry", a neutralizing power equivalent to that of an acid potassium phthalate having a purity of 100.00 percent was indicated. The alkali solution was standardized by means of a 0.1 *N* solution of hydrochloric acid prepared from redistilled acid, and standardized by precipitating silver chloride and drying the latter at 165° C. The weights of silver chloride and phthalate were corrected to the vacuum standard.

When the sample as issued is dried and titrated, the percentage increase in purity, on the basis of titration, is of about the same order of magnitude as the percentage loss of weight on drying. For example, when the uncrushed material was dried for 2 hours at 120° C. the loss was 0.02 percent and the indicated purity of the dried sample was 99.93 percent. When dried for 120 hours, the loss was 0.08 percent and the purity of the dried sample 99.99 percent.

STABILITY.—Tests show that, under the conditions existing in the average laboratory, standard aqueous solutions of acid potassium phthalate do not change in strength. However, such solutions are not of much advantage since the procedure of weighing the phthalate, dissolving it in water, and immediately titrating the solution with alkali is relatively simple. (National Bureau of Standards Research Paper RP852.)

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 quantities 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 ml of 0.1 *N* solution if the final volume of the titrated solution is 100 ml. An accuracy of at least 1 part in 1,000 can be attained if the correction is determined as follows: Add 3 drops of a 1-percent solution of phenolphthalein to 100 ml 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-percent 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 that is taken in the usual titration (preferably pH=4.2).

Buffered solutions of pH 3.8, 4.0, and 4.2 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, **1**, 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 ml amounted to an average of 0.13 ml of 0.1 *N* acid, and in a volume of 500 ml, to 0.52 ml of 0.1 *N* acid.

DIRECTIONS FOR USE IN ACIDIMETRY.—Lightly crush a few grams of the sample to a fineness of approximately 100 mesh and dry for 1 to 2 hours at 120° C. Cool in a desiccator containing a good desiccant, such as concentrated sulfuric acid. Accurately weigh about 1 g of the dried acid potassium phthalate and transfer it to a 300 ml flask which has been swept free of carbon dioxide. Add 50 ml 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-percent solution of phenolphthalein and titrate with an approximately 0.1 *N* solution of sodium hydroxide that is free from carbonates, taking precautions to exclude carbon dioxide.

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, 204.215 g of acid potassium phthalate is equivalent to 1.0078 g of hydrogen and 1.0211 g is equivalent to 50 ml of 0.1 *N* solution.

Washington, D. C.
March 7, 1938

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Director.

U. S. GOVERNMENT PRINTING OFFICE 59760