U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

PROVISIONAL CERTIFICATE OF ANALYSIS

OF

STANDARD SAMPLE 84b

ACID POTASSIUM PHTHALATE ($HKC_8H_4O_4$)
(Acidimetric or pH Standard)

Material

This lot of acid-potassium phthalate was prepared to insure material of high purity and uniformity. It is not, however, to be considered as entirely free from impurities such as traces of chlorides, sulfur compounds and heavy metals. On the basis of titration a purity of 100.04 percent was indicated.

Acidimetric Standard

DRYING.- This material is not appreciably hydroscopic. The sample as issued shows less than 0.01 percent loss on drying for 2 hours at 120° C.

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.

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 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 pH 7 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 1000 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 end point that is taken in the usual titration (preferably pH ≈ 4.2).

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.

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).

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 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 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, 204.216 g of acid potassium phthalate is equivalent to 1.0080 g of hydrogen and 1.0211 g is equivalent to 50 ml of 0.1 N solution.

pH Standard

The pH of a 0.05 Molal solution of standard 84b on the acitivity basis is 4.00 at 25° C.

This value is derived from equations involving emf data and values of natural constants accepted by the National Bureau of Standards. At other temperatures between 0° and 60° inclusive the pH is given, by the equation:

$$pH = 5.13 \log T + 1519.62/T + 0.01092\ T - 17.047.$$

$$T = t^{\circ}\ C + 273.16$$

Preparation of a 0.05 Molal solution.- Add 10.211 g of the salt (dried for one hour at 120° C) to 1000 g of CO_2- free distilled water (pH 6.7-7.3), 10.151 g of salt to one liter of water at 25° C or 10.193 g of salt to one liter of water at 20° C.

(Signed) LYMAN J. BRIGGS, Director
G.E.F.L.