U. S. DEPARTMENT OF COMMERCE
WASHINGTON

NATIONAL BUREAU OF STANDARDS

PROVISIONAL CERTIFICATE

STANDARD SAMPLE 84f

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

This lot of acid potassium phthalate was prepared to insure material of high purity and uniformity, but should not be considered as entirely free from impurities such as occluded water and traces of free phthalic acid, chlorides, sulfur compounds and heavy metals. The material assays 99.99 percent $KHC_8H_4O_4$. This value was obtained by comparison titration against NBS standard 84d, using the value of 99.98$_8$ for 84d obtained by R. G. Bates and E. Wichers. (Cf. "Precise Intercomparison of Acids by Differential Potentiometric Titration with Hydrogen Electrodes." J. Research 59. 9 (1957) RP2769).

DRYING.- The sample as issued contains some entrapped water which is removed rather slowly when the crystals are dried at 120° C. The loss in weight when the uncrushed crystals are dried at 120° C for 2 hours is about 0.01 percent and for 600 hours about 0.04 percent.

When the crystals are crushed to a fineness of approximately 100 mesh, most of the entrapped water is lost during the crushing. The crushed material when dried for 2 hours at 120° C shows less than 0.01 percent loss in weight and no further loss after heating at 120° C for 120 hours.

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 because 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).

DIRECTIONS FOR USE IN ACIDIMETRY.- Crush (do not grind) a few grams of the sample to a fineness of approximately 100 mesh and dry for 1 to 2 hours at 120° C. Place in a small glass-stoppered container and cool in a desiccator. 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 water (25 to 28° C) that is free from carbon dioxide, stopper the flask, and shake gently until the sample is dissolved. Titrate to a pH of 8.6 with

an approximately 0.1 N standard solution of sodium hydroxide free from carbonates, taking precautions to exclude carbon dioxide and using as an indicator either a pH meter of the glass-electrode type or 3 drops of a 1-percent solution of phenolphthalein. In the latter case the end point can be determined by comparison with the color of a buffer solution (pH 8.6) prepared by mixing 25 ml of an $M/5H_3BO_3$, M/5KCl solution with 6 ml of M/5NaOH, 3 drops of a 1-percent solution of phenolphthalein and diluting to 100 ml with water free from carbon dioxide, (Cf. The Determination of Hydrogen Ions, W. M. Clark, p. 201, 3d Ed., 1928).

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, 204.228 g of acid potassium phthalate is equivalent to 1.0080 g of hydrogen and 1.02114 g is equivalent to 50 ml of 0.1 N solution.

Signed: Edward Wichers, Chief
Chemistry Division

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Washington, D. C.