U. S. Department of Commerce
John T. Connor, Secretary
National Bureau of Standards
A. V. Astin, Director

Certificate of Analysis

Standard Reference Material 84h
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. It conforms to the American Chemical Society specification for analytical reagent grade material, but should not be considered as entirely free from impurities such as occluded water and traces of chlorides, sulfur compounds, and heavy metals. The material assays 99.99 percent ±0.01 percent $HKC_8H_4O_4$. This value was obtained by coulometric titration using a modification of the method described by J. K. Taylor and S. W. Smith. [Cf. "Precise Coulometric Titrations of Acids and Bases," J. Res. NBS **63A**, 153 (1959)]. The value of the faraday used in this work was 96,487.0 coulombs per gram-equivalent [Cf. "New Values for the Physical Constants Recommended by NAS-NRC," NBS Technical News Bulletin 47, 175 (1963)]. The 1961 values for the atomic weights, based on the C–12 nuclide, were used [Cf. "Report of the International Commission on Atomic Weights" (1961) by A. E. Cameron and E. Wichers, J. Am. Chem. Soc. 84, 4175 (1962)].

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 hr is less than 0.01 percent and for 100 hr 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 hr at 120 °C shows about 0.01 percent loss in weight and about 0.02 percent loss after heating at 120 °C for 120 hr.

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 [Cf. "Stability of Aqueous Solutions of Acid Potassium Phthalate" by James I. Hoffman, J. Res. NBS 15, 583 (1935)].

The acid potassium phthalate was obtained from J. T. Baker Company of Phillipsburg, New Jersey. All testing and evaluation of this material was carried out in the NBS Institute for Materials Research. The homogeneity testing was performed by Hannah R. Hetzer of the Electrochemical Analysis Section. The coulometric evaluation of acidity was performed by Maya Paabo of the Electrochemical Analysis Section and George Marinenko of the Microchemical Analysis Section.

WASHINGTON, D. C. 20234
September 27, 1966

W. WAYNE MEINKE, Chief
Office of Standard Reference Materials

(Over)

Direction 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 hr 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 *p*H 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 *p*H 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 (*p*H 8.6) prepared by mixing 25 ml of an M/5 H_3BO_3, M/5 KCl solution with 6 ml of M/5 NaOH, 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, 204.229 g of acid potassium phthalate is equivalent to 1.00797 g of hydrogen and 1.02114 g is equivalent to 50 ml of 0.1 *N* solution.