

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

PROVISIONAL CERTIFICATE OF ANALYSIS

of

STANDARD SAMPLE 84c

ACID POTASSIUM PHTHALATE ($\text{KC}_8\text{H}_4\text{O}_4$)
(Acidimetric Standard)

(Analysis by H. B. Knowles)

This lot of acid potassium phthalate was prepared to insure material of high purity and uniformity. It meets the American Chemical Society's specification for analytical reagent grade material, but should not be considered as entirely free from impurities such as traces of occluded water, free phthalic acid, chlorides, sulfur compounds and heavy metals. On the basis of titration a purity of 100.05 percent was indicated when the material was assayed as indicated in the section headed "Effective neutralizing power."

DRYING.-- The sample as issued contains a small amount of entrapped water (approximately 0.03 percent) which is removed rather slowly when the crystals are dried at 120° C. The loss in weight is less than 0.01 percent when the uncrushed crystals are dried at 120° C for 2 hours and about 0.027 percent for 240 hours.

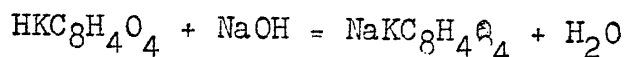
When the crystals are lightly crushed to a fineness of approximately 100 mesh most, if not all, of the entrapped water is lost during crushing. For example, the crushed material when dried for 2 hours at 120° C shows less than 0.01 percent loss in weight and no further loss after heating at 120° C for 120 hours.

EFFECTIVE NEUTRALIZING POWER.-- When the crushed sample was dried for 2 hours at 120° C and 1-g portions of the dried sample were dissolved in water and titrated at about 27° C with a standard solution of 0.1 N sodium hydroxide, a neutralizing power equivalent to that of an acid potassium phthalate of 100.05 percent was indicated. The end point was taken at pH 8.6 with a glass electrode. The alkali solution was standardized with a 0.1-N solution of hydrochloric acid prepared from redistilled acid, and standardized by precipitating silver chloride, drying the latter at 165° C, and applying the slight correction for entrained moisture. Weight burettes were used, and the weights of silver chloride and phthalate were corrected to the vacuum standard. All calculations were based on the 1941 International Table of Atomic Weights.

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.-- 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. 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/5H₃BO₃, 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:



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.

(Signed) LYMAN J. BEIGGS, Director.
GEFL

August 10, 1945.
Washington, D. C.