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FURTHER WORK ON ACID POTASSIUM PHTHALATE AS A STANDARD IN VOLUMETRIC ANALYSIS.

W. S. HENDRIXSON.

Nearly three years ago the writer showed that acid potassium and acid sodium phthalates may be used with advantage as standards in volumetric analysis.¹ They can be easily prepared in a state of great purity, they have large molecular weights, and the acid potassium salt has the distinct advantage of absence of water of crystallization. So little work on these salts has been done and so limited is the knowledge of them that it was thought desirable to make further study of the properties and value as a standard of at any rate the potassium salt. The particular objects in view were:

1. To determine the practicability of the preparation, and use of the salt as a standard, by other hands.

2. To determine the extent of the work of purification of the salt for ordinary use, and the yield on the basis of the phthalic anhydride used.

3. To study the hygroscopic character of the salt, the necessary means to dry it, and its stability at higher temperatures.

The laboratory work represented in this paper was done under the writer's direction by Mr. Sereno G. Norton, an undergraduate in Grinnell College, and to his skill and industry is due very largely whatever merit the paper may have.

Acid potassium phthalate was prepared by the method described in the paper already cited. Several preparations were made and titrated as described in the former paper. There is one chance of error which should be mentioned, since the error might easily be made by comparatively inexperienced hands. Phthalic anhydride is very sparingly soluble in water, even in hot water. If not quite enough potassium carbonate is used in neutralizing the anhydride a portion of the latter will be left undissolved, and may be easily overlooked since the sparingly soluble acid salt is likely to form a layer of crystals over the surface of the hot solution. Since the neutral potassium phthalate is extremely soluble even in cold water there is no danger of its contaminating the acid salt. Therefore a slight excess of potassium carbonate should be used, and of course the hot solution should be filtered in the first instance at any rate.

A large volume of standard hydrochloric acid was made by the method of Hulett and Bonner so as to contain .003647 gram of HCl to 1 c.c. This acid was further titrated with benzoic acid specially purified for such purposes by the Bureau of Standards, which gave .003648 gram HCl to 1 c.c. This concentration was confirmed by means of silver. A solution of sodium hydroxide was prepared as described in the first paper and was used as the intermediary between the acid potassium phthalate and the hydrochloric acid. The indicator was phenolphthalein, and as already pointed out its error in marking the neutral point was cancelled by the method of titration.

Titrations were made of samples of the acid phthalate which had been three, four and five times re-crystallized from hot water. The objects were to determine the number of crystallizations necessary to secure a sufficiently pure product, and to determine the constancy of the acid salt as an individual compound. It is well known that successive crystallization of some so-called acid salts does not secure a constant product. The results are shown in the following table: in which "Third," "Fourth" and "Fifth" show the number of re-crystallizations of the potassium acid phthalate, and following each the values given for the standard hydrochloric acid.

	THIRD	FOURTH	FIFTH
	.0036489	.0036470	.0036519
	.0036473	.0036475	.0036455
	.0036484	.0036449	.0036448
	.0036480	.0036450	.0036410
	.0036471		
	.0036475		
	.0036478		
	.0036463		
	.0036463		
	.0036455		
Average0036473	.0036461	.0036458

The results seem to show that there is nothing to be gained beyond three re-crystallizations. Other titrations seem to show that even two re-crystallizations, that is three crystallizations in all will give a salt pure enough for ordinary purposes. There seems to be no ground to doubt the constancy of the composition of the acid salt.

Working with no special care our results showed that about 50 per cent of the theoretical yield of the acid salt may be

obtained after four crystallizations from hot water. In normal times pure sublimed phthalic anhydride and pure potassium carbonate are cheap, and this standard is, therefore, one of the most cheaply and easily obtained.

We next directed our attention to the hygroscopic character of acid potassium phthalate. It is very desirable that a substance used as a standard should have the minimum of hygroscopicity. To test this character we carried out two sets of experiments. The first consisted in heating the air dried salt in an electric oven at different temperatures and determining the losses; the second in exposing the salt thus dried in air of known moisture content and determining the amount of moisture taken up from the air.

In carrying out the first series of experiments about twenty-one grams of the salt was heated in the electric oven in a platinum dish. At the end of each period the loss was determined and a portion of the salt was taken out without loss and titrated. The losses and titrations would thus form checks on each other. There were eleven such time periods with their corresponding losses and titrations. Since only four different temperatures were used I have reduced the results to this number.

Weight of Salt	Time	Temp.	Loss	Value of HCl
21.5940	4 hrs.	110	.0006	.0036471
20.4966	2 hrs.	110	.0024	.0036469
16.0100	8 hrs.	130	.0034	.0036490
11.6910	8 hrs.	150	.0006	.0036473

This is a very remarkable showing. We all know the difficulty of securing accurate weights of substances owing to their hygroscopic character or that of the containing vessels. Here an air dried substance weighing twenty-one grams and in a platinum dish lost no more on heating at 110° C. than could be accounted for on the basis of change in temperature in the balance case and the hygroscopic character of the dish itself. As stated there were eleven determinations. The total loss of weight was about 1 part in 3,000, which is within the limits of accuracy of volumetric analysis in any case. The loss was comparatively constant and apparently independent of the temperature and time of heating. It was probably due in part to a constant error in the method. This consisted in letting the dish and contents stand in the air of the room while taking the sample for titration and weighing it. The dish was then weighed for the next heating.

On the other hand the loss on heating was determined by weighing the dish as soon as taken from the desiccator.

To throw more light upon this low hygroscopic value about seven grams of the finely divided salt which had been dried in the above series of experiments was exposed in a desiccator over sulfuric acid of such concentration as to give a humidity of the enclosed air equal to 70 per cent, which is not likely to be exceeded in the ordinary laboratory. It was left over the acid forty-seven days and about every four days a weighing was made. There were small variations as always, due to changes of temperature and the like, the largest being two milligrams. Summing up the minus and plus differences they show a net loss of one-tenth milligram in the forty-seven days. These two sets of experiments seem to justify the conclusion that the hygroscopicity of the acid potassium phthalate is practically nil, and in using it and weighing it attention need be directed only to the containing vessel. By use of an open platinum crucible this source of error may be almost completely avoided.

It seemed desirable to know the solubility of acid potassium phthalate in water, and this was determined at three different temperatures. At 25° and 35° the method used was that recently described by the writer in these Proceedings.² For the solubility at the boiling point the method of Pawlewski was used.³ The following are the results:

	Wt. Solution	Wt. Acid Phthalate	Per Cent in Sol.
At 25 degrees	21.025	2.1531	10.23
	21.028	2.1553	10.25
At 35 degrees	21.198	2.6859	12.67
At Boiling Point	9.18	3.3158	36.12

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²Proc. Iowa Acad. Sci., Vol. XXII, pp. 217-224, 1915.

³Berichte d. Deutsch. Chem. Gesel., Vol. 32, p. 1040.