Sodium perchlorate and phosphoric acid are soluble in alcohol and do not cause an error in the determination of potassium.

The sulfate and ammonium ions were found to produce an error, but the error caused by the sulfate was not equivalent to all the sulfate ion that was present. It is necessary to remove all of the ammonium salts and most of the sulfates.

Summary.

The method of determining potassium as the perchlorate is accurate.

Sulfate and ammonium ions produce an error and must be removed. The sulfate is precipitated with barium chloride in a strongly acid solution avoiding a large excess. The ammonium salts are expelled by heating.

The phosphate ion does not produce an error and need not be removed.

The solution containing chlorides should be evaporated to dryness with a slight excess of perchloric acid, without stirring.

The precipitate should be washed only with alcohol containing 0.2% perchloric acid.

The time of making an analysis is short, the manipulations are simple, and the cost is almost negligible.

The method can be recommended for use in water analyses and in other analytical work where the content of potassium is desired.

ILLINOIS STATE WATER SURVEY,
UNIVERSITY OF ILLINOIS,
URBANA, ILL.

A NEW METHOD FOR THE PRECISE STANDARDIZATION OF HYDROCHLORIC ACID SOLUTIONS.

BY LAUNCELOT W. ANDREWS.

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When the greatest precision is desired in standardizing a volumetric solution, those methods should be avoided, which: (1) involve transferring or washing a precipitate; (2) depend upon the peculiarities of any particular indicator; (3) require the use of standard substances containing water of crystallization, or those that may contain impurities difficult to detect, *i. e.*, almost all organic compounds, or that can not be positively dried without danger of decomposition; (4) demand any peculiarities of technique or any exercise of personal judgment that may make it difficult for different observers to obtain nearly identical results.

With these criteria in mind, I have devised and for several years used in practice a method, the accuracy of which is only limited by the unavoidable errors of weighing. It depends upon the loss of weight caused by the replacement of NO_3 in silver nitrate by Cl. Since hydrochloric acid solutions kept in glass vessels, always contain traces of chlorides and of other non-volatile impurities derived from the glass, the process must be so conducted as to avoid error from this source. The method is carried out as follows, when the solution to be standardized is 0.2 *N:*

Select two porcelain, or better, silica dishes, of approximately like size, form, and weight, of 75 to 100 cc. capacity, each being provided with a light watch glass or silica cover, and one of them with a silica or glass rod, short enough to lie under the cover. The dish without a rod will be referred to as the "companion dish." In the former, place 1.9 to 2.0 g. of the purest silver nitrate. The absence of ammonium nitrate should be assured. Both dishes are put into an oven at 160°, the temperature raised to 240° and kept there till the weight is constant. At this temperature no water is retained. After cooling in a desiccator, weigh both dishes, covered. Leaving both covers in the balance case, remove the dishes.

By means of a pipet, measure 50 cc. of 0.2 *N* hydrochloric acid, to be standardized, into the dish with the silver nitrate, using, of course, every precaution to make the measurement as accurate as possible, and noting the temperature of the solution. At the same time, measure 50 cc. of the same acid into the companion dish.

Stir up the silver nitrate until all has dissolved and the silver chloride has clotted together, but do not remove the rod from the dish. Place both dishes in the steam bath at 95–100°, to evaporate the water without spattering, and finally dry at the temperature at which the silver nitrate was dried. Cool in the desiccator and weigh. The increase in the weight of the companion dish is assumed to represent the weight of non-volatile impurities contained in the acid, and its amount is subtracted, as a correction, from the observed weight of the silver chloride. This arrangement compensates for changes in the apparent weights due to atmospheric changes, so that, in case of very pure hydrochloric acid, the change of weight in the companion dish may be negative, a circumstance that need cause no apprehension.

By drying the silver nitrate, and, later, nitrate plus chloride, at the temperature of incipient fusion of the former, the same results are obtained as at 200° or 240°.

The normality of the solution is given by the expression:

$$\mathrm{N} = \frac{\mathrm{W} - \mathrm{W}_1 + w_1 - w}{0.02655\,\mathrm{V}}$$

in which,

V = Corrected volume of solution.
W = Weight in air of $AgNO_3$ + dish.
W_1 = Weight in air of $AgCl + AgNO_3$ + dish.
w = Weight of companion dish before.
w_1 = Weight of companion dish after.

In testing experimentally the precision of this standardizing method, it was esteemed better to eliminate errors due to volumetric measurement of the solution, and to weigh the latter instead. Agreement of the results among one another will show whether any chlorine escapes or whether any other variable sources of error lie in the chemical reaction itself. The results of four such determinations follow. The weight of dish, cover and rod was less than 70 g.

Exp. A. Dish + $AgNO_3$ minus weight after = 0.5311 g. $w_1 - w = 0.0001$. Wt. sol. 50.0445 g.

Exp. B. Dish + $AgNO_3$ minus weight after = 0.5316 g. $w_1 - w = 0.0004$. Wt. sol. 50.0548 g.

Exp. C. Dish + $AgNO_3$ minus weight after = 0.53105 g. $w_1 - w = 0.0001$. Wt. sol. 50.0391 g.

Exp. D. Dish + $AgNO_3$ minus weight after = 0.5310 g. $w_1 - w = 0.00005$. Wt. sol. 50.0396 g.

Hence, the loss (corrected by companion dish) is respectively, per 50 g. of solution taken: in A 0.53063, in B 0.53062, in C 0.53095, in D 0.53054. Since the balance was sensitive to only 0.05 mg. it is evident that errors of weighing alone account fully for the surprisingly small variations observed. It is, further, clear that these errors may be diminished if there were any object in so doing, by operating with normal instead of 0.2 *N* solutions. The disadvantage that the loss of weight is a smaller quantity than the absolute weight of the HCl determined, so that all errors are multiplied by the factor 1.37, is made unimportant by the great accuracy of the process in itself. In spite of its precision, the method is not time-consuming, as the evaporations require no oversight. The time spent is, practically, only that required by the weighings. Most of this work was done in the laboratory of the Andrews Chemical Works, Davenport, Iowa.

WASHINGTON, D. C.

CORRECTION.

Correction for article on "The Potential of Silver in Nonaqueous Solutions of Silver Nitrate," by Vernette L. Gibbons and F. H. Getman, which appeared in the August number of THIS JOURNAL, page 1645: The last five values in Table X should have been headed Table XI.—Solutions in Pyridine.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF WASHINGTON.]

BENZOYLATIONS IN ETHER SOLUTION.

BY WILLIAM M. DEHN AND ALICE A. BALL.

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In a former paper[1] it was shown that acetyl chloride reacts in anhydrous ether solutions with primary, secondary and tertiary bases. The initial products were invariably additive products and these, by simple splitting or by hydrolysis, yielded the ordinary acetylated products.

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[1] THIS JOURNAL, 34, 1399 (1912).