Samples 1 and 2, however, were subjected to a further distillation under atmospheric pressure and formed preparation B_a. Sample 5 was treated in as nearly the same manner as was possible at different times and formed preparation B_b.

The results obtained in this series seem to indicate that tantalum chloride either occludes some chlorine which may be removed only with the greatest care or that it is partially decomposed by distillation in dry air. The former would tend to give a low value for the atomic weight while the latter would undoubtedly give a value too high.

Summary.

During the present study of the ratios $2TaCl_5 : Ta_2O_5$ and $TaCl_5 : 5Ag$, the following observations were made:

1. Tantalum oxide, prepared from the chloride, not only occludes the nitric acid but is lost on ignition.
2. Silver forms with tantalic acid an insoluble compound, probably a tantalate.
3. Hydrochloric acid cannot be removed completely from tantalic acid by boiling with sulfuric acid either under atmospheric or reduced pressure.
4. Silver can be used to determine hydrochloric acid in the presence of tantalum fluoride if the precipitation is carried out in an approximately 5.5% solution of hydrofluoric acid. Three to four days, however, are necessary for equilibrium to become established.
5. Tantalum chloride, purified by replacing the chlorine atmosphere with dry air and by distilling either under atmospheric or reduced pressure, apparently varies slightly in composition.

In conclusion it may be pointed out that there is now available for the first time a method of analysis sufficiently accurate to detect slight differences in the composition of tantalum chloride. It now remains to make a careful study of the preparation of the chloride with regard to the definiteness of its composition. This problem is now under investigation and as soon as a satisfactory solution is obtained a final series of determinations will be published. It may be added that the possible value of this method of analysis for the determination of the atomic weights of other elements will be investigated.

URBANA, ILL.

[CONTRIBUTION FROM THE UNITED STATES GEOLOGICAL SURVEY.]

SOLUBILITY OF MIXTURES OF SODIUM AND POTASSIUM CHLORIDES IN SOLUTIONS OF HYDROCHLORIC ACID.[1]

BY W. B. HICKS.

Received February 3, 1915.

Some experiments conducted by Dr. R. C. Wells of the Geological Survey on the brines from Great Salt Lake indicated that potassium

---

[1] Published by permission of the Director of the U. S. Geological Survey.

chloride is more soluble in hydrochloric acid solutions than is sodium chloride, and led to the suggestion that the constant boiling point acid might serve as a useful reagent in the commercial separation and purification of these two salts. In order to investigate the possibilities in this direction, it was decided to determine the solubilities of mixtures of sodium and potassium chlorides in several different concentrations of hydrochloric acid, including that of the constant boiling point acid. For this purpose acid solutions of approximatley 8, 17, 20 and 32% were prepared, the exact strength being determined afterwards on the saturated solution of the mixed salts by titration with standard sodium hydroxide solution. While the results, as was to be expected, soon showed that the solubility of these salts in strong acid is too small to admit of the reagent being used commercially under ordinary circumstances, the experiments were not without interest, in that an increase in the potassium-sodium ratio went hand in hand with that of the concentration of the acid.

The apparatus used in all the solubility determinations was in principle similar to that described by C. M. van Deventer[1] and later used by H. Goldschmidt[2] and others, and is accurately represented in the following diagram.

The glass cylinder *a* is 20 cm. long and 3.8 cm. in diameter and carries two one-hole rubber stoppers *c* and *d*. The tube *e* is used for stirring and carries the pulley *f* by which it is rotated. The enlargement at *g* contains two small holes, *h* and *i*, for the purpose of increasing the efficiency of the stirrer. The hollow wooden cylinder *j* is held in place by the rubber stopper *c*, supports the pulley *f*, and serves as a bearing in which the stirrer *e* rotates. The glass rod *k* carries the rubber stopper *l* which closes the filtering tube *m*. The platinum cone *n* supports the asbestos filter *o*. The syphon *p* is sealed to *m*, and extends through the rubber stopper *q* into the 50 cc. flask *r*. The small tube *s* serves as an outlet for *r*.

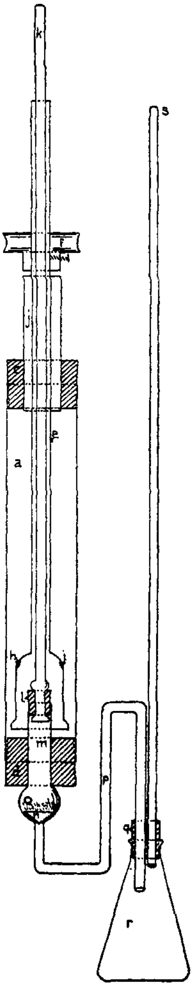

Fig. 1.

In conducting the experiments about 15 g. of pure potassium chloride and an equal quantity of sodium chloride were placed in the glass container *a* of the apparatus described above, and then 40 cc. of hydrochloric acid

---

[1] *Z. physik. Chem.*, 5, 559 (1890).

[2] *Ibid.*, 17, 153 (1895).

approximately of the strength desired were added. The whole apparatus was placed in an electrically controlled thermostat to such a depth that the water reached to within about one inch of the top of the container *a*, and was clamped in place. The thermostat fluctuated less than 0.05° from 25°. The mixture was stirred energetically for 4 hours, when the solution was considered to be saturated, preliminary experiments having shown that ordinarily equilibrium is attained in a much shorter time under such conditions. After allowing the undissolved salts to settle, the rubber stopper was removed from the filter tube by means of the glass rod *k*, and the saturated solution filtered at the thermostat temperature through the asbestos mat into the weighed flask *r*. Suction was applied at *s* to hasten the filtering operation. The whole apparatus was then removed from the thermostat, the flask washed with water at 25°, dried, removed from the apparatus and weighed.

The weighed solution, amounting to about 30 g., was made to definite volume and aliquots taken for analysis. In most cases the determinations were carried out in duplicate. The acidity was determined by titration with standard sodium hydroxide solution, using methyl orange as indicator. The potassium was determined by precipitating as potassium chloroplatinate and weighing the reduced platinum.[1] The total dissolved salts were estimated by evaporating a definite mount of the solution in platinum and weighing the residue after gentle ignition and cooling in a desiccator. The values for sodium were obtained by difference.

TABLE I.—SOLUBILITY OF MIXTURES OF SODIUM AND POTASSIUM CHLORIDES IN HYDROCHLORIC ACID SOLUTIONS AT 25°.

No.	% HCl.	% NaCl.	% KCl.	% NaCl + KCl.	% Na.	% K.	Ratio K/Na.
1..........	0	19.95	10.90	30.85	7.85	5.72	0.73
2..........	8.61	10.65	7.58	18.23	4.19	3.98	0.95
3..........	17.16	3.56	3.80	7.36	1.40	1.99	1.42
4..........	20.65	2.03	2.86	4.89	0.80	1.50	1.87
5..........	32.78	0.18	1.27	1.45	0.07	0.67	9.57

The results of the solubility determinations are given in Table I. In order to make the data more complete, the values of Precht and Wittjen[2] for the solubility of mixtures of sodium and potassium chlorides in pure water have been included, the figures for 25° having been obtained by interpolation. The results have also been plotted showing graphically the changes in solubility of these salts with variations in the concentration of the acid. This diagram is given in Fig. 2.

The rapid depression in the solubility of sodium and potassium chlorides with the increase in the concentration of the acid is very striking and is

---

[1] Hicks, W. B., "A rapid modified chloroplatinate method for the estimation of potassium," *J. Ind. Eng. Chem.*, 5, 650 (1913).

[2] *Ber.*, 14, 1667 (1881).

of the same order as that shown by the individual salts in solutions of hydrochloric acid.[1] It is much more pronounced for sodium chloride than for the potassium salt, so that the curves representing these two solubilities intersect at a point corresponding to 16.2% of hydrochloric acid. Here the solubilities of the two salts are equal, amounting to 4.4%. As the acid solution becomes still more concentrated the saturated mixture contains an increasingly larger proportion of potassium than of sodium chloride. However, in such strong acid solutions the solubility of both salts is very small, amounting to only 2.9% of potassium chloride and 2.2% of sodium chloride in the constant boiling point (20.3%) acid. Apparently this precludes the consideration of hydrochloric acid as a reagent in the commercial separation of the two salts in question.

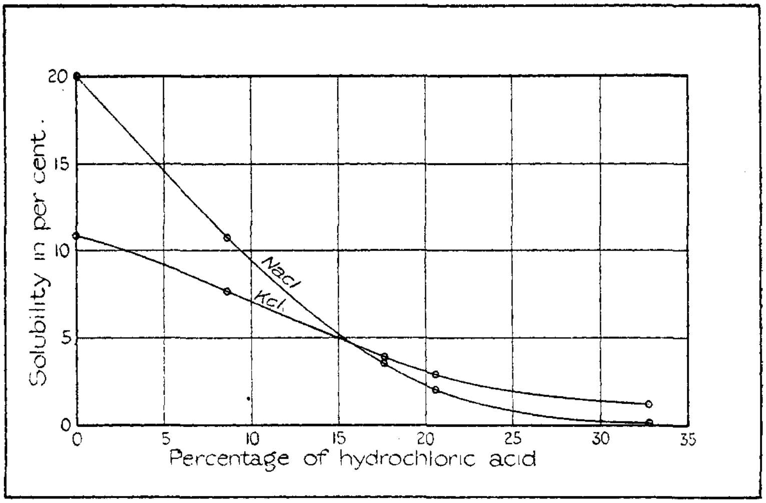

Fig. 2.

WASHINGTON, D. C.

AMMONOBASIC ALUMINIUM IODIDES.

By Edward C. Franklin.

Received January 15, 1915.

Introduction.

Some years ago the observation was made that very considerable quantities of potassium amide may be added to liquid ammonia solutions of aluminium iodide without causing the formation of a permanent precipitate. With further additions of the alkaline amide a precipitate is formed which then dissolves again in an excess of potassium amide.[2]

In the light of the many demonstrated analogies between the reactions

---

[1] Engel, *Ann. de Chim. Phys.*, [6] 13, 374 (1888).

[2] This Journal, 27, 849 (1905).