

3. The interpretation placed upon their results by Hantzsch and Kalb appears to be erroneous.

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[CONTRIBUTION FROM CHEMICAL LABORATORIES OF NEW YORK UNIVERSITY]

TERNARY SYSTEMS. XI. MAGNESIUM IODATE, SODIUM IODATE AND WATER. XII. SODIUM IODATE, POTASSIUM IODATE AND WATER. XIII. POTASSIUM IODATE, POTASSIUM CHLORIDE AND WATER. XIV. POTASSIUM IODATE, POTASSIUM SULFATE AND WATER¹

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RECEIVED AUGUST 1, 1931

PUBLISHED DECEMBER 10, 1931

In continuation of the study of three-component systems involving salts found in the Caliche of Chile, the following four systems have been investigated at 5, 25 and 50°. In addition to the solubility data there found, a brief discussion will be given of two points bearing upon all of the systems.

Anomalous Solubility Equilibria for Sodium Iodate and Potassium Iodate.—In an earlier paper² it was pointed out that satisfactory agreement for solubility at 25° approached from undersaturation and supersaturation could not be attained for sodium iodate even after long periods of agitation, even when care had been taken to insure that the salt had been completely hydrated before the solubility experiments were begun. Not only did the results from supersaturation and undersaturation fail to reach the same figure, but irregularities frequently were found of a magnitude many times that of the probable analytical error. It is to be added that potassium iodate, used in the present investigation, has also shown irregularities in solubility data, though of a much lesser amount. For this disturbing behavior of the two salts we have been unable to find an explanation in our own work. A publication from McBain and Kistler,³ appearing at nearly the same time as our publication upon sodium iodate, presents information which may contain the explanation for the behavior of these iodates. Subjecting a 0.33 *N* aqueous solution of potassium iodate to ultra-filtration under pressure through cellophane membranes, they found an increase in concentration in the unfiltered residue amounting to 12 to 16%, from which they feel justified in drawing the "remarkable conclusion that aqueous potassium iodate contains colloidal constituents." The presence of colloidal constituents in the solubility equilibrium would

¹ The material of this paper was presented by John E. Ricci in partial fulfillment of the requirements for the degree of Doctor of Philosophy at New York University.

² Hill and Donovan, *THIS JOURNAL*, **53**, 935 (1931).

³ McBain and Kistler, *J. Phys. Chem.*, **35**, 130 (1931).

naturally lead to irregularities in the total concentrations in samples removed by pipet as solution and analyzed as such, since the amount of colloidal material might vary with size of crystals, rapidity of agitation, etc. If this is the true explanation of the irregularity in behavior which we have noticed, it follows that solutions of sodium iodate, not tested by McBain and Kistler, should also show colloidal constituents, and that the phenomenon should be more pronounced than with the potassium salt, since the irregularities in solubility data are greater in the former case. In our solubility isotherms we have of course been unable to control this factor; the experiments have been run from one to two weeks in each case in the hope of coming as close as possible to a true equilibrium condition, and it may well be that the presence of a third component reduces this colloidal tendency. While the three-component experiments show in most cases a very satisfactory regularity, as tested by the method given in the next section, it may be doubted whether the true solubility of the two alkali iodates in water is correctly known within 1% relative to themselves, which is of the order of 0.1% as referred to the saturated solutions, except at 25°, where long periods of stirring have given agreement within 0.5%.

The Concordance of Three-Component Solubility Data as Tested by the Method of Algebraic Extrapolation of Tie-Lines.—The use of the tie-line in three-component experimentation as a means of determining the solid phase is a common practice. It follows from purely geometrical principles that if a straight line be drawn from the composition of the liquid phase through the composition of the original complex of materials taken, or through the composition of a wet residue as determined by direct analysis, its extrapolation must pass through the composition of the pure solid phase;⁴ by intersection of several tie-lines or by intersection of one of them with a side of the triangle the composition of the solid becomes known. The method has been very widely and successfully used, in cases where it is not conveniently possible to obtain the solid phase in condition of sufficient purity for analysis. Two adaptations however which seem not to have obtained general notice are the method of algebraic extrapolation and the use of algebraic extrapolation in testing concordance of results. The accuracy of graphic extrapolation is of course dependent upon the scale of the graph, and with the conventional size of graph paper on which the triangle has sides of eight inches, errors of considerable magnitude may be made. This purely mathematical error may be entirely avoided by the use of a simple proportion between the differences in the amounts of two components at the two known points, and the differences in the amounts of the same components at one of those points and at the composition of the solid phase. For example, if a complex consisting of

⁴ Schreinemakers, *Z. physik. Chem.*, **11**, 81 (1893); Bancroft, *J. Phys. Chem.*, **6**, 178 (1902).

A, B and water, and containing 80% water and 6.5% of component A, gives a solution containing 90% water and 7% of A, we can determine the amount of A in the solid phase (assuming it anhydrous) by the proportion 0.5:10:: x :90, where the first two terms represent differences between solution and original complex in A content and in water, and the latter two the differences between solution and solid phase with respect to the same components; $x = 4.5\%$, the solid containing therefore 2.5% A (7.0%–4.5%) and 97.5% of B. The proportion may of course be applied equally well on the assumption of a given amount of water in the solid phase corresponding to a hydrate. Its accuracy of course is wholly dependent upon the exactness with which the solution has been analyzed and the exactness with which the second fixed point on the line is known; for this second point, we have had greater success by using the synthetic composition of the whole system, as made up by weight of dried salts and water, rather than by analysis of a wet sample of solid and solution removed from the vessels after equilibrium has been attained; we believe the latter likely to bring in considerable errors due to evaporation or deliquescence during its transfer. It is true of course that the latter point is further removed from the composition of the solution and that therefore errors are not multiplied in the algebraic proportion to as great an extent as in the former case; but we are convinced that the high accuracy with which a complex of say 40 g. can be weighed *in situ* in the solubility tube is such that its error may be neglected, and all the error referred to the end-points on the straight line. We have therefore for some time used this method and believe it to afford an excellent test for concordance which has hitherto not generally been applied; by the variation of the extrapolated point from the composition of a definite compound, we can estimate the extent to which there is error in the analysis of the solution, or the extent to which the solid phase has failed to reach homogeneity. In the selection of such quantities of materials as will permit rapid settling of the solid and convenient filtration into the measuring pipet, we have usually arranged that the equilibrium solid phase shall be about one-tenth of the total system; it follows therefore from the proportion that errors in the analysis of the solution are multiplied by ten in the extrapolations, which permits a very rigid test of their accuracy. As will be seen in the following sections of the paper, systems in which the analytical control is good will give extrapolations to the solid phase which vary in the neighborhood of 1% only from the stoichiometric point; where the analysis is less dependable, the fact becomes patent. Should there be systematic variation from the solid phase, we should of course deduce the presence of solid solution or that the solid phase had not yet reached homogeneity. Such tests have given us much aid in determining the general concordance of the points on a solubility curve and also the time necessary to reach homogeneity in the solid phase; it should be pointed out, however.

that the test is purely mathematical in its implications and does not in any sense indicate whether or not the solution is in equilibrium with the solid phase.

XI. MAGNESIUM IODATE, SODIUM IODATE AND WATER

The slowness with which sodium iodate is hydrated, referred to in a previous publication,² and its anomalous behavior in the attainment of internal equilibrium discussed above, were factors of difficulty in the study of this system which were added to by the difficulty in finding an analytical method which is both accurate and reasonably quick. Several methods were carefully tested and discarded for the one reason or the other, including the direct determination of magnesium by precipitation as phosphate and weighing as pyrophosphate, which was found time-consuming because of the double precipitation found necessary; also its accuracy fell off when the magnesium content became low. As most convenient for our purpose, though not of the highest accuracy, was found the volumetric method of Kolthoff.⁵ A sample of solution to be analyzed, from 5 to 20 cc. depending on the magnesium content, was withdrawn by pipet and weighed in a 100-cc. volumetric flask. To this was added from a buret about 75 cc. of 0.13 *N* sodium hydroxide and the solution brought up to the mark after shaking. After standing for four hours the precipitate of Mg(OH)₂ settled well and permitted the withdrawal of an aliquot sample of supernatant liquid, which was titrated back with standard hydrochloric acid, using methyl orange as indicator. To reduce the errors introduced by adsorption, the sodium hydroxide was standardized by a similar method against a solution of known magnesium content. From the titration, the content of magnesium iodate became known; by a titration for total iodate by the iodimetric method, the content of sodium iodate could be calculated, and the total composition of the solution was thereby obtained. Tests upon twenty-four sets of duplicates showed that the average relative disagreement between duplicates was 0.5%.

The complexes used for the ternary system were made up from weighed amounts of water, dried sodium iodate and the hydrate Mg(IO₃)₂·4H₂O, which has previously been shown⁶ to possess a constancy of composition quite remarkable for a hydrate. For the 5° isotherm, the solids were first dissolved by heating and the solution inoculated, after cooling, with the expected solid phase. The methods of stirring, thermostatic control, etc., were as given in earlier papers of this series.^{2,6} The time of stirring varied from four days to as much as forty days, as was found necessary in individual cases for attainment of equilibrium by analysis of samples from time to time; in the majority of cases, the time used was about

⁵ Kolthoff, *Z. anorg. Chem.*, **100**, 143 (1917); **112**, 172 (1920).

⁶ Hill and Moskowitz, *THIS JOURNAL*, **53**, 941 (1931).

fourteen days. The densities are calculated from the weight delivered by a volumetric pipet.

TABLE I
Mg(IO₃)₂-NaIO₃-H₂O

Temp., °C.	Original complex		Saturated solution			Solid phase
	Wt. % Mg(IO ₃) ₂	Wt. % NaIO ₃	Wt. % Mg(IO ₃) ₂	Wt. % NaIO ₃	Density	
5	...	0.00	4.37	0.00	1.038	Mg(IO ₃) ₂ ·10H ₂ O
	11.06	1.263	3.74	1.45	1.047	Mg(IO ₃) ₂ ·10H ₂ O
	3.33	2.53	1.055	Mg(IO ₃) ₂ ·10H ₂ O + NaIO ₃ ·5H ₂ O
	2.01	7.98	2.19	2.68	1.043	NaIO ₃ ·5H ₂ O
	0.00	...	0.00	3.28	1.028	NaIO ₃ ·5H ₂ O
25	...	0.00	8.54	0.00	1.078	Mg(IO ₃) ₂ ·4H ₂ O
	15.07	2.49	7.24	2.80	1.091	Mg(IO ₃) ₂ ·4H ₂ O
	13.87	5.38	6.05	6.16	1.113	Mg(IO ₃) ₂ ·4H ₂ O
	5.86	7.49	1.125	Mg(IO ₃) ₂ ·4H ₂ O + NaIO ₃ ·H ₂ O
	13.56	6.77	5.73	7.66	1.128	Mg(IO ₃) ₂ ·4H ₂ O (m) ^a
	5.57	8.79	1.139	Mg(IO ₃) ₂ ·4H ₂ O + NaIO ₃ (m)
	5.00	13.90	5.42	8.74	1.134	NaIO ₃ (m)
	5.00	13.90	5.40	7.52	1.121	NaIO ₃ ·H ₂ O
	4.97	13.90	5.33	7.50	1.122	NaIO ₃ ·H ₂ O
	4.54	14.15	4.76	7.77	1.119	NaIO ₃ ·H ₂ O
	2.80	14.04	2.99	7.94	1.102	NaIO ₃ ·H ₂ O
	1.09	13.93	1.18	8.30	1.081	NaIO ₃ ·H ₂ O
0.00	...	0.00	8.57	1.076	NaIO ₃ ·H ₂ O	
50	...	0.00	11.97	Mg(IO ₃) ₂ ·4H ₂ O
	26.01	3.54	9.95	4.59	...	Mg(IO ₃) ₂ ·4H ₂ O
	25.53	6.78	8.41	8.94	...	Mg(IO ₃) ₂ ·4H ₂ O
	7.67	11.97	...	Mg(IO ₃) ₂ ·4H ₂ O + NaIO ₃ ·H ₂ O
	4.62	17.55	4.93	12.42	...	NaIO ₃ ·H ₂ O
	3.56	17.06	3.73	13.05	...	NaIO ₃ ·H ₂ O
	2.91	17.34	3.05	13.26	...	NaIO ₃ ·H ₂ O
	2.52	18.26	2.64	13.27	...	NaIO ₃ ·H ₂ O
	1.48	17.27	1.55	13.54	...	NaIO ₃ ·H ₂ O
	0.00	...	0.00	13.49	...	NaIO ₃ ·H ₂ O

^a m., metastable solid phase.

The composition of the solid phases, and the concordance of results, were tested by the algebraic extrapolation of tie-lines as discussed above. Notwithstanding the limited accuracy of the analytical results, the extrapolations for the eighteen cases where a single solid phase is assumed in the ternary solution gave an average deviation from the composition of the assumed solid amounting to only 0.72% and a maximum deviation of 2.18%. In the 25° isotherm, an isothermally invariant point for Mg(IO₃)₂·4H₂O and metastable anhydrous NaIO₃ was obtained which remained unchanged for thirteen days; upon being seeded with the hydrate, it changed slowly to the composition of the stable invariant point. A metastable point on the 25° curve for anhydrous NaIO₃ was also obtained, which

behaved in the same manner. In general, it was found that wherever the magnesium content was high, the unhydrated sodium iodate would not change spontaneously to the hydrate even after many days, inoculation with the hydrate being essential to initiate the change. Further, the attainment of external equilibrium with the liquid phase seemed to be slower at 50° than at the lower temperatures, giving a solubility curve at that temperature far less smooth than in the other cases. The various quintuple points which must occur between 5 and 50° , through changes in the degree of hydration of the solid phases, were not investigated by thermal means since the slowness of the changes indicated that such experiments would be futile. The curves for the three isotherms are given in condensed form in Fig. 1.

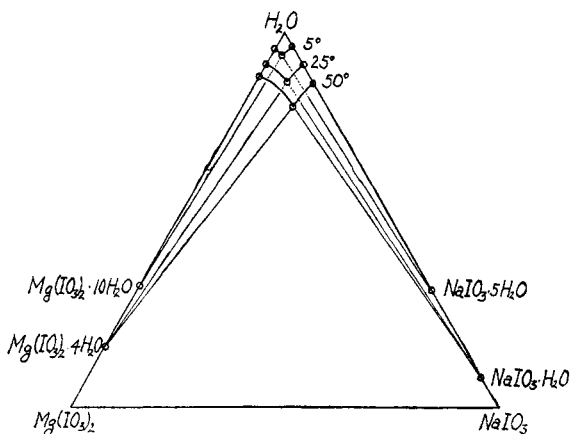


Fig. 1.— $\text{Mg}(\text{IO}_3)_2$ - NaIO_3 - H_2O .

XII. SODIUM IODATE, POTASSIUM IODATE AND WATER

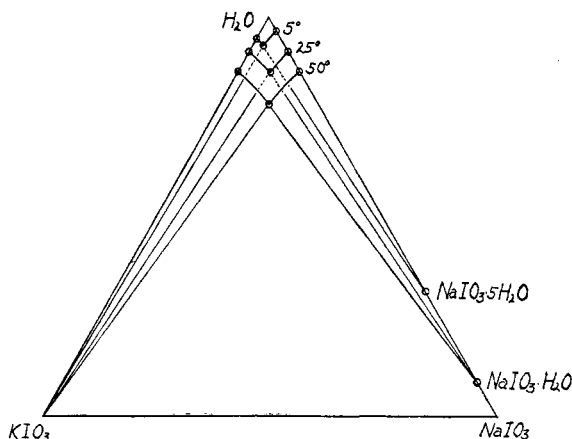
For this system, using the same procedure as before, the synthetic complexes were dissolved completely by raising the temperature, and were then seeded with the appropriate hydrate of sodium iodate after cooling to the temperature of the experiment. The time of rotation in the thermostat was about thirteen days, which time was shown by analysis of solution to be sufficient for attainment of external equilibrium. For the analysis, samples of filtered solution were evaporated to dryness at 110° , and other samples titrated for iodate by the method of iodimetry; although the errors of analysis are multiplied largely in the calculation, yet the results are shown below to be moderately concordant, and the method has the advantage of speed.

The algebraic extrapolation of the tie-lines indicates the lessened accuracy of the analytical method here used; the average deviation from the composition of the assumed solid phases is 1.45%, the maximum 3.5%.

TABLE II
 NaIO₃-KIO₃-H₂O

Temp., °C.	Original complex		Saturated solution		Density	Solid phase
	Wt. % NaIO ₃	Wt. % KIO ₃	Wt. % NaIO ₃	Wt. % KIO ₃		
5	0.00	...	0.00	5.16	1.043	KIO ₃
	1.24	15.03	1.41	4.71	1.051	KIO ₃
	2.17	4.72	1.060	KIO ₃ + NaIO ₃ ·5H ₂ O
	12.81	2.64	2.48	3.19	1.046	NaIO ₃ ·5H ₂ O
	...	0.00	3.28	0.00	1.028	NaIO ₃ ·5H ₂ O
25	0.00	...	0.00	8.45	1.071	KIO ₃
	3.55	20.09	4.26	7.09	1.098	KIO ₃
	7.13	6.73	1.126	KIO ₃ + NaIO ₃ ·H ₂ O
	14.90	3.52	7.79	3.79	1.103	NaIO ₃ ·H ₂ O
	...	0.00	8.57	0.00	1.074	NaIO ₃ ·H ₂ O
50	0.00	...	0.00	13.21	...	KIO ₃
	3.52	25.07	3.92	11.92	...	KIO ₃
	7.02	25.01	7.70	11.14	...	KIO ₃
	10.92	10.61	...	KIO ₃ + NaIO ₃ ·H ₂ O
	21.96	6.50	11.41	7.93	...	NaIO ₃ ·H ₂ O
	22.51	3.57	12.55	4.24	...	NaIO ₃ ·H ₂ O
...	0.00	13.49	0.00	...	NaIO ₃ ·H ₂ O	

This deviation is of course insufficient to raise any doubt as to identity of the solid phases themselves. The curves for the three isotherms are given in condensed form in Fig. 2.


 Fig. 2.—KIO₃-NaIO₃-H₂O.

The two salts are not far from the same solubility, expressed either in weight per cent. or in mole per cent., and therefore permit a test of the theorem of Kuklin,⁷ that if the solubilities of two salts with a common ion

⁷ Kuklin, *J. Russ. Phys.-Chem. Soc.*, **61**, 667 (1929); from *Chem. Abstracts*, **24**, 1270 (1930).

do not differ much, the solution saturated with respect to both will contain them in the ratio of their solubility as pure salts. The data are assembled in Table III for 25 and 50°, at which temperatures the solubilities of the two salts are fairly near together.

TABLE III

Temp., °C.	SOLUBILITY RATIOS OF KIO ₃ AND NaIO ₃		Ratio (a)/(b)
	(a) $\frac{\text{Soly. NaIO}_3}{\text{Soly. KIO}_3}$ in water	(b) $\frac{\text{Soly. NaIO}_3}{\text{Soly. KIO}_3}$ at mutual saturation	
	Wt. %	Wt. %	
25	1.014	1.060	0.957
50	1.021	1.029	.992

The ratio $a:b$ should be unity for agreement with the theorem. The experimental ratio approaches that value most closely at 50°, although the solubility ratio is closer to unity at 25°. The use of molar ratios would change the figures of the last column only slightly.

XIII. POTASSIUM IODATE, POTASSIUM CHLORIDE AND WATER

Using the same procedure as before in the making up of phase complexes, and rotating the samples for periods of two or three days up to two weeks which time was shown by analysis of solution to be sufficient for attainment of external equilibrium, the saturated solution was analyzed iodi-

TABLE IV
KIO₃-KCl-H₂O

Temp., °C.	Original complex		Saturated solution		Density	Solid phase
	Wt. % KIO ₃	Wt. % KCl	Wt. % KIO ₃	Wt. % KCl		
5	...	0.00	5.16	0.00	1.043	KIO ₃
	19.03	2.52	2.91	3.03	1.044	KIO ₃
	17.81	8.47	1.79	10.13	1.084	KIO ₃
	17.03	16.46	1.50	19.56	1.147	KIO ₃
	1.44	22.64	1.170	KIO ₃ + KCl
	0.00	...	0.00	22.84	1.155	KCl
25	...	0.00	8.45	0.00	1.071	KIO ₃
	17.90	2.42	5.83	2.78	1.066	KIO ₃
	18.20	5.06	4.29	5.93	1.073	KIO ₃
	17.68	10.49	3.01	12.37	1.109	KIO ₃
	17.35	16.63	2.40	19.64	1.153	KIO ₃
	2.10	25.82	1.197	KIO ₃ + KCl
	0.00	...	0.00	26.36	1.179	KCl
50	...	0.00	13.21	0.00	...	KIO ₃
	24.00	5.59	7.42	6.83	...	KIO ₃
	22.92	12.60	4.71	15.64	...	KIO ₃
	21.03	19.01	3.74	23.17	...	KIO ₃
	3.07	29.08	...	KIO ₃ + KCl
	1.26	50.02	1.77	29.46	...	KCl
	0.00	...	0.00	30.03	...	KCl

metrically for iodate content and by evaporation to dryness for water content. In the latter procedure, in order to secure the highest accuracy, the initial evaporation at 100–110° was followed by a period of heating in an oven for one to two hours at 220°, to remove the traces of water which are held by potassium chloride at lower temperatures.

These figures may be used to give a good exhibit of the method of graphic extrapolation of tie-lines when the analysis is one of high accuracy. For the eleven above cases capable of being tested by the method, the average deviation of the extrapolated point from the true composition of the iodate or chloride is only 0.07%, the maximum 0.33%. It seems definite that this represents a better analysis than could be carried out by direct means

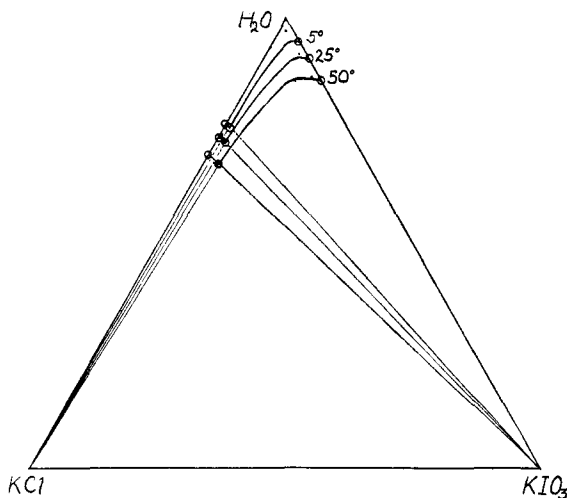


Fig. 3.—KIO₃-KCl-H₂O.

on a sample taken directly from the system and freed from mother liquor by mechanical means; it would be hard to believe that the liquid could be so reduced in amount as to leave an impurity amounting to less than 0.33%. Of course, where compounds show congruent solubility and are therefore capable of recrystallization without decomposition, direct analysis may be made to give the more accurate results; but in other cases it seems clear that the indirect method of graphic extrapolation may be made the more dependable.

When the solubility curves for this system are plotted on a sufficiently large scale, there appears a peak in the curve representing a minimum solubility not far from the point for pure potassium iodate. This is a natural result of the wide difference in molecular weight of the two salts; by the common ion effect, the added chloride precipitates approximately its equivalent of the heavier iodate, thus reducing the percentage concen-

tration of the solution. In the 25° isotherm a minimum density corresponding to this point was noted. If replotted on the basis of mole percentages, the minimum of course disappears. The curves are shown in condensed form in Fig. 3.

In this system there appear no compounds corresponding to those previously described⁸ for the corresponding sodium salts. This stands in conflict with the early statements of Serullas⁹ and of Filhol,¹⁰ who were doubtless misled by mixtures; Filhol's solid phase was probably recognized by him as a mixture.

XIV. POTASSIUM IODATE, POTASSIUM SULFATE AND WATER

The experimental procedure for this system was the same as for the others discussed. The time of rotation was about two weeks at 5°, about five days at 50°, which times were shown by analysis of the solutions to be sufficient for attainment of external equilibrium. For the analysis of the saturated solutions, a sample was tested iodimetrically for iodate concentration and a second sample evaporated to dryness at 100°, followed by heating at 220° for two hours to remove the traces of water otherwise retained by potassium sulfate. The results are given in Table V and shown graphically in Fig. 4.

TABLE V
KIO₃-K₂SO₄-H₂O

Temp., °C.	Original complex		Saturated solution		Density	Solid phase
	Wt. % KIO ₃	Wt. % K ₂ SO ₄	Wt. % KIO ₃	Wt. % K ₂ SO ₄		
5	...	0.00	5.16	0.00	1.043	KIO ₃
	17.04	3.49	3.14	4.07	1.060	KIO ₃
	2.57	7.08	1.081	KIO ₃ + K ₂ SO ₄
	1.51	22.01	1.80	7.25	1.077	K ₂ SO ₄
	0.00	...	0.00	7.64	1.062	K ₂ SO ₄
25	...	0.00	8.45	0.00	1.071	KIO ₃
	20.33	4.01	5.66	4.75	1.085	KIO ₃
	20.02	6.50	4.72	7.74	1.103	KIO ₃
	4.30	9.65	1.117	KIO ₃ + K ₂ SO ₄
	2.12	21.96	2.44	10.10	1.103	K ₂ SO ₄
	0.00	...	0.00	10.76	1.083	K ₂ SO ₄
50	...	0.00	13.21	0.00	...	KIO ₃
	30.01	6.06	8.68	7.90	...	KIO ₃
	30.01	8.61	7.39	11.43	...	KIO ₃
	7.06	12.06	...	KIO ₃ + K ₂ SO ₄
	3.05	29.99	3.78	13.00	...	K ₂ SO ₄
	0.00	...	0.00	14.18	...	K ₂ SO ₄

⁸ Foote and Vance, *Am. J. Sci.*, **18**, 375 (1929).

⁹ Serullas, *Ann. chim. phys.*, [2] **43**, 121 (1830).

¹⁰ Filhol, *J. Pharm. Chim.*, **25**, 431 (1839); **25**, 506 (1839); *J. prakt. Chem.*, [1] **18**, 457 (1839); see Mellor, "Comprehensive Treatise," 1922, Vol. II, p. 335.

The solubility of potassium sulfate at the three temperatures agrees with the interpolated figures given in "International Critical Tables" within $\pm 0.02\%$. The algebraic extrapolation of tie-lines gives here results as gratifying as in the previous instance; the average deviation from the composition of the solid phase is 0.06% , the maximum 0.20% . Compound formation, such as occurs in the case of the corresponding sodium salts,¹¹ is missing.

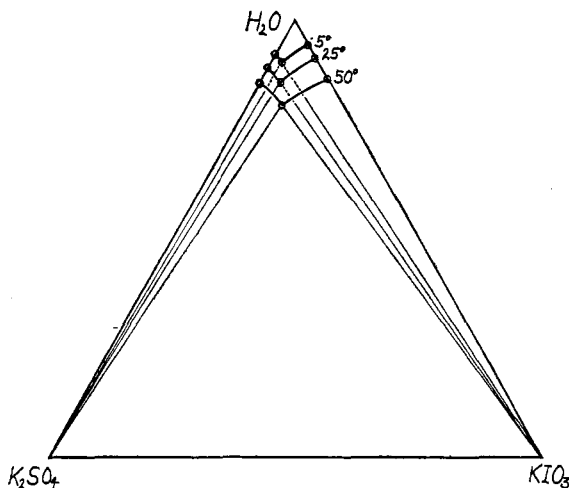


Fig. 4.— $\text{KIO}_3\text{-K}_2\text{SO}_4\text{-H}_2\text{O}$.

Summary

At the three temperatures of 5, 25 and 50° the three-component systems consisting of water and the following salt pairs have been studied; magnesium iodate and sodium iodate, sodium iodate and potassium iodate, potassium iodate and potassium chloride, potassium iodate and potassium sulfate. Double salt formation has not been found in any of these cases. The existence of colloidal constituents in the solutions of sodium iodate and potassium iodate has been discussed. The method of algebraic extrapolation of tie-lines has been discussed, and its usefulness shown in indicating the accuracy of solubility determinations in three-component systems.

NEW YORK, N. Y.

¹¹ Foote and Vance, *Am. J. Sci.*, 19, 203 (1930).