321. *The Iodous Sulphates.*

By Irvine Masson and Cyril Argument.

The preparation of Chrétien's yellow crystalline iodous sulphate by decomposing iodic acid by heat in concentrated sulphuric acid is shown to be a mingling of three or more reactions, one thermal and the others isothermal at room temperature. The thermal reaction $I_2O_5 \longrightarrow I_2O_3 + O_2$ occurs quantitatively in fuming sulphuric acid at 215—220°, the product being there stabilised as an iodous sulphate. In the cold, the reaction $2I_2 + 3I_2O_5 \longrightarrow 5I_2O_3$ occurs, with a 100% yield of Chrétien's sulphate, when the reagents are shaken in concentrated sulphuric acid.

By two methods, Chrétien's sulphate is proved to be I_2O_3,SO_3, not hydrated as its discoverer and others represented it. In acid more dilute than H_2SO_4,H_2O it decomposes, reversing its own synthesis. In fuming sulphuric acid it undergoes transition into a white crystalline iodous sulphate, determined as $I_2O_3,4SO_3,xH_2O$, where probably $x = 1$. The converse (isothermal) transition, on dilution of the fuming acid to below 100% H_2SO_4 by addition of concentrated sulphuric acid, leads to the yellow crystals again, but does so through the remarkable *intermediate* step of precipitating iodine and pentoxide, which disappear again. This is discussed, and it is concluded that the " white " solutions in fuming acid contain their tervalent iodine in a complex ion (possibly negative), while the " yellow " solutions in concentrated acid contain the cations of an equilibrium $IO^+ + 2H^+ \rightleftharpoons I^{+++} + H_2O$; the bare ion I^{+++} is considered to be the agent responsible both for the curious features of the transition and, in general, for the organic reactions of iodous salts, elsewhere treated.

It was well recognised by Fichter and his associates at Basel that tervalent iodine is essentially base-forming, yielding with suitable acids salts which are either normal (" Jodi- ") or basic (" Jodyl "). All these are rapidly decomposed by water into iodic acid, iodine, and the acid whose radical they contain.

Such compounds (other than iodine trichloride) have, in general, been prepared by methods of two types. One is the oxidation of elementary iodine at the ordinary temperature by such agents as ozone or chlorine monoxide, in the presence of the acid—as far as possible anhydrous—whose iodous compound is desired. By such means Schützenberger (*Compt. rend.*, 1861, **52**, 135; 1862, **54**, 1026) obtained the white crystalline iodine triacetate, doubtless a covalent compound; Fichter, Kappeler, and Krummenacher (*Z. anorg. Chem.*, 1915, **91**, 134) obtained an unstable perchlorate represented as $I(ClO_4)_3,2H_2O$; Kappeler (*Ber.*, 1911, **44**, 3496) made a nitrate of uncertain composition; Beger (*Chem. Z.*, 1906, **33**, 1232) and Fichter and Rohner (*Ber.*, 1909, **42**, 4092) oxidised iodine with ozone in chloroform to I_4O_9, which the latter authors regard as $I(IO_3)_3$.

The other method, derived from old experiments by Millon, has involved the partial decomposition of iodic acid by heat in the presence of concentrated sulphuric acid. By this process Chrétien (*Compt. rend.*, 1896, **123**, 814; *Ann. Chim. Phys.*, 1898, **15**, 358) obtained a yellow crystalline compound represented by him as $I_2O_3,SO_3,\frac{1}{2}H_2O$; and Pattison Muir (J., 1909, **95**, 656) made and studied a lemon-yellow solid of analytical composition IO_2, which chemists follow Fichter in regarding as a basic iodous iodate I_2O_3,I_2O_5, or IO,IO_3 iodyl iodate, rather than as an oxide of quadrivalent iodine. Beger's " normal " iodate above mentioned was also got by Fichter and Kappeler (*loc. cit.*) by the thermal method from iodic acid, in a medium of metaphosphoric acid.

Besides these compounds, others have been reported whose reality Fichter and his associates have disproved or questioned. With Kappeler and Helfer (1915, *loc. cit.*) he made a further iodous substance, by heating Chrétien's sulphate with sulphur trioxide in a sealed tube, which was represented as " Neutrales Jodisulfat," $I_2(SO_4)_3$, in approximate accord with the analytical data; and the authors remarked " Durch diese neue Untersuchung ist einstweilen die Chemie der Sulfate des dreiwertigen Jods abgeschlossen." However, Bahl and Partington (J., 1935, 1258), in repeating the experiments of Muir and of Chrétien (*locc. cit.*), were unable to confirm Chrétien's formula for his sulphate, assigning it (by analytical difference) $1H_2O$ per I_2O_3,SO_3 instead of $0{\cdot}5H_2O$; thereupon Fichter and Dinger (*Helv. Chim. Acta*, 1936, **19**, 607) adversely criticised Bahl and Partington's data and adduced their own analyses of the solid in support of Chrétien's original formula. It will be shown (p. 1704) that all three sets of authors have been misled, and that the composition of Chrétien's yellow sulphate is simply anhydrous I_2O_3,SO_3, that is $(IO)_2SO_4$; and that it can be turned into another iodous sulphate, a white compound, with interesting properties.

Our work, prompted by the need to prepare properly an iodous sulphate as a reagent with which to attack aromatic compounds (Masson and Race, J., 1937, 1718; Masson and Hanby, preceding paper), has enabled us to obtain light upon the general equilibria between the elementary, the iodic, the hypoiodous, and two, perhaps three, iodous conditions of iodine. These equilibria have two aspects: one is the effect of high temperature; the other is the effects of changes in composition at room temperature; and in both aspects, a powerful factor proves to be the composition of the acidic medium in which the equilibria must be studied if they are to be made distinct.

As is well known, if iodine pentoxide is heated by itself it dissociates into its elements irreversibly; the process begins to be noticeable towards 350°, and at this temperature some of the iodine is retained by the residual solid in a complex of uncertain nature. Heated in concentrated sulphuric acid, however, the pentoxide dissociates more easily, as Chrétien showed (using iodic acid), oxygen being gradually evolved at 250—260°; the yellowish liquid, when cooled, deposits mixtures of iodous and iodic compounds. At slightly higher temperatures, iodine as well as oxygen is strongly evolved, and the liquid turns deep brown. This is the condition used by Chrétien and subsequent authors in order to obtain his sulphate: the brown liquor (to which more iodine may with advantage have been added during the heating, according to Chrétien and to Fichter) is cooled and allowed to stand in a desiccator for 1—2 days or longer, whereupon it turns yellow in depositing yellow crusts of the material. Consideration shows, however, not only that this process is inconvenient and far from quantitative, but also that it confuses in uncertain proportions at least three distinct reactions; and experiment proves that these can readily be separated, studied, and made to proceed quantitatively.

The primary thermal decomposition of iodine pentoxide

$$I_2O_5 \longrightarrow I_2O_3 + O_2 \quad . \; . \; . \; . \; . \; . \; . \; . \; . \quad (1)$$

is, in the absence of an acidic medium, indistinguishable from the further decomposition

$$2I_2O_3 \longrightarrow 2I_2 + 3O_2 \quad . \; . \; . \; . \; . \; . \; . \; . \; . \quad (2)$$

Iodine sesquioxide is itself unknown; but in a suitably powerful acid, its basic quality comes into play to stabilise it as a salt, and to lessen the temperature at which, owing to reaction (1) alone, an evolution of oxygen from heated iodine pentoxide begins. Nevertheless, ordinary concentrated sulphuric acid (used by the several authors named) is scarcely of powerful enough acidity usefully to protect the iodous product against the thermal decomposition represented in (2). We find, however, that fuming sulphuric acid will do so; it also lowers still further the temperature needed to bring reaction (1) to rapid completion. By heating iodine pentoxide in fuming sulphuric acid (20% SO_3) at 215—220° in connexion with a gas burette, one obtains a brisk evolution of oxygen which ceases sharply when 1·00 mol. of measured oxygen per mol. of I_2O_5 has been released, the white solid finally disappearing into solution at the same time; no iodine whatever is produced, the liquid formed is of a very pale yellow tint, and its analysis corresponds to a sulphate of an oxide $I_2O_{3\cdot00}$ as the solute. This, then, affords one easy way quantitatively to prepare a crystallisable iodous sulphate; the other way depends upon reactions at room temperature, as follows.

In Chrétien's method the thermal decomposition of iodic acid must be carried so far as to give copious fumes of iodine and a deep brown liquor; and it is this liquor which, when kept at the ordinary temperature, becomes the source of the desired product. But we find that the same liquor can be got by merely mixing iodine and iodine pentoxide in concentrated sulphuric acid without any application of heat: so that Chrétien's and Fichter's thermal method may be said to be mainly a crude way of producing some free iodine, which shall eventually react in the cold with some undecomposed iodine pentoxide. We mix the two powdered reagents in the concentrated acid (92—98% H_2SO_4) in the exact proportions of the equation

$$2I_2 + 3I_2O_5 = 5I_2O_3 \qquad (3)$$

and, without applying heat, turn the whole for a day or two on a wheel: the initial brown liquor and the temporary excess of solid pentoxide quickly give place to a very pale liquid and a finely crystalline, brilliantly yellow precipitate of Chrétien's sulphate. The yield is quantitative, including a small measured solubility, *viz.*, 0·024 mol. as I_2O_3 per litre of 96% sulphuric acid. Heating the mixture does not facilitate but rather tends to reverse the reaction.

The nature of the several equilibria involved in the preparative reaction summarised in equation (3) is treated by one of us in the succeeding paper on the brown solute, since the properties of this substance prove to dominate the matter. It is clear, however, that essentially the process consists in a quantitative reversal, by means of concentrated sulphuric acid at the ordinary temperature, of the familiar aqueous decomposition which converts the lower oxy-compounds of iodine into iodic acid and the element.

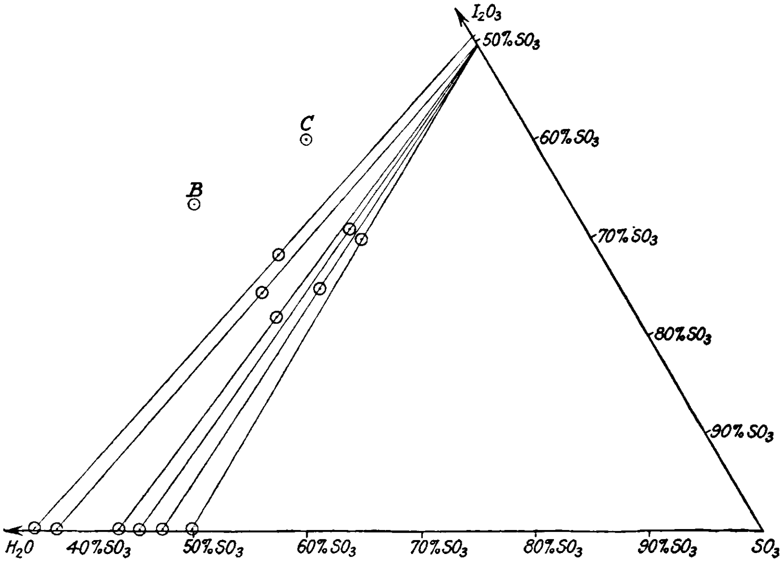
Composition of the Yellow Sulphate.—In the work of previous authors on Chrétien's sulphate, one difficulty, inherent in the preparative method used by them, was to stop the heating at the right stoicheiometrical point. The ensuing purification of the solid product for analysis, as performed by all these workers, consisted in spreading it on porous tiles in a desiccator. Fichter and Dinger left it thus for 1—1½ hours only, stating that a longer time affected the purity. It is obvious that a finely grained material, which Chrétien's sulphate is, cannot be expected easily to give up all its adherent mother-liquor to porous porcelain; and that in the necessary grinding, some exposure to moist air can scarcely be avoided without special apparatus which seems not to have been used. Such sources of error can readily affect the apparent composition of a very hygroscopic solid, in which the difference between a mono- and a hemi-hydrate of I_2O_3,SO_3 amounts only to 2·3% by weight and the water content is assessed by difference; this is pointed out by Fichter and Dinger in criticising Bahl and Partington, and we venture to apply the same remark in turn to the difficulty of distinguishing between a hemihydrate and an anhydrous compound by this method, which we ourselves tried. In any case, however, having found the quantitative methods just described for making Chrétien's compound on any scale, we have further been enabled to avoid these risks of under-purification or of exposure to moisture, by resorting to two independent principles of analysis, in each of which the solid is merely filtered through sintered glass in a dry atmosphere from its isothermally saturated solution in the sulphuric medium. In one of these methods the medium was dosed with pure, dried barium sulphate, which dissolved in it but was (as it proved) unable to form solid solutions with the crystalline iodous material, and was therefore of use as an indicator: hence the barium sulphate content of the analysed "wet" solid, by comparison with that of the analysed filtrate from it, gave immediately the quantity of liquid adhering to the crystals, and consequently the true composition of the latter. The other principle was that of "wet residues" commonly ascribed to Schreinemakers, in which we varied the composition of the solvent in the ternary system I_2O_3–SO_3–H_2O through the range from H_2SO_4,H_2O to H_2SO_4, *i.e.*, over the whole range of stability of the yellow sulphate at the ordinary temperature. The results of both methods, which are recorded in the experimental section, agreed conclusively with the composition I_2O_3,SO_3, and forbade either the hemihydrate of Chrétien and of Fichter and Dinger, or the monohydrate of Bahl and Partington. Fig. 1 shows the results of the second method graphically.

Effect of Water upon the Yellow Sulphate.—In a medium of H_2SO_4,H_2O, the synthetic reaction, by which the substance was prepared in a more concentrated acid, already begins to be slightly reversed, liberating a little iodic acid and iodine, the latter partaking in a secondary reaction which forms visible traces of the brown solute. (In Fig. 1 the imperfect convergence of the line belonging to this particular medium is due to this cause.) In very slightly more aqueous media the reversal is almost complete (and can be made finally so by dilution, warmth, and extraction of the iodine); almost the whole iodous salt is decomposed, with valency-bifurcation, in the sense of the schematic equation

$$5I_2O_x \longrightarrow xI_2O_5 + (5 - x)I_2 \qquad . \; . \; . \; . \; . \; . \; . \qquad (4)$$

which governs the aqueous behaviour of derivatives of all lower oxides of iodine and is the basis of their quantitative analysis.

FIG. 1.



System I_2O_3–SO_3–H_2O *at* 15·5—18·0°. *Wet solids and solutions of yellow iodyl sulphate. Compositions converge upon* I_2O_3,SO_3. (*For erratic line, cf. text.*) B *is position of* I_2O_3,SO_3,H_2O (*Bahl and Partington*) *and* C *is position of* $I_2O_3,SO_3,\frac{1}{2}H_2O$ (*Chrétien; Fichter*).

White Iodous Sulphate.—In more anhydrous media, Chrétien's sulphate ceases to be stable in sulphuric acid of concentration exceeding 100% H_2SO_4: the bright yellow crystals, put into such an acid, turn into another crystalline sulphate, practically white, with a faintly yellow solution, and increasingly soluble in the medium as its SO_3 content is raised. In this solid the iodine is still wholly tervalent, but the proportion of sulphur trioxide is much higher than in the yellow solid. The composition has been sought by the same two methods as were successful with the yellow form; but, despite care, it has proved difficult to define it completely with confidence. In the "wet residues" method, five pairs of duplicate analyses gave a diagram with so narrow a parallax (owing mainly to the limited range of fuming acids available as liquid solvents) that this group of data merely set approximate limits of $I_2O_3,4SO_3,H_2O$ and $I_2O_3,3SO_3$ to the possible formula. The use of barium sulphate as indicator, however, was more satisfactory, except in the difficult point of deciding the degree of hydration (if any); one experiment gave $I_2O_3,4{\cdot}11SO_3,1{\cdot}3H_2O$, and a second gave $I_2O_3,4{\cdot}08SO_3,0{\cdot}4H_2O$ (the respective water-contents are only 3·7% and 1·1% by weight). We conclude that our white solid is $I_2O_3,4SO_3,xH_2O$, adding that probably $x = 1$, and if so the substance is $H_2SO_4,I_2(SO_4)_3$, a complex containing the $I_2(SO_4)_3$ of Fichter, Kappeler, and Helfer (*loc. cit.*).

Local wetting with water of the white solid in fuming sulphuric acid, so long as the general composition of the medium does not fall below 100% H_2SO_4, turns it to a deep manganate-green or an intense blue colour, which remains permanent in a sealed specimen. This occurrence is due to the local liberation of elementary iodine, which at once reduces fuming sulphuric acid to sulphur sesquioxide in being reoxidised to the iodous state. The evidence for this explanation is qualitative: direct experiments with iodine and fuming sulphuric acids gave solutions of the same colours, which evolved sulphur dioxide, and which showed the same colours and behaviour as solutions of sulphur in these acids, wherein various other authors have recognised a coloured substance S_2O_3.

The Transition between the Two Sulphates.—The transition yellow ⟶ white in media exceeding 100% H_2SO_4 proceeds normally, like that from a crystalline sulphate to the corresponding hydrogen sulphate; but the reverse change, induced by adding the white compound in fuming acid to concentrated (96%) acid until the medium is a little less than 100% H_2SO_4, takes a path which is of a type quite new to us in any reversible transition among inorganic salts; and its interpretation throws light upon the natures of iodous compounds. The very pale yellow liquid at first remains clear, then it grows turbid and grey with a suspension of solid iodine and (if the proportions are suitable) a white solid, apparently pentoxide; as one watches, the grey turns to dusky greenish and then yellow, and in a short time the temporarily precipitated iodine (and any white suspension) has vanished again and is wholly replaced by pure crystals of the yellow I_2O_3,SO_3, the stable phase in the new concentration of acid. In the later stages, the brown colour is seen which iodine gives when put into sulphuric acid solutions of the pentoxide or of the yellow sulphate; but it is weak and transient. Temperature changes are not concerned in the series of events. Fig. 2 shows microphotographs of (*a*) the white sulphate in fuming acid, (*b*) the dusky greenish stage of transition during slight dilution, the large irregular needles of iodine being interspersed with small crystals of the yellow product which is superseding them, (*c*) the yellow I_2O_3,SO_3 (somewhat rounded by stirring) in concentrated acid.

The interest of this perfectly reversible transition between the white and the yellow compound lies in the fact that, although the iodine begins and ends in the tervalent state, it spontaneously undergoes valency-bifurcation and reassembly, *en route* from one to the other. Thus:

(White). *Media*: $H_2SO_4 + nSO_3$.

$$\begin{tikzcd} & I^{I} + I^{V} \arrow[dr] & \\ 2I^{III} \arrow[ur] \arrow[rr, dashed, shift right] & & 2I^{III} \arrow[ll, shift right] \end{tikzcd}$$

(Yellow). *Media*: H_2SO_4—H_2SO_4,H_2O.

That an atomic change so extreme as valency partition occurs, only to be spontaneously followed by valency redintegration to the original value, must mean that the tervalent iodine of the white sulphate is in a radical different from that in which the tervalent iodine exists in the yellow sulphate. The two compounds cannot be related in the same way as interconvertible sulphates and hydrogen sulphates, which have one and the same cation in common; and our view of the matter is as follows.

The ion in the yellow crystals $(IO)_2SO_4$ is, we do not doubt, the iodyl cation IO^+; and their solution in concentrated sulphuric acid behaves towards aromatic compounds (as we show elsewhere) so as to suggest that it contains some proportion, not necessarily more than quite small, of iodous cations I^{+++}, produced in an equilibrium

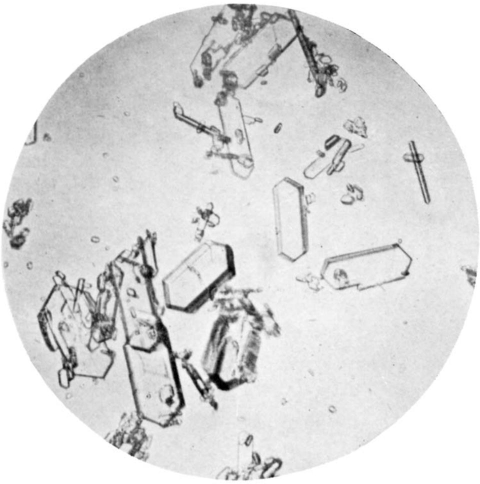
$$IO^+ + 2H^+ \rightleftharpoons I^{+++} + H_2O.$$

The monohydrate formula for the white crystals, $I_2O_3,4SO_3,H_2O$, or $HI(SO_4)_2$, might suggest the possibility that their ion is a negative one, $[I(SO_4)_2]^-$; without, however, entering into detailed speculation on this point, we are at all events entitled to regard their solution in fuming acid as containing the tervalent iodine in the form of a complex ion containing sulphur trioxide and kept in stable equilibrium by the excess of sulphur trioxide in the medium. On removal of this excess by dilution to slightly below 100% H_2SO_4, the complex dissociates; and we conceive that its tervalent iodine is thus all momentarily released as simple I^{+++}, not a stable entity in so high a concentration. At such a concentration, therefore, it is able to produce, by the electron partition $2I^{III} \longrightarrow I^{I} + I^{V}$, free iodine and the pentoxide faster than these can undergo their normal reassembly to give yellow iodyl sulphate, which later visibly proceeds in the liquor; and faster also than the establishment of the equilibrium

$$I^{+++} + H_2O \rightleftharpoons IO^+ + 2H^+,$$

the progress of which also constitutes yellow iodyl sulphate. Although this hypothesis may be mistaken in naming I^{+++} as the immediate precursor of the observed iodine and

FIG. 2.



(a) $I_2O_3,4SO_3,xH_2O$; *white iodous sulphate.*

(b) *Transition: showing iodine as a metastable intermediate, and developing crystals of*

(c) I_2O_3,SO_3; *yellow iodyl sulphate.*

pentoxide, yet the dynamics of the whole train of processes, coupled with our other work, are clarified by invoking it; and, on the grounds of electronic structure, it appears improbable that this ion could exist *per se* except in minute equilibrium concentration or as a transitory product intermediate between one complex, that of the white compound, and another, that of the yellow. The same instability of I^{+++} is held to account for the swift organic reactions of tervalent iodine which we have elsewhere described.

EXPERIMENTAL.

In syntheses of the yellow iodyl sulphate by the method described, at a late stage of the process the product should be ground under its own liquor, to release traces of enclosed solid reagents. Precautions against the access of water vapour must be strict in handling the yellow compound, and are difficult to make strict enough for the white compound. Our sintered-glass filtering apparatus was specially designed to further these precautions and to simplify sampling with a minimum of exposure. For each analysis of a solid or of a solution, duplicate samples were used. In dilutions of samples with water, appropriate vessels and care are needed to prevent loss of vapour, especially in systems involving the white sulphate and fuming acid.

Determination of the Degree of Oxidation of Iodine.—Given that, in warm water,

$$5I_2O_x,\text{aq.} \longrightarrow xI_2O_5,\text{aq.} + (5 - x)I_2,$$

we warm the well-diluted material under reflux, adding pure carbon tetrachloride; the iodine is extracted with this solvent, and the extract, after the addition of water, potassium iodide solution, and a few drops of dilute acid, is titrated with thiosulphate; to the acid aqueous layer, freed from iodine by the extraction, is added a bare excess of potassium iodide solution, and the liberated iodine is extracted with tetrachloride and is similarly titrated. If the numerical ratio of the second titre to the first is R, we have $x = 5R/(6 + R)$; hence for $x = 1, 2, 3, 4, 5$, R is respectively 1·5, 4, 9, 24, ∞, which gives a very sensitive measure of the degree of oxidation, as others have noted who have used some form of this method. All the preparations and samples analysed for the work of this paper gave values of R lying between 9·019 and 9·004, corresponding with $I_2O_{3\cdot003}$ to $I_2O_{3\cdot000}$.

Sulphur trioxide was always determined gravimetrically as barium sulphate in the aqueous solution from the foregoing extractions. In the indicator method, separate samples were required for determining the indicator content; in them, after dilution, the liberated iodine was removed by distillation at reduced pressure before collecting the precipitated indicator on a filter; and the comparability of these samples with those taken for the remaining constituents was tested and proved by their giving identical figures for the content of iodic acid.

Analytical Results.—A. *Yellow solid.*

(i) " Barium sulphate indicator " method. One experiment. Temp. 14·5—17°.

	Rel. wts. in filtrate.		Rel. wts. in wet solid.		Rel. mols. in pure solid.	
	(*a*).	(*b*).	(*a*).	(*b*).	(*a*).	(*b*).
$BaSO_4$	1·0000	1·0000	0·0161	0·0161	—	—
I_2O_3	0·0798	0·0800	0·5452	0·5456	1·000	1·000
SO_3	14·507	14·502	0·3781	0·3784	1·000	1·001
H_2O (diff.)	3·548	3·553	0·0606	0·0599	0·065	0·043
Total	19·135	19·135	1·0000	1·0000	—	—

(ii) " Wet residues " method. A preliminary set of 9 solids and their liquids having virtually confirmed the results just given, the following definitive data were obtained; these these are the data used for Fig. 1, and (excepting col. 1) are expressed in *molecular* percentages, those for H_2O (diff.) being for brevity omitted in each half of the table.

Temp. 15·5—18°.

Solvent acid : H_2SO_4, wt. %.	Wet solids.		Solutions.	
	I_2O_3.	SO_3.	I_2O_3.	SO_3.
86·3	28·00, 28·48	43·05, 43·63	0·170, 0·167	36·06, 35·67
88·6	24·36, 24·33	43·90, 43·93	0·143, 0·143	37·72, 37·93
94·2	21·73, 21·70	46·42, 46·40	0·084, 0·084	43·34, 43·29
95·8	30·86, 30·72	48·43, 48·20	0·065, 0·065	45·24, 45·02
97·5	24·74, 24·68	48·76, 48·73	0·066, 0·067	46·97, 47·35
99·8	30·33, 29·39	49·95, 49·82	0·121, 0·121	49·74, 49·76

B. *White solid.*

(i) " Barium sulphate indicator " method. Two experiments. Weights :—

	Experiment 1 : 16·5—18·5°.				Experiment 2 : 16·0—19·0°.			
	Filtrate.		Wet solid.		Filtrate.		Wet solid.	
$BaSO_4$	1·0000	1·000	0·0447	0·0444	1·0000	1·0000	0·0463	0·0468
I_2O_3	0·0754	0·0749	0·2785	0·2794	0·1092	0·1099	0·2763	0·2635
SO_3	6·866	6·868	0·6067	0·6059	7·369	7·347	0·6213	0·6266
H_2O (diff.) ...	1·076	1·078	0·0701	0·0703	1·235	1·247	0·0561	0·0631
Total ...	9·017	9·021	1·0000	1·0000	9·713	9·704	1·0000	1·0000

These yield formulæ : Expt. 1, $I_2O_3,4{\cdot}11SO_3,1{\cdot}34H_2O$;
Expt. 2, $I_2O_3,4{\cdot}08SO_3,0{\cdot}41H_2O$.

(ii) " Wet residues " method. Molecular percentages (except col. 1); H_2O (diff.) omitted from table. Temp. 16·5—18·5°.

Solvent acid : free SO_3, wt. %.	Wet solids.		Solutions.	
	I_2O_3.	SO_3.	I_2O_3.	SO_3.
17·4	7·42, —	60·07, —	0·115, 0·115	55·95, 55·80
18·7	6·99, —	59·35, —	0·111, 0·111	56·35, 55·13
25·8	7·44, 7·47	61·13, 61·08	0·172, 0·171	58·85, 58·81
32·6	10·91, 10·55	65·38, 63·84	0·181, 0·182	61·16, 61·76
33·7	8·34, 8·30	64·41, 64·11	0·188, 0·188	61·79, 62·04

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