

202. *Solutions in Sulphuric Acid. Part XIII.* The Electrical Conductivity of Solutions of Hydrogen Disulphates and the Self-dissociation of Sulphuric Acid.*

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The results of measurement of conductivities of solutions of hydrogen disulphates are discussed and utilised in conjunction with other conductivity data to obtain a value for the autoprotolysis constant of sulphuric acid at 25°. The value obtained is compared with that obtained by cryoscopy at 10·4° (Part IV; *J.*, 1950, 2516) and with estimates made by other authors.

ATTEMPTS have been made by several authors to determine the nature and extent of the self-dissociation of sulphuric acid. Hammett and Deyrup (*J. Amer. Chem. Soc.*, 1932, **54**, 2731) recognised that two processes probably contribute to the self-dissociation, *viz.*, an

* Part XII, *J.*, 1952, 221.

autoprotolysis $2\text{H}_2\text{SO}_4 = \text{H}_3\text{SO}_4^+ + \text{HSO}_4^-$ and some other process involving sulphur trioxide which they wrote as $3\text{H}_2\text{SO}_4 = \text{H}_3\text{O}^+ + \text{H}_2\text{S}_2\text{O}_7 + \text{HSO}_4^-$. They calculated a value for the concentration of hydrogen sulphate ion in the pure acid of 0.027 molal at 25° from the results of their colorimetric study of the ionisation of 2 : 4 : 6-trinitroaniline in 96–100.1% sulphuric acid, utilising the relationship $\log [\text{BH}^+]/[\text{B}] + \log [\text{HSO}_4^-] = K$, where B is any basic indicator and K is a constant; K was obtained from the values of $[\text{BH}^+]/[\text{B}]$ in slightly aqueous solvents where $[\text{HSO}_4^-]$ was assumed to be equal to the stoichiometric concentration of water. The logarithm of the ratio $[\text{BH}^+]/[\text{B}]$ was then plotted against the logarithm of the molality of water and the curve extrapolated to zero water concentration. From the extrapolated value of $[\text{BH}^+]/[\text{B}]$ the concentration of hydrogen sulphate ion in the pure acid may be calculated. The value they obtained is, however, certain to be high because the indicator itself produces hydrogen sulphate ion, the concentration, of course, depending on the concentration of the indicator, but it could easily be comparable with the concentration resulting from the self-dissociation. Since the concentration of the indicator was not given, no correction can be made. In any case the unavoidable errors of the colorimetric method may lead to appreciable errors in any estimate of the hydrogen sulphate ion concentration by this method.

Recently Reinhardt (*J. Amer. Chem. Soc.*, 1950, **72**, 3359) has made similar calculations, although he gives no details, using Hammett and Deyrup's results, and claims to have obtained values of $K_{\text{ap}} = [\text{H}_3\text{SO}_4^+][\text{HSO}_4^-]$ ranging from 3×10^{-3} to 5×10^{-3} . Hammett and Deyrup's value of the hydrogen sulphate ion concentration leads to a value of $K_{\text{ap}} = 7.3 \times 10^{-4}$, not 2.4×10^{-2} as stated by Reinhardt. This author also attempted to calculate the extent of the autoprotolysis from the conductivity of the pure acid by assuming that the conductivities of the hydrogen and hydrogen sulphate ions are equal and that they can be estimated from the known transport number of the barium ion in sulphuric acid, the ionic conductivity of barium in water, and the viscosities of water and sulphuric acid, the conductivity-viscosity product for the barium ion being assumed to be the same in water and in sulphuric acid. He obtained a value of $K_{\text{ap}} = 6.0 \times 10^{-4}$. Another estimate was made by a simple linear extrapolation of the straight portions of the freezing-point curves for water and sulphur trioxide to zero concentration to obtain the freezing point of hypothetical undissociated sulphuric acid, from which a value of $K_{\text{ap}} = 4 \times 10^{-4}$ was calculated. However, it has been shown in Parts I, II, and IV (*J.*, 1950, 2473, 2493, 2516) that the extrapolation cannot be made in such a simple manner, and that in any case the concentration of dissociation products obtained in this way includes those formed by the self-dehydration $2\text{H}_2\text{SO}_4 = \text{H}_3\text{O}^+ + \text{HS}_2\text{O}_7^-$ and not only by the autoprotolysis as assumed by Reinhardt. It was finally concluded from the rough agreement of all these estimates that the dissociation involving sulphur trioxide was of no importance.

All this earlier information refers to 25°, and the only really exact confirmation that we have concerning the self-dissociation of sulphuric acid comes from the cryoscopic measurements described in Parts I, II, and IV (*loc. cit.*) and hence refers to the freezing point of sulphuric acid, *i.e.*, 10.36°. It was shown that sulphuric acid undergoes an autoprotolysis, $2\text{H}_2\text{SO}_4 = \text{H}_3\text{SO}_4^+ + \text{HSO}_4^-$, and a self-dissociation, which is not negligible, $2\text{H}_2\text{SO}_4 = \text{H}_3\text{O}^+ + \text{HS}_2\text{O}_7^-$ (at the very low concentration of these ions the incompleteness of the ionisation of water and disulphuric acid may be ignored), and it was found that

$$[\text{H}_3\text{SO}_4^+] = [\text{HSO}_4^-] = 0.013 \text{ molal}$$

hence

$$K_{\text{ap}} = [\text{H}_3\text{SO}_4^+][\text{HSO}_4^-] = 1.7 \times 10^{-4}$$

and that

$$[\text{H}_3\text{O}^+] = [\text{HS}_2\text{O}_7^-] = 0.0083 \text{ molal}$$

hence

$$K_{\text{id}} = [\text{H}_3\text{O}^+][\text{HS}_2\text{O}_7^-] = 7 \times 10^{-5}.$$

We now attempt to make a more accurate estimate of the degree of self-dissociation of sulphuric acid at 25° on the basis of the results presented in Parts IX—XII (*J.*, 1952, 204) and some further results of conductivity measurements presented later in this paper.

Before we can proceed to estimate the degree of self-ionisation of sulphuric acid from our conductivity data it is essential to establish which of the ions that result from the self-dissociation processes contribute significantly to the conductivity of the pure solvent. It has been shown in Part XII (*loc. cit.*) that the hydroxonium ion has a very small mobility like that of other cations except the hydrogen ion, and we shall now show, by considering the results of some conductivity measurements on solutions of ammonium and potassium disulphates, that the hydrogen disulphate ion has a much smaller mobility than the hydrogen sulphate ion.

The Conductivity of Solutions of Ammonium and Potassium Sulphate in Dilute Oleum.— Since it is not easy to prepare pure specimens of crystalline hydrogen disulphates, they were prepared in solution by addition of the appropriate sulphate to a dilute oleum. The conductivity of a dilute oleum solution was measured, and hence its composition determined. Sulphate was then added in successive weighed amounts, the conductivity being measured after each addition, until more than sufficient had been added to convert all the disulphuric acid initially present into hydrogen disulphate. (Some similar measurements have recently been reported by Reinhardt, *loc. cit.*)

The results are presented in Table 1 and Fig. 1. Concentrations of the various solutes have been calculated on the assumption that a dilute oleum is a dilute solution of di-

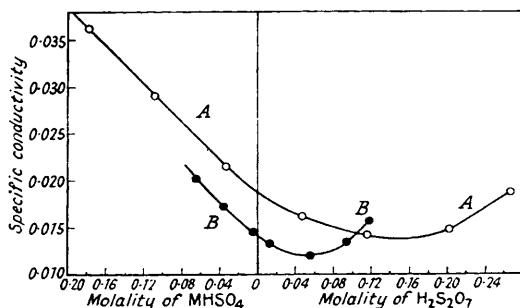


FIG. 1. Specific conductivities of solutions of ammonium and potassium sulphates in dilute oleum.

A, NH_4HSO_4 . B, KHSO_4 .

sulphuric acid in sulphuric acid and contains no other molecular species, and that sulphate is quantitatively converted into hydrogen disulphate in the presence of disulphuric acid according to the equation $2\text{H}_2\text{S}_2\text{O}_7 + \text{SO}_4^{--} = 2\text{HS}_2\text{O}_7^- + \text{H}_2\text{SO}_4$.

On the right ("acidic" side) of the central vertical axis in the figure, conductivities are plotted against the molality of disulphuric acid. The central vertical axis (neutral line) corresponds to the composition of the solvent sulphuric acid or of solutions whose composition can be expressed in terms of "neutral" salts such as ammonium hydrogen disulphate. On the left ("basic" side) of the neutral line, the conductivities are plotted against the molality of potassium or ammonium hydrogen sulphate. At the points where

TABLE 1. The conductivity of solutions of ammonium and potassium sulphate in dilute oleums.

Molalities			$10^5\kappa$, ohm^{-1} cm^{-1}	Molalities			$10^5\kappa$, ohm^{-1} cm^{-1}
$\text{H}_2\text{S}_2\text{O}_7$	$\text{NH}_4\text{HS}_2\text{O}_7$	NH_4HSO_4		$\text{H}_2\text{S}_2\text{O}_7$	KHS_2O_7	KHSO_4	
0.2668	—	—	2067	0.1177	—	—	1561
0.2023	0.0645	—	1572	0.0939	0.0138	—	1338
0.1170	0.1498	—	1406	0.0565	0.0612	—	1195
0.0474	0.2194	—	1612	0.0132	0.1045	—	1318
—	—	0.0334	2145	—	—	0.0054	1443
—	—	0.1092	2799	—	—	0.0356	1710
—	—	0.1786	3610	—	—	0.0646	2022

the two curves in the figure cut the "neutral" line these solutions have the analytical composition of ammonium or potassium hydrogen disulphates at a molality equal to that of the disulphuric acid in the original oleum, and the conductivities at these points are thus

the conductivities of the hydrogen disulphate solutions. The values thus obtained are given in Table 2 together with the conductivities of ammonium and potassium hydrogen sulphates, interpolated from the results given in Part XII (*loc. cit.*), for comparison.

It may be seen at once that the conductivities of the hydrogen disulphates ($\kappa_{\text{obs.}}$) are considerably smaller than those of the corresponding hydrogen sulphates at the same concentration. Moreover, it must be remembered that the autoprotolysis of the solvent is not repressed by the hydrogen disulphate ion, and hence the conductivity of the solvent should be subtracted from the observed conductivity of the hydrogen disulphate solutions in order to obtain the conductivity due to the hydrogen disulphate only. These corrected conductivities ($\kappa_{\text{obs.}} - \kappa_{\text{solvent}}$) are also given in Table 2, and it can be seen that they are

TABLE 2. Comparison of the conductivities of ammonium and potassium hydrogen sulphates and hydrogen disulphates.

	MHSO ₄		MHS ₂ O ₇		
	κ	κ	$\kappa_{\text{obs.}}$	$(\kappa_{\text{obs.}} - \kappa_{\text{solvent}})$	$[\kappa_{\text{obs.}} - \kappa(\text{H}_2\text{SO}_4 + \text{HSO}_4^-)]$
NH ₄	0.2668 <i>m</i>	0.0429	0.0198	0.0095	0.0048
K	0.1177 <i>m</i>	0.0245	0.0142	0.0039	0.0017

only about 20% of the conductivity of the corresponding hydrogen sulphate. It has been shown by cryoscopic measurements (Part IV, *loc. cit.*) that the hydrogen disulphate ion undergoes solvolysis, $\text{HS}_2\text{O}_7^- + \text{H}_2\text{SO}_4 = \text{H}_2\text{S}_2\text{O}_7 + \text{HSO}_4^-$, which, although far from being complete, is quite appreciable, amounting, for example, to approximately 10% in a 0.1*m*-solution at 10°. Thus a part of the observed conductivity of a hydrogen disulphate is due to the hydrogen sulphate produced by solvolysis, although this is to some extent counterbalanced by the repression of the autoprotolysis by the hydrogen sulphate.

It would not seem unreasonable to conclude on the basis of the above evidence that the mobility of the hydrogen disulphate ion is considerably smaller than that of the hydrogen sulphate ion. Moreover, it is extremely probable from theoretical considerations that, because of the special nature of the process by which they conduct, the mobilities of the hydrogen and hydrogen sulphate ions are much greater than those of any other ion (cf. Part X). A more detailed semi-quantitative analysis of the conductivities of solutions of hydrogen disulphate is given later (p. 969) after a value for the autoprotolysis constant at 25° has been deduced.

The Autoprotolysis of Sulphuric Acid.—We may assume then that from conductivity measurements we can only obtain information about that part of the self-dissociation which is due to autoprotolysis, as the conductivity of sulphuric acid is due almost entirely to the ions resulting from the autoprotolysis, the mobilities of the hydroxonium and hydrogen disulphate ions being much smaller than those of the hydrogen and hydrogen sulphate ions. In order to estimate a value for the autoprotolysis constant at 25°, it will be convenient first to consider the conductivities of dilute solutions of water at 10.4° in the light of the known autoprotolysis constant at that temperature. We will assume that water is fully ionised and that the conductivity of its solutions is due entirely to the hydrogen sulphate ion. The first of these assumptions cannot lead to much error at the very low concentrations we shall be considering, and the second is certainly justifiable as the mobility of the hydroxonium ion is of the same very small order of magnitude as that of the ammonium and alkali-metal ions (Part XII, *loc. cit.*).

From the autoprotolysis constant at 10.4°, $K_{\text{ap}} = 1.7 \times 10^{-4}$ (Parts I and IV, *loc. cit.*), we may calculate the total concentration of hydrogen and hydrogen sulphate ions for any concentration of added hydrogen sulphate (*e.g.*, water); some results of such calculations are given in Table 3.

In Fig. 2 specific conductivity values of water at 10.4° taken from Part IX are shown plotted against (a) the concentration of added water (*i.e.*, hydrogen sulphate) and (b) the total concentration of hydrogen and hydrogen sulphate ions, the values listed in col. 4 of Table 3 being used. It is justifiable to plot this second curve, since the mobility of the hydrogen ion is equal to that of the hydrogen sulphate ion. Curve (b) has been extrapolated

to zero concentration from 0.026*m*, which is the total concentration of hydrogen and hydrogen sulphate ions in pure sulphuric acid. Below 0.026*m* therefore the curve is purely hypothetical, since one can never have a total ionic concentration of less than 0.026*m* at 10.4°. From the probable shape of this extrapolated portion of the curve it would appear that the previously observed increase in the mobility of the hydrogen sulphate ion with decreasing concentration continues down to very low concentrations (cf. Part XII, *loc. cit.*). Curve (b) presumably also represents what the specific conductivity curve of water would be in the absence of any self-dissociation of the solvent. Thus this theoretical specific conductivity of water is always smaller than the actual observed conductivity but becomes less so as its concentration increases and the autoprotolysis is correspondingly repressed. From the theoretical specific conductivity curve

FIG. 2. Observed and theoretical conductivity curves.

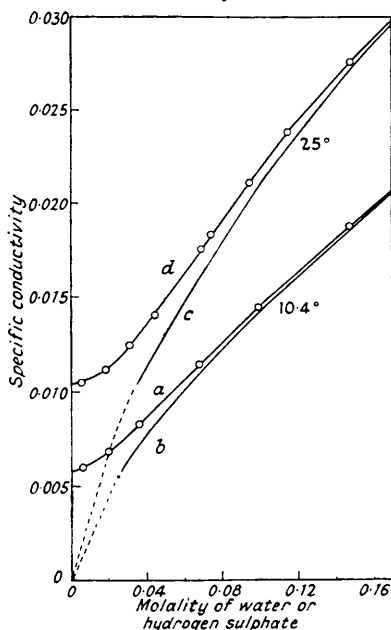


TABLE 3. Repression of the autoprotolysis at 10.4°.

Molalities				
Added	Total	Total		
HSO ₄ ⁻	HSO ₄ ⁻	H ₃ SO ₄ ⁺	H ₃ SO ₄ ⁺ + HSO ₄ ⁻	
0.0115	0.0200	0.0085	0.0285	
0.0358	0.0400	0.0042	0.0442	
0.0572	0.0600	0.0025	0.0625	
0.0779	0.0800	0.0021	0.0821	
0.0982	0.1000	0.0017	0.1017	
0.1489	0.1500	0.0011	0.1511	
0.1991	0.2000	0.0008	0.2008	
0.2493	0.2500	0.0007	0.2507	
0.2994	0.3000	0.0006	0.3006	
0.4997	0.5000	0.0003	0.5003	
0.9998	1.0000	0.0002	1.0002	

for water (b) we may calculate theoretical equivalent conductivity values for water by means of the expression $\Lambda = 1000\kappa/md$, where d is the density of the solution and m molality of the water, and these are given in Table 4, together with equivalent conductivities

TABLE 4. Observed and theoretical equivalent conductivities for water at 10.4°.

Molality of water	κ , obs.	κ , calc.	Λ , obs.	Λ , calc.	Molality of water	κ , obs.	κ , calc.	Λ , obs.	Λ , calc.
0.01	0.0061	0.0030	332	163	0.06	0.0106	0.0101	96	91
0.02	0.0068	0.0048	185	130	0.08	0.0126	0.0122	88	83
0.03	0.0077	0.0063	139	114	0.10	0.0145	0.0141	79	77
0.04	0.0087	0.0078	118	106	0.15	0.0191	0.0189	69	68
0.05	0.0097	0.0090	105	98					

(calculated from the observed specific conductivities) for water. The density d of the solution was taken to be the same as the density of sulphuric acid at 10°, *i.e.*, 1.841 (Domke and Bein, *Z. physikal. Chem.*, 1905, **43**, 125).

We are now in a position to consider further the extent of the autoprotolysis at 25°. It seems certain that the autoprotolysis will be an endothermic process, as is the autoprotolysis of water, and hence the increase in the specific conductivity of sulphuric acid

from 0.0058 at 10.4° to 0.0103 at 25° may be wholly or partly due to an increase in the extent of the autoprotolysis. This assumption enables us at once to set an upper and a lower limit to the extent of the autoprotolysis at 25°. The total concentration of the autoprotolytic ions cannot be less than 0.026*m*, which is their concentration at 10.4°, nor can it be greater than $0.026 \times 0.0103/0.0058 = 0.045m$ if it is assumed that all the increase in conductivity is due to the increased autoprotolysis. It is evident, however, from the increase in the slope of the conductivity-concentration curve for water with increasing temperature (see Fig. 2) that the mobility of the hydrogen sulphate ion does increase with temperature, and so the actual concentration of autoprotolytic ions must lie between the above two limits. We can obtain a fairly good estimate of the actual value by first calculating equivalent conductivities from the specific conductivities of water at 25° given in Part IX (*loc. cit.*), and then calculating the ratio $\Lambda_{25^\circ}/\Lambda_{10.4^\circ}$. The results of these calculations for various concentrations of water are given in Table 5. It may be seen that

TABLE 5. Observed equivalent conductivities for water at 25°.

Molality of water	κ , obs.	Λ , obs.	$\Lambda_{25^\circ}/\Lambda_{10.4^\circ}$	Molality of water	κ , obs.	Λ , obs.	$\Lambda_{25^\circ}/\Lambda_{10.4^\circ}$	Molality of water	κ , obs.	Λ , obs.	$\Lambda_{25^\circ}/\Lambda_{10.4^\circ}$
0.01	0.0105	562	1.70	0.04	0.0133	174	1.50	0.08	0.0192	128	1.47
0.02	0.0112	300	1.62	0.05	0.0148	158	1.50	0.10	0.0220	118	1.49
0.03	0.0121	216	1.55	0.06	0.0162	144	1.50	0.15	0.0283	101	1.46

the ratio $\Lambda_{25^\circ}/\Lambda_{10.4^\circ}$ is reasonably constant at 1.5, and it seems justifiable to assume therefore that the mobility of the hydrogen sulphate ion is raised 1.5 times when the temperature is increased from 10.4° to 25°. Since the mobility of the hydrogen ion is equal to that of the hydrogen sulphate ion at both temperatures, it follows that its mobility is also increased 1.5 times. The rather higher values of $\Lambda_{25^\circ}/\Lambda_{10.4^\circ}$ obtained at the very lowest concentrations are of little significance as the observed equivalent conductivity is not even approximately proportional to the mobility of the hydrogen sulphate ion at these concentrations. Now the equivalent conductivity of the hydrogen and hydrogen sulphate ions at 10.4° is given by $\Lambda = 1000\kappa/md$ where $\kappa = 0.0058 \text{ ohm}^{-1} \text{ cm.}^{-1}$, $m = 0.026$, and $d = 1.841$, and hence has the value 121. To a first approximation the equivalent conductivity Λ of the hydrogen and hydrogen sulphate ions at 25° will be $1.5 \times 121 = 182$, and hence the total molality of hydrogen and hydrogen sulphate, which is given by $m = 1000\kappa/\Lambda d$, where $\kappa = 0.01033$ and $d = 1.8269$, will be 0.031. Since, however, the molality of the autoprotolytic ions is greater at 25° than at 10.4°, we should have initially used the equivalent conductivity value at 10.4° corresponding to this higher concentration. Utilising the equivalent conductivity values listed in col. 5 of Table 5, we obtained a value of 0.034*m* for the total concentration of autoprotolytic ions at 25° by the method of successive approximations. Thus we have $[\text{H}_3\text{SO}_4^+] = [\text{HSO}_4^-] = 0.017m$, and hence $K_{ap}(25^\circ) = [\text{H}_3\text{SO}_4^+][\text{HSO}_4^-] = 2.9 \times 10^{-4}$. Using this value of the autoprotolysis constant, one can calculate as before the extent to which the autoprotolysis is repressed by addition of water or any other hydrogen sulphate and hence obtain the total concentration of hydrogen and hydrogen sulphate ions for any concentration of added hydrogen sulphate. The results of the calculations are shown in Table 6. Utilising the values listed in col. 4 of this table, we can also plot a theoretical conductivity curve for water at 25°, *i.e.*, a hypothetical curve for the conductivity of water in the absence of any autoprotolysis of the solvent. This is shown in Fig. 2 (curve *c*) together with the observed conductivity curve (*d*) for water at 25°.

From the change in K_{ap} with temperature it can be calculated that the heat of autoprotolysis is approximately 2000 cal. In view of possible inaccuracies in the K_{ap} values and the fact that K_{ap} was measured at only two temperatures, this value for the heat of autoprotolysis can only be regarded as very approximate. It does, however, definitely seem to be much smaller than the heat of autoprotolysis of water, which is approximately 14,000 cal.

Conductivity of the Hydrogen Disulphate Ion.—Having now obtained a value for the autoprotolysis constant at 25° we can give a more quantitative discussion of the

conductivities of ammonium and potassium hydrogen disulphate solutions that we considered earlier. We have no exact knowledge of the solvolysis constant of the hydrogen disulphate ion at 25°, but remembering that $K_b(\text{HS}_2\text{O}_7^-) = K_{ap}/K_a(\text{HS}_2\text{O}_7^-) = 0.008$ at 10° (Part IV) and that $K_{ap}(25^\circ)/K_{ap}(10.4^\circ) = 2.9/1.7 = 1.7$, it would not seem

TABLE 6. *Repression of the autoprotolysis at 25°.*

Molalities				Molalities			
Added	Total		Total	Added	Total		Total
HSO_4^-	HSO_4^-	H_3SO_4^+	$\text{H}_3\text{SO}_4^+ + \text{HSO}_4^-$	HSO_4^-	HSO_4^-	H_3SO_4^+	$\text{H}_3\text{SO}_4^+ + \text{HSO}_4^-$
0.0055	0.0200	0.0145	0.0345	0.1986	0.2000	0.0015	0.2015
0.0328	0.0400	0.0072	0.0472	0.2488	0.2500	0.0012	0.2512
0.0552	0.0600	0.0048	0.0648	0.2990	0.3000	0.0010	0.3010
0.0764	0.0800	0.0036	0.0836	0.4996	0.5000	0.0006	0.5006
0.0971	0.1000	0.0029	0.1029	0.9997	1.0000	0.0003	1.0003
0.1481	0.1500	0.0019	0.1519				

unreasonable to assume that at 25° $K_b(\text{HS}_2\text{O}_7^-) = 1.7 \times 0.008 = 0.014$. Utilising this value for the solvolysis constant and the value for the autoprotolysis constant at 25°, we can calculate the total concentration of hydrogen and hydrogen sulphate ions in any hydrogen disulphate solution, then from curve (c) in Fig. 2 we can read off the conductivity due to these ions and hence, by subtraction, obtain the conductivity due to the cation and the hydrogen disulphate ion only. The values thus obtained are given in the last column of Table 2. They are of the order of 10% of the conductivity of the corresponding hydrogen sulphate; hence, it being assumed that the mobilities of the cation and the hydrogen disulphate ion are roughly equal, the hydrogen disulphate ion has a conductivity which is approximately 5% of that of the hydrogen sulphate ion. We may recall for comparison that the conductivity of the potassium ion is approximately 3% of that of the hydrogen sulphate ion (Part X, *loc. cit.*).

The general shape of the conductivity curves given in Fig. 1 may be interpreted in the following way. The initial effect of added sulphate is to remove hydrogen ions, $\text{SO}_4^{--} + 2\text{H}_3\text{SO}_4^+ = 3\text{H}_2\text{SO}_4$, thus replacing highly conducting hydrogen ions by poorly conducting potassium or ammonium ions. There will simultaneously be an increase in the concentration of the hydrogen disulphate ion as potassium and ammonium hydrogen disulphate are fully ionised, whereas disulphuric acid is not, but since the hydrogen disulphate ion is rather poorly conducting, the increase in its concentration will have little effect on the conductivity, which will therefore initially fall. The continued removal of hydrogen ion allows the autoprotolysis of the solvent to increase gradually until it makes a quite large contribution to the conductivity, and this, together with the increasing concentration of hydrogen sulphate ion produced by solvolysis of the hydrogen disulphate, eventually causes the conductivity to pass through a minimum and then increase. After the vertical, neutral line has been reached, addition of further sulphate merely causes formation of the corresponding amount of hydrogen sulphate and the conductivity increases rapidly.

Experimental.—The apparatus used for the conductivity measurements and the preparation and purification of the materials used have all been described in Parts IX, X, and XII (*loc. cit.*).