842. *Standard Potential of the* In|In^{3+} *Electrode.*

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The standard potential of the indium–indic couple in solutions of indium (indic) chloride and sulphate has been determined between 0° and 60° from cells without transference, by using silver–silver chloride or mercury–mercurous sulphate reference electrodes. At 25° the values found for $E°(In^{3+}|In)$ were $-338{\cdot}2$ and $-341{\cdot}0$ mv, respectively, and the temperature coefficient was $+40$ μv deg.$^{-1}$. The values from the two cells at each temperture differ by about 2 mv, but those from the sulphate cell are thought to be less reliable because of doubts about the extrapolation of e.m.f. results owing to the hydrolysis of mercurous sulphate.

Very few e.m.f. studies have been made of multivalent electrolyte solutions by using cells without transference, owing to the lack of suitable reversible electrodes. Most of our knowledge about such solutions comes from measurements of their water activity, *i.e.*, from isopiestic and freezing-point measurements. Even when suitable reversible electrodes are available, there are difficulties in extrapolating the e.m.f. results to infinite dilution to obtain the standard e.m.f. of the cell, knowledge of which is required before activity coefficients can be evaluated.

Of the metals showing a valency of three, only gallium and indium give stable reversible potentials with their tervalent salts in aqueous solution. A number of workers [1-4] have determined the standard (reduction) potential of the In|In^{3+} couple [$E°(In^{3+}|In)$] and values reported are $-0{\cdot}336$, $-0{\cdot}340$, $-0{\cdot}335_8$ v, all at 25°, and $-0{\cdot}334$ v at 20°.

References to work before 1930 are given by Hakomori [1] who studied the cell:

$$In|InCl_3(m_1),HCl(m_2)|AgCl|Ag \qquad (Ia)$$

at 25° with $m_1 = 0{\cdot}015$—$0{\cdot}027$ and $m_2 = 0{\cdot}02$ (m = molality). The hydrochloric acid was added with the intention of preventing hydrolysis of indium chloride.

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[1] Hakomori, *J. Amer. Chem. Soc.*, 1930, **52**, 2372.
[2] Hattox and deVries, *J. Amer. Chem. Soc.*, 1936, **58**, 2126.
[3] Kangro and Weingartner, *Z. Elektrochem.*, 1954, **58**, 505.
[4] Sundén, *Z. Elektrochem.*, 1953, **57**, 100.

Hattox and deVries [2] studied the cell

$$\mathrm{In}|\mathrm{In_2(SO_4)_3}(m_3)|\mathrm{Hg_2SO_4}|\mathrm{Hg} \qquad \text{(IIa)}$$

at 0°, 15°, 25°, and 35°; and Lietzke and Stoughton [5] extended the concentration and temperature range but employed the silver–silver sulphate instead of the mercury–mercurous sulphate reference electrode:

$$\mathrm{In}|\mathrm{In_2(SO_4)_3}|\mathrm{Ag_2SO_4}|\mathrm{Ag} \qquad \text{(IIb)}$$

However, recalculation of these data from the cells without liquid junction revealed some doubts about their interpretation. Values for the standard electrode potential have also been derived by Sundén [4] and Kangro and Weingartner [3] from measurements in indium perchlorate and sulphate solutions, respectively, using calomel reference electrodes with liquid junction.

New measurements over a temperature range of 0—60° have been made on cell (Ia) with $m_2 = 0$ (cell Ib):

$$\mathrm{In}|\mathrm{InCl_3}(m_1)|\mathrm{AgCl}|\mathrm{Ag} \qquad \text{(Ib)}$$

and $m_1 = 0{\cdot}002$—$0{\cdot}02$ and on cell (IIa) with $m_3 = 0{\cdot}01$—$0{\cdot}2$.

Theory.—For cell (Ib) the spontaneous cell reaction is

$$\mathrm{In + 3AgCl \longrightarrow InCl_3 + 3Ag}$$

and the e.m.f. (E) is given by

$$E = E^\circ - \tfrac{1}{3}k \log a_1,$$

where a_1 is the activity of indium chloride in solution of molality m_1, E° is the standard e.m.f. of the cell, and $k = \boldsymbol{R}T\ln 10/\boldsymbol{F}$. a_1 is related to $\gamma_\pm$, the mean stoicheiometric activity coefficient of indium chloride, by

$$a_1 = 27m_1{}^4\gamma_\pm{}^4.$$

Thus
$$E = E^\circ - \tfrac{4}{3}k \log (27)^{\frac{1}{4}}m_1\,\gamma_\pm.$$

Introducing the two-parameter form of the Debye–Hückel equation on the assumption that the electrolyte is completely dissociated (B will take care of a small amount of association), we have

$$\log \gamma_\pm = -\frac{Az_+|z_-|\mathrm{I}^{\frac{1}{2}}}{1 + \rho I^{\frac{1}{2}}} + BI, \qquad (1)$$

where A is the Debye–Hückel constant, I is the ionic strength, z_+, z_- are the valencies of the ions, and ρ and B are the parameters. Then defining

$$E^{\circ\prime} \equiv E + \tfrac{4}{3}k \log (27)^{\frac{1}{4}}m_1 - 4k\,\frac{AI^{\frac{1}{2}}}{1 + \rho I^{\frac{1}{2}}},$$

we have
$$E^{\circ\prime} = E^\circ - \tfrac{4}{3}kBI. \qquad (2)$$

If the assumption is correct, a reasonable choice of the ion-size parameter ρ should yield, in the range of ionic strength studied, a plot of $E^{\circ\prime}$ against I which is a straight line, of slope $\tfrac{4}{3}kB$ and intercept E°.

For the cell (IIa), the spontaneous cell reaction is:

$$\mathrm{2In + 3Hg_2SO_4 \longrightarrow In_2(SO_4)_3 + 6Hg}$$

and the e.m.f. (E) of cell (IIa or b) is:

$$E = E^\circ - \tfrac{1}{6}k \log a_3,$$

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[5] Lietzke and Stoughton, *J. Amer. Chem. Soc.*, 1956, **78**, 4520.

where a_3 is the activity of the indium sulphate in solution of molality m_3.

$$a_3 = 108 m_3^5 \gamma_\pm^5$$

where $\gamma_\pm$ is the mean ionic activity coefficient of indium sulphate.

Thus
$$E = E^\circ - \tfrac{5}{6}k \log (108)^{1/5} m_3 \gamma_\pm$$

and, after introduction of equation (1),

$$E^{\circ\prime} \equiv E + \tfrac{5}{6}k \log (108)^{1/5} m_3 - 6k \cdot \frac{AI^{\frac{1}{2}}}{1 + \rho I^{\frac{1}{2}}} = E^\circ - \tfrac{5}{6}kBI. \qquad (3)$$

Again, if the assumption of complete or almost complete dissociation is correct, $E^{\circ\prime}$ should be a linear function of I for a reasonable choice of ρ.

The values of k used were those of Robinson and Stokes[6] and of A were from the compilation of Manov, Bates, Hamer, and Acree.[7]

EXPERIMENTAL

Indium Chloride.—A solution (Mining and Chemical Products Ltd.), prepared from $\not<$99·95%-pure metal and containing 400 g. of indium chloride per l., was filtered to remove solid particles and diluted with triple distilled water to give a stock solution for analysis.

Indium Sulphate.—A solution (New Metals and Chemicals Ltd) containing 430 g./l. was filtered and diluted to give a 2m-stock solution.

Hydrochloric Acid.—" AnalaR " acid was treated with chlorine and boiled until free from other halogens. 0·1m-Acid prepared by dilution of a constant-boiling distillate[8] was used as a primary standard for chloride analysis and in the preparation of silver–silver chloride electrodes.

Analysis.—The stock solution of indium chloride was analysed by Volhard's method.[9]

Indium sulphate was analysed by direct titration with 0·05M-ethylenediaminetetra-acetic acid (EDTA) with Xylenol Orange as indicator. The method was checked by analysis of the indium chloride stock solution. Details of the method, which was based on the procedure of Kinnunen and Wennerstrand,[10] were as follows: The pH of the solution containing indium was adjusted to 3—5 by means of aqueous ammonia and acetic acid. 1—2 drops of a 0·2% aqueous solution of Xylenol Orange (Hopkin and Williams Ltd.) was added and the solution titrated with EDTA solution (which had been standardised with zinc chloride solution[11] by the same method), until the colour changed from wine-red to yellow. Ion-exchange water was used for all work with EDTA.

Electrodes.—*Indium metal electrodes.* Indium metal (Consolidated Mining and Smelting Co. of Canada) in two grades of purity (99·99 and 99·999%) was supplied in the form of cast blocks. Rod of 5 mm. diameter was prepared from these by cold drawing. Short lengths of this rod were sealed with Araldite into Pyrex tubing mounted on B19 cones, electrical connection being made by soldering a copper wire to the indium within the tubing. These electrodes gave bias potentials of several mv and they were therefore electropolished in alcoholic nitric acid at 0° according to the directions of Goss and Vernon.[12] They were washed in absolute alcohol and dried in a current of hot air before storage in indium-containing solutions. No difference in electrochemical behaviour was detected between the standard and the high-purity grades of indium.

Electrodeposited indium electrodes. These were prepared by deposition of indium from indium chloride solution[2] on to a spiral of platinum wire sealed into the end of a Pyrex glass tube mounted on a B19 cone. The bias potentials of these electrodes were found to increase with time, but electrodes prepared from a sulphate bath [indium sulphate (30 g./l.), sodium gluconate (45 g./l.), sodium sulphate (10 g./l.); pH adjusted to 2·0—2·4 with ammonia] were found not to

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[6] Robinson and Stokes, " Electrolyte Solutions," Butterworths, London, 2nd edn., 1959, p. 469.

[7] Manov, Bates, Hamer, and Acree, *J. Amer. Chem. Soc.*, 1943, **65**, 1765.

[8] Foulk and Hollingsworth, *J. Amer. Chem. Soc.*, 1923, **45**, 1220.

[9] Vogel, " A Textbook of Quantitative Inorganic Analysis," Longmans, London, 1945, pp. 318—322.

[10] Kinnunen and Wennerstrand, *Chemist-Analyst*, 1957, **46**, 92.

[11] Flaschka, " E.D.T.A. Titrations," Pergamon Press, London, 1959, p. 63.

[12] Goss and Vernon, *Proc. Phys. Soc.*, 1952, **65**, *B*, 905.

suffer from this defect. A current density of 0·03—0·3 A cm.$^{-2}$ was used for the deposition. The electrodes were washed well with distilled water before storage in indium-containing solutions.

Silver–silver chloride electrodes of the thermal-electrolytic type were prepared according to Bates's directions.[13] Their bias potentials were reduced by heating them in water at 50° for 2 hr.,[14] and the bias potentials of those selected were $<$0·04 mv.

Mercurous sulphate was prepared anodically.[15] The form of the mercury–mercurous sulphate electrodes and their preparation was identical with that of Beck, Dobson, and Wynne-Jones.[16] The electrodes were stored in 0·1m-indium sulphate and the bias potentials of those chosen for use were $<$0·08 mv.

Experimental Procedure.—The electrode vessel was of the multicompartment type with B19 sockets and allowed up to eight indium electrodes to be compared. These eight compartments were separated by a greased tap (opened only when measurements were being made) from two compartments for appropriate reference electrodes.

E.m.f. measurements were made to 0·01 mv on a Pye vernier potentiometer and a Tinsley galvanometer of sensitivity 163 mm. μA^{-1}. Water thermostat-baths at 0°, 15°, 25°, 35°, and 45° ($\pm$0·05°) were controlled by mercury–alcohol thermoregulators, and the tanks at the two lowest temperatures included copper cooling-coils through which ethylene glycol from a commercial refrigerator was circulated. For 60° a Townson and Mercer oil-filled thermostat bath was used. Measurements were made in the order 25°, 35°, 45°, 60°, 25°, 15°, 0°.

Results and Discussion

Cell (Ib).—The electrode vessel contained six indium rod electrodes, two electrodeposited electrodes, and two silver–silver chloride reference electrodes. The bias potential of the reference electrodes was checked between measurements at different concentrations and remained constant throughout the series. The cells took some hours to reach steady e.m.f. values and were usually left overnight, whereafter they were found to remain constant for some hours. The mean deviation of the equilibrium values in all solutions was $\pm$0·3 mv at all temperatures, except 35° where it was somewhat greater. The electrodeposited electrodes tended to the positive side of the distribution. The mean values for all electrodes are shown in Table 1 for temperatures of 15°, 25°, 35°, 45° and 60°.

TABLE 1.

Mean values for the observed and standard e.m.f. (mv) of cell (Ib):
In|InCl$_3$(m_1)|AgCl|Ag at various temperatures.

10^3m_1 (mole kg.$^{-1}$)	15°	25°	35°	45°	60°
1·723	762·6	762·0	761·9	761·8	760·4
2·450	752·9	752·4	751·7	751·6	749·5
4·737	735·3	734·0	732·9	732·3	729·6
5·643	731·6	730·4	729·1	728·5	725·6
8·380	722·5	721·2	719·1	718·5	715·4
11·06	716·6	715·3	712·9	712·3	708·9
E°	568·1	560·8	553·6	546·6	534·6

The results were analysed by means of equation (2), and the value of $\rho = 1$ in equation (1). Straight-line plots (Fig. 1) were obtained, from the intercepts of which E° values given in Table 1 were evaluated. Smoothed values of $\gamma_\pm$ at round molalities are given in Table 2. The value $\rho = 1{\cdot}5$ in equation (1) was tried for the 25° results and also gave a straight line with an E° value 0·5 mv higher than that from the analysis with $\rho = 1$.

Hakomori [1] used the limiting law [$\rho = 0$, $B = 0$ in equation (1)] to extrapolate his four measurements, at $I = 0{\cdot}03$—0·18, to zero indium molality and to correct for the activity

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[13] Bates, " Electrometric pH Determination," Wiley, New York, 1954, pp. 205—207.

[14] Ashby, Crook, and Datta, *Biochem. J.*, 1954, **56**, 190.

[15] Hulett, *Phys. Rev.*, 1911, **32**, 257.

[16] Beck, Dobson, and Wynne-Jones, *Trans. Faraday Soc.*, 1960, **56**, 1172.

TABLE 2.

Smoothed stoicheiometric activity coefficients of $InCl_3$.

m (mole kg.$^{-1}$)	15°	25°	35°	45°	60°
0·002	0·699	0·694	0·689	0·684	0·676
0·004	0·607	0·601	0·596	0·590	0·581
0·006	0·546	0·540	0·534	0·528	0·519
0·008	0·500	0·493	0·488	0·483	0·472
0·010	0·463	0·456	0·451	0·444	0·435

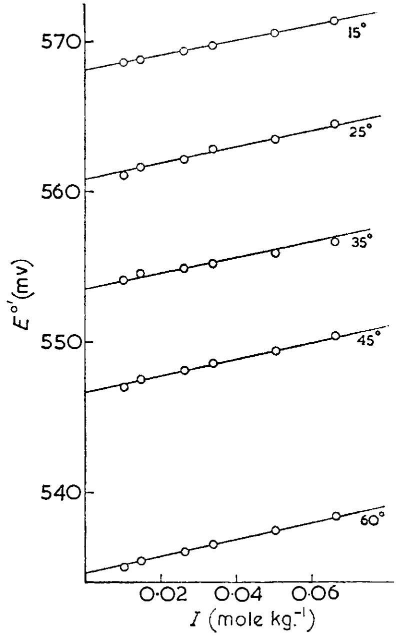

FIG. 1. Extrapolation of results for cell (Ib). Diameter of circles, 0·5 mv.

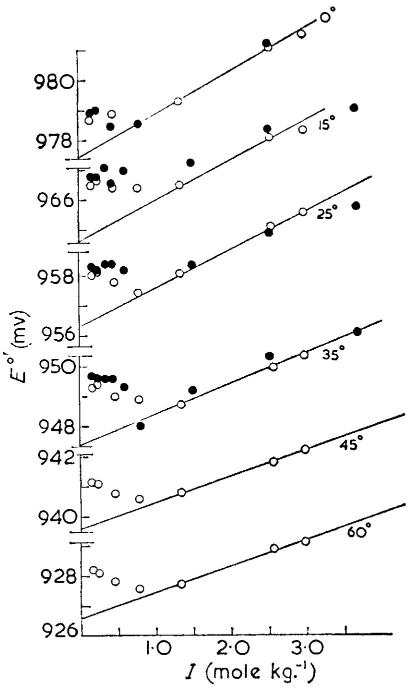

FIG. 2. Extrapolation of results for cell (IIa), with $\rho = 1·4$ (eqn. 2). Diameter of circles, 0·25 mv.

(○) Present work. (●) Hattox and deVries.[2]

coefficient of a trace of indium chloride in 0·02m-hydrochloric acid in order to obtain E°. Both the use of the limiting law and the extrapolation itself are doubtful. The discrepancy of 3 mv between Hakomori's value of E° at 25° and that from the present work is therefore not surprising. There is no adequate theory of mixed-electrolyte solutions of different valency type which permits a sound evaluation of E° from Hakomori's results for cell (Ia). The addition of hydrochloric acid to the cell electrolyte will suppress hydrolysis of indium chloride, but it is not essential to the reversible functioning of the cell and makes accurate evaluation of the standard e.m.f. impossible.

Cell (IIa).—The metal-rod indium electrodes were repolished before use in indium sulphate solutions, and new electrodeposited electrodes were prepared. Again a total of eight indium electrodes was used and the mean deviation of the results at all temperatures did not exceed ±0·2 mv. Cell (IIa) appeared to be more reproducible than cell (Ib) and came to equilibrium quicker; six hours were required at the lower temperatures but only two hours at the higher. Results are given in Table 3. Plots of $E^{\circ\prime}$ (equation 3) against

TABLE 3.

Mean values for the observed and standard e.m.f. (mv) of cell IIa:
$In|In_2(SO)_3(m_3)|Hg_2SO_4|Hg$ at various temperatures.

10^3m_3 (mole kg.$^{-1}$)	0°	15°	25°	35°	45°	60°
10·12	1083·8	1078·1	1074·3	1070·3	1066·8	1061·2
16·02	1080·1	1074·3	1070·4	1066·4	1062·7	1057·1
30·21	1075·0	1068·9	1064·9	1060·9	1057·2	1051·7
52·41	1072·1	1064·5	1060·1	1056·3	1052·6	1047·2
89·70	1066·7	1060·2	1056·2	1051·6	1048·4	1042·9
170·2	1062·8	1055·8	1051·6	1046·8	1043·4	1038·0
198·5	1061·7	1054·6	1050·8	1045·6	1042·2	1036·8
$E°$	977·4	964·7	956·4	947·4	939·6	926·6

I were constructed for the results at 25° with different ρ values in the range 1·0—1·5. For $\rho = 1{\cdot}0$ a curve concave upwards with a negative gradient was obtained and for $\rho = 1{\cdot}5$ a straight line with a positive slope. However, the extrapolated value from the latter plot gave a value for the standard potential of the indium–indic couple about 5 mv higher than that obtained by adding the standard potential of the mercury–mercurous sulphate electrode to that determined from cell (Ib). The results of Hattox and deVries [2] (corrected to abs. v), analysed similarly, were found to be in close accordance. These authors used Hitchcock's method [17] of analysis [$\rho = 0$ in equation (1)], but a recalculation of both sets of results by this method produced a curve very difficult to extrapolate with certainty. Choice of $\rho = 1{\cdot}3$ and 1·4 (Fig. 2) gave much more sensitive plots (in that the range of $E°'$ values was only a few mv), showing minima at molalities of 0·04—0·08.

The literature is contradictory about the use of the mercury–mercurous sulphate electrode in weakly acid solutions and solutions of low sulphate-ion concentration and the risk of hydrolysis and basic salt formation. Ives and Smith [18] consider the risk of hydrolysis has been overemphasised. Akerlöf [19] stated that the electrode could not be used in neutral solution and studied salt solutions 0·001m in acid. Harned and Hamer [20] apparently used the electrode successfully in sodium sulphate solution but no experimental details were given. Lietzke and Stoughton [21] compared the potentials of the mercury–mercurous sulphate and the silver–silver sulphate electrodes in sulphuric acid with a calculated thermochemical value and concluded that hydrolysis caused the e.m.f. of the cell to be too high below 0·05m. For this reason it seems justifiable to neglect the e.m.f. values for indium sulphate molalities below 0·07 as being due to hydrolysis. By using $\rho = 1{\cdot}4$, $E°$ values at different temperatures were obtained and are given in Table 3. Smoothed values for the activity coefficient of indium sulphate are given in Table 4. These values are reasonably consistent with those given by Hattox and deVries [2] at molalities above 0·1.

TABLE 4.

Smoothed stoicheiometric activity coefficients of $In_2(SO_4)_3$.

m (mole kg.$^{-1}$)	0°	15°	25°	35°	45°	60°
0·01	0·182	0·175	0·169	0·164	0·158	0·149
0·05	0·0741	0·0701	0·0672	0·0642	0·0611	0·0558
0·10	0·0427	0·0403	0·0385	0·0378	0·0348	0·0313
0·15	0·0324	0·0306	0·0293	0·0282	0·0266	0·0238
0·20	0·0265	0·0251	0·0241	0·0232	0·0221	0·0196

Lietzke and Stoughton [5] studied cell (IIb), obtaining curves which they could not

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[17] Hitchcock, *J. Amer. Chem. Soc.*, 1928, **50**, 2076.

[18] Ives and Smith, in " Reference Electrodes," ed. Ives and Janz, Academic Press, New York, 1961, p. 393.

[19] Akerlöf, *J. Amer. Chem. Soc.*, 1926, **48**, 1160.

[20] Harned and Hamer, *J. Amer. Chem. Soc.*, 1935, **57**, 33.

[21] Lietzke and Stoughton, *J. Amer. Chem. Soc.*, 1953, **75**, 5226.

extrapolate to obtain $E°$. They assumed the dissociation of indium sulphate as a 2 : 1 or 1 : 1 electrolyte:

$$In^{3+} + SO_4^{2-} \rightleftharpoons InSO_4^{+}$$
$$In^{3+} + 2SO_4^{2-} \rightleftharpoons In(SO_4)_2^{-}$$

(for which there is some support from Raman studies [22]) instead of a 3 : 1 electrolyte, but with no greater success. The same is true of the present results.

Silver sulphate is appreciably more soluble ($pK_{sp} = 4{\cdot}8$) than mercurous sulphate ($pK_{sp} = 6{\cdot}17$).[23] Although they measured the solubility of silver sulphate in indium sulphate at 25° Lietzke and Stoughton apparently failed to apply a correction for the increased sulphate ion concentration at the silver–silver sulphate electrode. Applying this correction to the results interpolated graphically at 25° and using their solubility data gives a straight line ($\rho = 1{\cdot}4$). Lietzke and Stoughton [21] found that the silver–silver sulphate electrode is 42·3 mv more positive than the mercury–mercurous sulphate electrode. Subtracting this value from the corrected e.m.f. data should bring it exactly into line with that from cell (IIa), but the original 20 mv discrepancy is reduced only by about 10 mv. The remaining difference (Fig. 3) could be due to the liquid junction potential produced for which it is not possible to apply a correction.

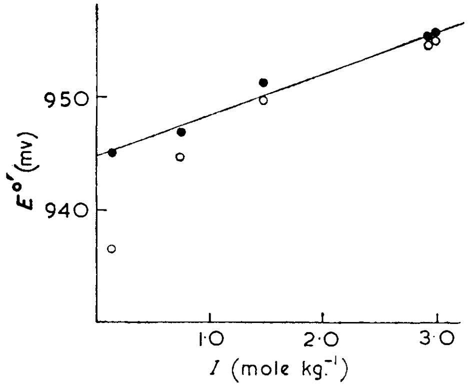

FIG. 3. Recalculation of results for cell (IIb) (Lietzke and Stoughton [5]) at 25°.
(○) Uncorrected. (●) With correction for additional sulphate molality.

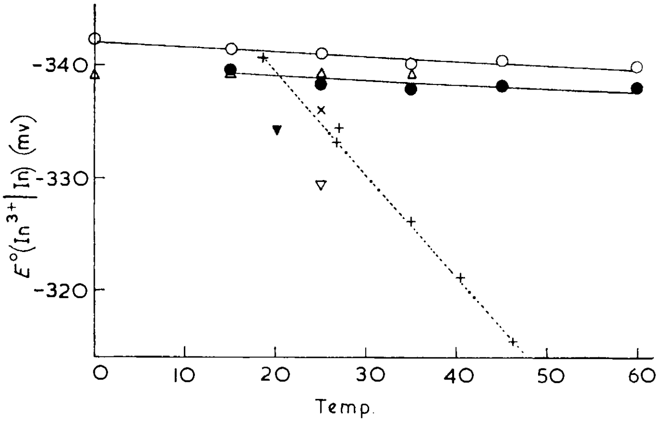

FIG. 4. Standard potentials of the indium–indic electrode. (○) Cell (IIa). (●) Cell (Ib). (Δ) Hattox and deVries.[2] (×) Hakomori.[1] (+) Kangro and Weingartner.[3] (▼) Sundén.[4] (▽) Lietzke and Stoughton [5] (recalculated).

Conclusions.—In Fig. 4 and Table 5 are shown values of $E°$ for the indium-indic couple obtained previously and those derived from cells (Ib) and (IIa) by using appropriate values for the standard potentials of the reference electrodes.[24,20] There is a constant

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[22] Hester, Plane, and Walrafen, *J. Chem. Phys.*, 1963, **38**, 249.

[23] " Stability Constants. Part II. Inorganic Ligands," *Chem. Soc. Special Publ.* No. 7, 1958.

[24] Bates and Bower, *J. Res. Nat. Bur. Stand.*, 1954, **53**, 283.

TABLE 5.

Values (mv) for $-E°(\mathrm{In}^{3+}|\mathrm{In})$ at various temperatures.

	0°	15°	25°	35°	45°	60°
From cell (Ib)	—	339·5	338·2	337·9	338·3	338·1
From cell (IIa)	342·2	341·1	340·2	340·2	340·4	339·8

2 mv difference between the results from cells (Ib) and (IIa). This difference could be due to inaccuracy of the extrapolation procedure for cell (IIa), which uses only three or four points, with the arbitrary neglect of values where hydrolysis is suspected, or the use of Harned and Hamar's results[20] which are the only ones available for the standard potential of the mercury–mercurous sulphate electrode at temperatures other than 25°. At this temperature, Beck, Dobson, and Wynne-Jones[16] found a value different by 0·46 mv and have demonstrated that the previous work[20] is inconsistent with vapour-pressure data. The standard electrode potential varies almost linearly with temperature and the temperature coefficient is 40 μv deg.$^{-1}$. The accuracy of the results obtained with either cell does not justify the calculation of partial molal enthalpies.

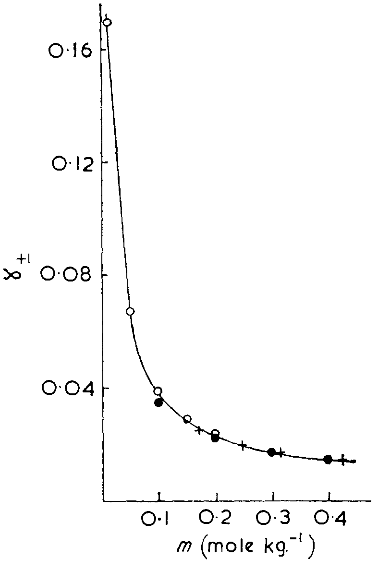

FIG. 5. Comparison of the activity coefficients of indium and aluminium sulphates.

$\mathrm{In_2(SO_4)_3}$, (○) present work, (+) Hattox and deVries[2] ($m > 0·16$ only). $\mathrm{Al_2(SO_4)_3}$, (●) Robinson.[25]

The values given by Kangro and Weingartner[3] were derived from measurements in a single solution, with temperature-invariant activity coefficients interpolated from Hattox and deVries's values.[2] Therein may lie the reason for the low values at the higher temperatures, resulting in an anomalously high temperature coefficient.

It is worth noting that the stoicheiometric activity coefficient of indium sulphate is closely similar (Fig. 5) to that of aluminium sulphate[25] which may indicate that the salts are similarly dissociated. The activity coefficient of indium chloride is somewhat lower than those of lanthanum and the rare-earth chlorides.[26]

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[25] Ref. 6, p. 503.

[26] Spedding and Atkinson, in "The Structure of Electrolytic Solutions," ed. Hamer, Wiley, New York, 1959, p. 319.