# NOTES.

## *The Mechanism of the Reaction between Aluminium, Iodine, Ethyl Alcohol, and Water. The Preparation of Ethyl Iodide.* By A. O. Jones and G. Melling Green.

Further work has thrown much light on the chemistry of the above reaction (J., 1926, 2760). What was thought to be a series of reactions involving the formation of an organo-aluminium complex has shown itself to be one proceeding according to the following scheme :

$$\text{Aluminium} + \text{Iodine} \longrightarrow \text{Aluminium iodide.}$$

$$\text{Aluminium iodide} \xrightleftharpoons{\text{Hydrolysis}} \text{Hydrogen iodide} + Al(OH)_3.$$

$$\text{Hydrogen iodide} + \left\{\begin{array}{l}\text{Aluminium}\\ \\ \text{Ethyl alcohol}\end{array}\right. \longrightarrow \left\{\begin{array}{l}\text{Aluminium iodide}\\ \text{Ethyl iodide}\\ \text{Hydrogen.}\end{array}\right.$$

Thus the main products of the reaction are ethyl iodide, aluminium iodide, and hydrogen, the hydrogen iodide produced by the hydrolysis of the aluminium iodide distributing itself between the ethyl alcohol and the aluminium in a ratio which depends upon the concentration of the water in the alcohol used.—Technical College, Huddersfield. [*Received, January 7th,* 1927.]

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## *New Fluorides of Selenium.* By Edmund Brydges Rudhall Prideaux and Charles Beresford Cox.

*Selenium oxyfluoride* has been prepared by the action of dry silver fluoride on selenium oxychloride in a platinum bottle, with subsequent distillation and condensation in a platinum condenser (water-cooled). It is a colourless, fuming liquid, with an ozone-like odour similar to that of some organic compounds which have been treated with fluorine. It was analysed after decomposition with water, both by titration with two indicators (J., 1926, 170) and by gravimetric methods. Three separate analyses on quantities of about 1 g. gave the following results, the weights of selenium and fluorine found being referred to the weights of compound taken in calculating the percentages: Se, 59·1, 59·3, 59·9 (as $Ag_2SeO_3$); F, 28·3, 28·2, 28·3 (volumetric), 28·3 (as $CaF_2$). Hence Se (mean), 59·4; F (mean), 28·3 ($SeOF_2$ requires Se, 59·5; F, 28·5%).

*Selenium tetrafluoride* has been prepared by the same method from silver fluoride and selenium tetrachloride. The first reaction is violent and distillation of the product gives a colourless, fuming liquid having much the same appearance and smell as the oxyfluoride. The first portion of the distillate, collected separately, was deep red, and possibly may contain a lower fluoride, $Se_2F_2$, formed by the reducing action of the silver present in the silver fluoride. 0·998 G. of this liquid was analysed as described above: Se, 51·8 (as $Ag_2SeO_3$), 50·6 (as Se); F, 45·5 (volumetric), 46·7 (volumetric HF with gravimetric Se), 46·9 (as $CaF_2$). Hence Se (mean), 51·2; F (mean), 46·3 ($SeF_4$ requires Se, 51·0; F, 49·0%).

The properties and reactions of these compounds are being further examined.—UNIVERSITY COLLEGE, NOTTINGHAM. [*Received, February 26th*, 1927.]

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### *Studies of Precipitated Solids, especially of Strontium and Calcium Sulphates.* By PETR P. VON WEIMARN.

THE statement by Bertram Lambert and his collaborators W. Hume-Rothery and J. Schaffer (J., 1926, 2637, 2648) that, previous to their work, no "detailed or systematic study of the effects of the conditions of precipitation upon the type, nature and size of the precipitated particles of strontium sulphate and of calcium sulphate had been carried out" is incorrect. As far back as 1905 I had begun a systematic study of the effects of the conditions of precipitation upon the type, nature, and size of the precipitated particles of different salts. My paper published in 1906 (*J. Russ. Phys. Chem. Soc.*, 1906, **38**, 264) included strontium and calcium sulphates amongst the 62 salts investigated, and announced the law that *the mean size of the precipitated crystals decreases with increasing concentration of the reacting solutions*, provided, and this is very important, that the crystallisation process is complete and the course of the process is not disturbed by the influence of variables foreign to the normal reaction. The graphic representation of this law is a curve without a maximum. Curves with maxima are obtained only if the crystallisation process is not complete, if aggregates of crystals are mistaken for individual crystals, or if variables foreign to the normal precipitation process exert an influence.

From the beginning of 1906 my results were published periodically (*J. Russ. Phys. Chem. Soc.*, 1906, **38**, 624; *Annals Mining Inst. S. Petersburg*, 1908, **1**, 239; *Jap. J. Chem.*, 1926, **3**, 20; *Koll.-Chem.*

*Beih.*, 1923, **18**, 44, and succeeding years; *Chem. Reviews*, 1925, **2**, 217). Particular mention, with photomicrographs, of the precipitation of strontium and calcium sulphates occurs in my book "Kolloides und Kristalloides Lösen und Niederschlagen," Vol. II, pp. 661—680 and Figs. 90, 91 (Kioto, 1921), and in the second edition of Volume I of this book, "Die Allgemeinheit der Kolloidzustandes," Figs. 92, 93 (Dresden, 1925).

The investigations of Lambert and his collaborators of precipitates of calcium and strontium sulphates are important as independent confirmation of previous work, and are in some respects more detailed. I believe, however, that in other points my investigations (*Koll.-Chem. Beih.*, 1923, **17**, 27) were more extensive, as they included wider limits of concentration and alcoholic as well as aqueous solutions, and colloidal solutions of both salts were obtained.

Subsequently, between 1919 and 1926, detailed investigations of precipitated solids were carried out by Sven Odén (Svedberg, "Formation of Colloids," London, 1921, pp. 94—100, Fig. 20; *Z. Koll.*, 1926, **40**, 92), confirming my results, the latter paper including the examination of strontium sulphate.

As far as I know the literature on the subject, Lambert and his collaborators were the first to study by crystallographic measurement the influence of anions and kations on the precipitation of calcium and strontium sulphates from aqueous solutions and also the influence of certain factors on the transformation of hydrates of strontium sulphate into the anhydrous salt, although I had studied in detail the influence of ions on barium sulphate precipitated from 48% alcohol.—The Imperial Industrial Research Institute, Osaka, Japan. [*Received, March 2nd*, 1927.]

## *Separation of* d-*Neobornylamine from* d-*Bornylamine.* By JOHN AUGUSTUS GOODSON.

It has been shown by Forster (J., 1898, **73**, 390) that reduction of *d*-camphoroxime with sodium in boiling amyl alcohol yields a mixture of *cis*- and *trans*-isomerides, distinguished as *d*-bornylamine and *d*-neobornylamine, respectively. The isolation of *d*-neobornylamine from this mixture has presented considerable difficulty (compare Forster and Hart-Smith, J., 1900, **77**, 1152; Pope and Read, J., 1910, **97**, 987; 1913, **103**, 454). Separation of the two amines can be effected in the following way.

A mixture of the two amine hydrochlorides (223 g.), resulting from the reduction of *d*-camphoroxime (290 g.), on recrystallisation from water gave a crop of crystals (77·3 g.). An ethereal solution of the bases from the crystals was shaken consecutively with 500 and 300 c.c. of dilute hydrochloric acid (approx. 0·6*N*), giving solutions 1 and 2. A similar solution of the bases from the mother-liquor was shaken with three quantities of 500 c.c. of the dilute acid, giving solutions 3, 4, and 5. These five preparations were evaporated to dryness, giving the following products:

| No. | Weight. | $[\alpha]_D$ in dry alcohol ($c = 4·4$). | Bornylamine hydrochloride. Calc. | Neobornylamine hydrochloride. Calc. |
|---|---|---|---|---|
| 1 | 49·8 g. | +19·1° | 46·9 g. | 2·9 g. |
| 2 | 27·5 | +12·9 | 23·6 | 3·9 |
| 3 | 50·85 | + 3·7 | 37·15 | 13·7 |
| 4 | 50·35 | − 6·2 | 29·9 | 20·45 |
| 5 | 44·5 | −22·6 | 16·6 | 27·9 |

Pure *d*-bornylamine hydrochloride was easily obtained from the dextrorotatory fractions by recrystallisation from water as shown by Forster (*loc. cit.*) and Frankland and Barrow (J., 1909, **95**, 2017). The specimen giving the highest rotation had $[\alpha]_D^{20°}$ + 23·3° ($c = 4·4$ in dry alcohol), the previous highest value recorded being + 22·7° (Forster, *loc. cit.*). The ethereal solutions of the bases from the lævorotatory fractions were shaken with small quantities of dilute hydrochloric acid until the $[\alpha]_D$ of the hydrochloride of the base remaining in the ethereal solution became constant. *d*-Neobornylamine hydrochloride having $[\alpha]_D^{20°}$ − 49·4° ($c = 4·4$ in dry alcohol) was thus obtained, the previous highest value recorded being − 44·2° (Forster and Hart-Smith, *loc. cit.*).—WELLCOME CHEMICAL RESEARCH LABORATORIES. [*Received, March* 19*th*, 1927.]