

NOTES.

The Preparation of Ethyl β -Iodopropionate. By JOHN W. BAKER.

ETHYL β -chloropropionate (254 g.) was refluxed for 24 hr. with 350 g. of NaI in 300 c.c. of EtOH. The product was largely diluted with Et₂O, and the filtered solution washed with H₂O, Na₂CO₃ aq., and dil. Na₂S₂O₃ aq. and dried. Fractionation gave (1) 20 g., b. p. up to 93°/20 mm., mainly impure iodo-ester, and (2) 344 g., b. p. 85–95°/14 mm., of almost colourless iodo-ester. From the Na₂CO₃ extract, approx. 25 g. of β -iodopropionic acid, m. p. 80–82° after draining, were obtained. The conversion is 92%, and the yield of almost pure iodo-ester 81%.

The ester condensed with ethyl sodioethanetricarboxylate to give an 80% yield of ethyl butane- $\alpha\beta\beta\delta$ -tetracarboxylate (Kay and Perkin, J., 1906, **89**, 1642, obtained an 85% yield).

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The Absorption Spectra of 2 : 2'-Dimethyl- and 2 : 2'-Diethyl-selenocarbocyanine Iodides and of p-Dimethylaminostyrylbenzthiazole Methiodide. A Correction. By L. MARSHALL CLARK.

THE two iodides have been examined by Dr. F. M. Hamer and Miss N. I. Fisher (this vol., p. 191), who inform me that the spectra of their products are different from those described by me (J., 1928, 2313). I have therefore examined alcoholic solutions of my old specimens and of a new preparation of 2 : 2'-diethylselenocarbocyanine iodide, kindly supplied by Dr. Hamer, on a direct-reading Hilger spectrometer. From the results now obtained, it is clear that the previously reported data are incorrect, and that the error is due to a personal misunderstanding of entirely accurate photographs of the spectra supplied by Messrs. Ilfords Ltd.

The wave-lengths of the absorption bands are as follows : 2 : 2'-Dimethylselenocarbocyanine iodide, λ 570 $\mu\mu$; 2 : 2'-diethylselenocarbocyanine iodide, λ 570 $\mu\mu$; *p*-dimethylaminostyrylbenzthiazole methiodide, λ 516 $\mu\mu$.—[Received, January 30th, 1933.]

The Identification of Alcohols in Dilute Aqueous Solution. By HERBERT HENSTOCK.

THE Schotten-Baumann reaction, with certain modifications, was found to be suitable for the purpose. 2½% Aq. EtOH at 15° gives no yield of ester, but does so at a lower temp. Menalda (*Rec. trav. chim.*, 1930, **49**, 967) has described a similar phenomenon. If, in conjunction with lowered temp., a salt is added prior to the *p*-nitrobenzoyl chloride, the yield is further raised by an amount dependent on the nature of the salt.

A solution of 2 g. KOH and 1 g. NaOAc, 3H₂O (or an equiv. molar quantity of other salt) in aq. alcohol of known percentage was cooled to –15°, shaken with 1 g. of *p*-nitrobenzoyl chloride for 1 min., cooled, and shaken again. Another 1 g. of reagent was added, and the process repeated. After dilution with 30–40 c.c. H₂O and subsequent shaking, the mixture was kept for 30 min. or until it became clear. The filtered ester was washed with H₂O, air-dried, and recrystallised from suitably dil. alcohol. For concns. of alcohols below 2½% one-half the wts. were used.

| Alcohol. | M. p. of ester. | % Yield at –15° with NaOAc. | % Yield at –15° without salt. | % Yield at 15° with salt. | Lowest % alcohol identifiable. |
|-------------------------|-----------------|-----------------------------|-------------------------------|---------------------------|--------------------------------|
| Methyl | 96° | 60 | 35·4 | 15·5 | 0·25 |
| Ethyl | 57 | 35·2 | 10·0 | 0·0 | 1·00 |
| <i>n</i> -Propyl | 32 | 32·3 | 12·9 | 25·8 | 0·50 |
| <i>iso</i> Propyl | 55·5 | 19·4 | 0·0 | 0·0 | 1·50 |
| <i>n</i> -Butyl | 35 | 33·3 | 22·2 | 29·6 | 0·25 |
| <i>iso</i> Butyl | 67 | 51·8 | 48·0 | 22·2 | 0·25 |
| <i>n</i> -Amyl | 54 | 37·0 | — | — | 0·50 |

There is an optimum concn. for each salt, KOH, and alcohol corresponding to a max. yield of ester.

Yields with other salts were : sodium formate and propionate 26·4, *n*-butyrate 8·8, succinate 22·0, citrate 10·8, sulphate 29·3, carbonate 23·4, nitrate 17·6, chloride 14·6%, and potassium acetate 11·7, propionate 3·5, *n*-butyrate 16·0, and succinate 6·1% from EtOH.—UNIVERSITY COLLEGE, EXETER. [Received, March 10th, 1932.]