NOTES.

Corrected Data for the 2-Alkylamino-β-naphthathiazoles. By G. M. DYSON, R. F. HUNTER, and R. W. MORRIS.

THE " 2-alkylamino-β-naphthathiazoles " obtained by treating *s*-α-naphthylalkylthiocarbamides with Br (Dyson, Hunter, and Soyka, J., 1926, 2964) are mixtures containing monobromo-substitution products, probably 8-bromo-2-alkylamino-β-naphthathiazoles. The 2-alkylamino-β-naphthathiazoles can be obtained pure in the way described below (compare Hunter and Jones, J., 1930, 947).

The *s*-α-naphthylalkylthiocarbamides previously obtained by condensing α-naphthylamine with the requisite alkylthiocarbimide are more readily prepared pure by treating a solution of α-naphthylthiocarbimide in EtOH with a 30% excess of the corresponding alkylamine, the solution being boiled, cooled, and concentrated under reduced press. at room temp., and the thiocarbamide washed with a little Et_2O before recrystalln. In the condensation of α-naphthylthiocarbimide with *n*-amylamine and *n*-heptylamine, *s*-di-α-naphthylthiocarbamide was formed as a by-product.

s-α-Naphthylmethylthiocarbamide crystallised from EtOH in rhombic plates, m. p. 198° (Dyson and Hunter, *Rec. trav. chim.*, 1926, **45**, 421, recorded needles, m. p. 192°). *s*-α-Naphthylethylthiocarbamide had m. p. 121° (previously recorded as 118°). The following are the revised m. p.'s for the naphthylthiocarbamides described by Dyson, Hunter, and Soyka; the original figures are given in parentheses: *n*-propyl, 101—102° (67°); *n*-butyl, 100° (98°); *n*-amyl, 104° (103°); *iso*amyl, 95° (92°); *n*-heptyl, 65—66° (62°).

The 2-alkylamino-β-naphthathiazoles were prepared as follows: The naphthylthiocarbamide (1 g.) in $CHCl_3$ (10 c.c.) was refluxed with Br (in 2 vols. $CHCl_3$; 5—10% in excess of the 2 equivs. required for cyclisation) for 1—2 mins., and the cooled solution treated with excess of H_2SO_3 and SO_2 and made alkaline with NH_3 aq. The product was obtained from the washed $CHCl_3$ layer and recrystallised from MeOH. The bases had the following m. p.'s: 2-methyl, 161°; 2-ethyl, 107°; 2-*n*-propyl, 86°; 2-*n*-butyl, 67°; 2-*iso*butyl, 71°; 2-*n*-amyl, 78°; 2-*iso*amyl, 90°; 2-*n*-hexyl, 70°; 2-*n*-heptyl, 72°.—IMPERIAL COLLEGE, LONDON, S.W. 7, and ALIGARH MUSLIM UNIVERSITY, INDIA. [*Received, March 30th,* 1932.]

The Identification of α-Ethoxypropionic Acid. By A. DEMOLIS and G. A. R. KON.

THE acid (Gauthier, *Ann. Chim. Phys.*, 1909, **16**, 289) on treatment with thionyl chloride gives the chloride, b. p. 32°/13 mm., and this with NH_3 aq. gives the amide, fine plates from C_6H_6, m. p. 64° (Colson, *ibid.*, 1897, **12**, 235). The *anilide*, obtained from the chloride and NH_2Ph in Et_2O, crystallises from petroleum (b. p. 40—60°) in fine plates resembling asbestos, m. p. 66—67° (Found: C, 68·2; H, 8·0. $C_{11}H_{15}O_2N$ requires C, 68·3; H, 7·8%).—IMPERIAL COLLEGE, LONDON, S.W. 7. [*Received, July 18th,* 1932.]

The Preparation of Pure Zinc Chloride. By R. T. HAMILTON and J. A. V. BUTLER.

BAXTER and LAMB's method (*Amer. Chem. J.*, 1904, **31**, 229) was found unsuitable for the prep. of large quantities.

Pure dry Zn (30 g.) and Na-dried redistilled Et_2O (100 c.c.) are placed in a flask provided with a short bent delivery tube entering the flask through a ground-in glass stopper and drawn out to a jet at the other end. Dry HCl is passed in until the Zn has almost completely dissolved: the stopper is then inserted, and the flask set aside until solution is complete, H escaping through the jet. The clear liquid generally separates into an upper mobile layer, probably consisting of a solution of $ZnCl_2$ in Et_2O, and a lower viscous layer, which is probably a solution of Et_2O in $ZnCl_2$. The flask is warmed on the water-bath and the Et_2O and excess of HCl are distilled in vac., leaving pure

$ZnCl_2$ as a white, excessively hygroscopic powder, sol. in H_2O at all concs. without hydrolysis. Further heating in vac. at about 170° gives a pure product (Found: Cl, 51·8. Calc.: Cl, 52·0%).

The method appears to be suitable for other metals.—THE UNIVERSITY, EDINBURGH. [*Received, June 23rd,* 1932.]