NOTES.

The Preparation of Glutaconic Ester and Acid. By BHAWAN S. GIDVANI.

OWING to the tedious nature of the preparation of glutaconic acid and its ester by the customary methods, the following simplified method may be of interest.

Ethyl β-hydroxyglutarate [25 g.; prepared as described by Dreifuss and Ingold (J., 1923, **123**, 2964)] was dissolved in C_5H_5N (19·3 g.), and $SOCl_2$ (14·6 g.) was added gradually during 2 hrs., the flask being cooled in a freezing mixture. The mixture was left in the cold for a further 2 hrs., then poured into ice-cold dil. HCl, and the ester isolated in the usual way. Ethyl glutaconate (Found : C, 57·9; H, 7·5. Calc. for $C_9H_{14}O_4$: C, 58·1; H, 7·5%) (yield 11·4 g.; 50%) boiled at 132—134°/18 mm., and had $d_{4°}^{20°}$ 1·0496, $n_D^{20°}$ 1·4470 and $[R_L]_D$ 47·38; it gave a 70% yield of the acid on hydrolysis with 50% HCl aq. The dehydration of the ethyl hydroxyglutarate was also carried out by means of P_2O_5 (compare Kon and Nargund, this vol., p. 2462), the yield of glutaconic ester being 61·4%.—IMPERIAL COLLEGE OF SCIENCE, S. KENSINGTON. [*Received, August 5th,* 1932.]

The Reaction of Quinoline and Benzaldehyde. By W. M. CUMMING and J. G. GILLAN.

THE base (m. p. 99—100°) obtained by Cumming and Howie (J., 1931, 3184) has now been identified (mixed m. p.) as benzylidenequinaldine and was derived from a small quantity of quinaldine present in the quinoline employed (Found : C, 88·0; H, 5·7; N, 6·2; *M*, 232. Calc. for $C_{17}H_{13}N$: C, 88·3; H, 5·7; N, 6·1%; *M*, 231). The action of Br vapour on a solution of the base in light petroleum gave the dibromide, colourless plates, m. p. 178° (Found : Br, 41·3. Calc. for $C_{17}H_{13}NBr_2$: Br, 40·9%) (cf. Wallach and Wüsten, *Ber.*, 1883, **16**, 2008). The base forms an anhydrous *hydrochloride*, lemon-yellow needles, m. p. 221—221·5° (see original paper) (Found : N, 5·4. $C_7H_{13}N,HCl$ requires N, 5·2%), and a *dihydrated* form, lemon-yellow needles, m. p. 106—107° (Found: N, 4·8. $C_{17}H_{13}N,HCl,2H_2O$ requires N, 4·6%).—ROYAL TECHNICAL COLLEGE, GLASGOW. [*Received, September 10th,* 1932.]

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*The Recognition of Dithio-*o*-toluidine as 2 : 2′-Diamino-5 : 5′-ditolyl Disulphide.* By REGINALD CHILD.

HODGSON and FRANCE (this vol., p. 1987) identified dithio-*o*-toluidine as 2 : 2′-diamino-5 : 5′-ditolyl disulphide by its prepn. from 5-bromo-2-nitrotoluene. This identification has already been established by Child and Smiles (J., 1926, 2696) by a different method, starting from the corresponding 2-aminotoluene-5-sulphonic acid (Nevile and Winther, *Ber.*, 1880, **13**, 1941).—COCONUT RESEARCH SCHEME, LUNUVILA, CEYLON. [*Received, September 12th,* 1932.]

A New Stereoisomeride of 2 : 3 : 5 : 6-Tetrachlorodioxan. By WILSON BAKER.

IN preparing " 2 : 3-dichlordioxan " (Böeseken, Tellegen, and Henriquez, *Rec. trav. chim.*, 1931, **50**, 909; see Baker and Field, this vol., p. 88), the author isolated one of the five possible stereoisomerides of 2 : 3 : 5 : 6-tetrachlorodioxan, the constitution of which was established by hydrolysis, whereby glyoxal, isolated as its *p*-nitrophenylosazone, was obtained in almost theoretical yield. Butler and Cretcher (*J. Amer. Chem. Soc.*, 1932, **54**, 2987; see also Summerbell and Christ, *ibid.*, p. 3778) have described two other 2 : 3 : 5 : 6-tetrachlorodioxans, m. p. 59—60° and 143—144°, differing from the new product, which is hence a third stereoisomeride. In view of the two recent publications the work will not be continued.

Dioxan was chlorinated as described by Böeseken, Tellegen, and Henriquez (*loc. cit.*), but the product was distilled directly under diminished press. " 2 : 3-Dichlordioxan " came over, followed by viscous higher-boiling fractions, which deposited crystals. These separated from ligroin (b. p. 40—60°) in colourless, highly refracting prisms, m. p. 101° (Found : Cl, 62·5. $C_4H_4O_2Cl_4$ requires Cl, 62·8%). The *substance* has an odour recalling that of pentachloroethane; it sublimes readily in thin prisms below 100° and is exceedingly volatile in steam.

The compound (0·5 g.) and H_2O (25 c.c.) were heated in a sealed flask at 100°. After 3 hrs., the hydrolysis was apparently complete (cf. hydrolysis of the 2 : 3 : 5 : 6-tetrachlorodioxans prep. by Butler and Cretcher), and after 4 hrs. a solution of *p*-nitrophenylhydrazine (1·4 g.) in a little dil. HCl was added. Orange-red glyoxal *p*-nitrophenylosazone was pptd., collected, thoroughly washed, and dried at 120°; m.p. 310° (yield, 1·39 g.; theo., 1·45 g.).—THE DYSON PERRINS LABORATORY, OXFORD. [*Received, September 22nd*, 1932.]