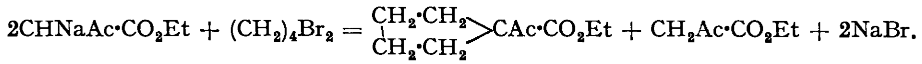
NOTES.

2-Hydroxy-6-methoxy-4-methylquinoline. By O. G. Backeberg and W. O. Kermack.

The m. p.'s given in the literature for the above compound are: 253° (Rabe, *Ber.*, 1931, **64**, 2492), 268° (Monti and Verona, *Gazzetta*, 1932, **62**, 14), 255° (Kermack and Muir, J., 1933, 302), and 272° (Backeberg, J., 1933, 1031); and Kermack and Muir obtained a compound, m. p. 271°, from their 2-hydroxy-6-methoxy-4-methylquinoline by conversion through the 2-chloro-compound into 2 : 6-dimethoxy (or 6-methoxy-2-ethoxy)-4-methylquinoline, followed by removal of the 2-methyl (or ethyl) group. Further investigation has shown that the compound, m. p. 255° (unsharp), has its m. p. gradually raised by repeated crystallisation, though it still melts significantly below 272° and somewhat indefinitely, with decomposition. The latter circumstance renders the results of mixed m. p. determinations somewhat uncertain. We consider that the most probable explanation is that the true m. p. of 2-hydroxy-6-methoxy-4-methylquinoline is 272° (uncorr.) and that the compounds obtained by Rabe, Monti and Verona, and Kermack and Muir contain some tenacious impurity. [*Received, February* 12*th*, 1934.]

The Reaction of Ethyl Sodioacetoacetate and Tetramethylene Dibromide. By Leonard J. Goldsworthy.

The action of ethyl sodioacetoacetate upon tetramethylene dibromide is similar to that upon αδ-dibromopentane, βε-dibromohexane, or *o*-xylylene dibromide (W. H. Perkin, jun., J., 1888, **53**, 187; Solonina, *J. Russ. Phys. Chem. Soc.*, 1904, **36**, 947, 1209) in that the main product is a *cyclo*pentane derivative:



$$2\mathrm{CHNaAc{\cdot}CO_2Et} + (\mathrm{CH_2})_4\mathrm{Br_2} = \left.\begin{array}{l}\mathrm{CH_2{\cdot}CH_2}\\ \mathrm{CH_2{\cdot}CH_2}\end{array}\right>\mathrm{CAc{\cdot}CO_2Et} + \mathrm{CH_2Ac{\cdot}CO_2Et} + 2\mathrm{NaBr}.$$

The constitution of the product follows from its conversion (a) into ethyl *cyclo*pentanecarboxylate and ethyl acetate, and (b) into *cyclo*pentyl methyl ketone.

Tetramethylene dibromide (43·6 g.) and ethyl acetoacetate (52·0 g.) were added to a solution of sodium (9·2 g.) in dry alcohol (128 c.c.) and heated in a sealed bottle at 100° for 8 hours; the alcohol was then distilled off, and the residue mixed with water. The oil extracted by ether, twice distilled under diminished pressure, gave (1) ethyl acetoacetate below 100°/30 mm., (2) ethyl 1-acetyl*cyclo*pentane-1-carboxylate at 120—128°/30 mm., b. p. 125—127°/30 mm. on redistillation (Found: C, 63·8; H, 8·6. Calc. for $C_{10}H_{16}O_3$: C, 65·2; H, 8·7%)* and (3) a small quantity of a yellow oil at 200—205°/30 mm., which was not further investigated.

(a) The ketonic ester (10 g.) was heated on a water-bath for 2 hours with sodium (0·2 g.) dissolved in absolute alcohol (10 c.c.). From the cold reaction mixture, diluted with water and neutralised with dilute hydrochloric acid, ether extracted an oil, which was separated by distillation into ethyl acetate and an ester having a characteristic rancid odour, b. p. 171—172°/760 mm. (Found: C, 67·1; H, 9·9. Calc. for $C_8H_{14}O_2$: C, 67·6; H, 9·9%). Hydrolysis of this ester gave *cyclo*pentanecarboxylic acid, b. p. 214—215°/760 mm., which had a strong rancid odour of perspiration (cf. Wislicenus, *Annalen*, 1898, **273**, 337) (0·7760 g. of the silver salt gave 0·3790 g. Ag. Calc. for $C_6H_9O_2Ag$, 0·3792 g.).

(b) The ketonic ester (19·5 g.) was refluxed for 24 hours with a considerable excess of potash

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* The low value for carbon suggests that the substance, like the corresponding compound prepared by Perkin (*loc. cit.*) from dibromopentane, contained bromine.

(19 g.) in alcohol (150 c.c.), two-thirds of the alcohol then distilled off, the residue refluxed for 3 hours, the bulk of the alcohol finally removed, saturated brine added, and the ketone extracted with ether. The oil obtained from the extract distilled at 150—160°/760 mm. and yielded nearly 4 g. of a colourless oil, b. p. 153—155°/760 mm., on redistillation. The *semicarbazone*, prepared from the ketone and a slight excess of equivalent amounts of semicarbazide and sodium acetate in saturated solution, alcohol just sufficient to clear the solution being finally added, rapidly separated; m. p. 145° after recrystallisation from acetone (Found: N, 24·5. $C_8H_{15}ON_3$ requires N, 24·8%).—UNIVERSITY COLLEGE, RANGOON, BURMA. [*Received, February 12th*, 1934.]