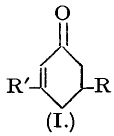
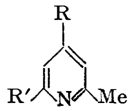
NOTE.

2-cyclo*Hexenones from 2-Methylpyridines.* By ARTHUR J. BIRCH.

DERIVATIVES of several methyl-2-*cyclo*hexenones were required for comparison with those obtained from the reduction products of methylanisoles and methyldimethylanilines (*J.*, 1944, 430; 1946, 593). Ketones of type (I; R′ = alkyl) have been obtained by the cyclisation of 1 : 5-diketones, but for the formation of type (I, R′ = H) the more difficultly accessible 1 : 5-keto-aldehydes are necessary (cf. Koetz and Steinhorst, *Annalen*, 1911, **379**, 20). Derivatives of both kinds of dicarbonyl compound have been obtained from 1 : 4-dihydropyridines (Shaw, *J.*, 1937, 300), and it has now been found that reduction of 2-methylpyridines with sodium and alcohol in liquid ammonia followed by refluxing with acid leads to hydrolysis of the dihydropyridine and cyclisation with direct formation of the *cyclo*hexenone.


(I.)


(II.)

The 2-methylpyridine (10 g.) was reduced with sodium (5 g.) and alcohol (20 c.c.) in ammonia (150 c.c.) (see Birch, *J.*, 1946, 595), water (150 c.c.) added, the product isolated by ether extraction, and refluxed for 6 hours with sulphuric acid (*d* 1·84, 15 c.c.) and water (50 c.c.), and the solution saturated with ammonium sulphate and extracted with ether. The ether solution was dried (K_2CO_3) and the product distilled.

Pyridine.	*cyclo*Hexenone.	Derivatives.
2-Methyl- (II; R = R′ = H).	2- (I; R = R′ = H), b. p. 164—168°, 12%.	2 : 4-Dinitrophenylhydrazone, m. p. 164—165°.
2 : 4-Dimethyl- (II; R = Me, R′ = H).	5-Methyl-2- (II; R = Me, R′ = H),[1] b. p. 179—183°, 15%.	2 : 4-*Dinitrophenylhydrazone*,[2] m. p. 145—146°; semicarbazone,[3] m.p. 175—176°.
2 : 6-Dimethyl- (II; R = H, R′ = Me).	3-Methyl-2- (I; R = H, R′ = Me),[4] b.p. 194—197°, 17%.	2 : 4-Dinitrophenylhydrazone, m. p. 174°; semicarbazone, m. p. 200—202°.
2 : 4 : 6-Trimethyl- (II; R = R′ = Me).	3 : 5-Dimethyl-2- (I; R = R′ = Me),[5] b. p. 205—210°, 30%.	2 : 4-Dinitrophenylhydrazone, m. p. 163—164°; semicarbazone, m. p. 176—177°.

[1] Found: C, 76·4; H, 9·3. Calc. for $C_7H_{10}O$: C, 76·4; H, 9·1%.
[2] Found: C, 53·7; H, 4·8. $C_{13}H_{14}O_4N_4$ requires C, 53·7; H, 4·8%.
[3] Found: C, 57·5; H, 7·7. Calc. for $C_8H_{13}ON_3$: C, 57·5; H, 7·8%.
[4] Found: C, 76·1; H, 9·1. Calc. for $C_7H_{10}O$: C, 76·4; H, 9·1%.
[5] Found: C, 77·8; H, 9·5. Calc. for $C_8H_{12}O$: C, 77·5; H, 9·7%.

The yields of *cyclo*hexenones are poor, and the method has no advantage over the standard ones for their preparation in quantity, except where the alkypyridine is readily available and other methods are difficult, *e.g.*, 5-methyl-2-*cyclo*hexenone from 2 : 4-dimethylpyridine (cf. Koetz and Steinhorst, *loc. cit.*). For the preparation of small amounts of derivatives the method is rapid and convenient.

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