

## NOTE.

*Stable and Labile Semicarbazones from Methyl n-Amyl Ketone.* By W. S. RAPSON and R. G. SHUTTLEWORTH.

THE isolation of a stable and a labile semicarbazone from methyl *n*-amyl ketone is recorded. In an attempt to characterise a sample of this ketone, it was treated with semicarbazide hydrochloride and sodium acetate in an aqueous alcoholic solution in the usual way. There separated, however, a semicarbazone, m. p. 96—97°, which remained stable during its recrystallisation to purity. After standing overnight, the m. p. of this material altered to 121—123°, a value agreeing with that recorded for the semicarbazone of methyl *n*-amyl ketone. In further experiments it was established that this change occurred without loss of weight, and that the final product was definitely a monomeric form of the expected semicarbazone [Found: C, 56.0; H, 10.0; N, 24.2; *M* (ebullioscopic in ethyl alcohol), 155. Calc. for  $C_8H_{17}ON_3$ : C, 56.1; H, 9.9; N, 24.6%; *M*, 171]. The change took place with equal ease if the labile material were left in solution in ethyl alcohol in the cold, or if it were kept in the dark. Inoculation of solutions of the labile with the stable form did not aid the separation of the latter, and an attempt to convert the stable into the labile form by exposure of its alcoholic solution to the ultra-violet light from a mercury lamp was unsuccessful.

Successive preparations of the labile semicarbazone were more and more unstable, until it became unobtainable in the pure condition, crude materials melting between 103° and 110° being formed which altered rapidly to the pure stable form on keeping or on recrystallisation. Many variations in the conditions of preparation were tried without effecting any changes in the nature of the final product, and after 2 months it was found impossible—using the same materials and conditions—to produce anything but the higher-melting modification.

Analogous phenomena have not been met with in a study of the semicarbazones of methyl *n*-butyl and methyl *n*-hexyl ketones. In each of these cases only the one semicarbazone was isolated, though the rather high m. p. (123.5—124.5°) of the material obtained from methyl *n*-butyl ketone renders its identity with the semicarbazone, m. p. 118°, recorded by Michael (*J. Amer. Chem. Soc.*, 1919, 41, 416) rather doubtful.

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