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OPPI BRIEFS: N-ISOHEXYL-N-METHYLAMINE

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OPPI BRIEFS

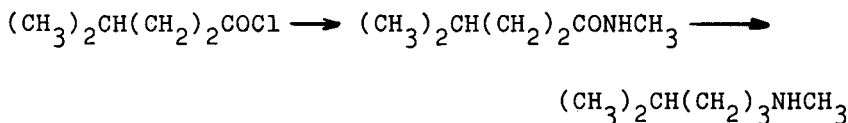
(By J. A. Moore, Associate Editor)

N-ISOHEXYL-N-METHYLAMINE

Submitted by S. Wawzonek and S. M. Heilmann
(4/30/73)

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N-Isohexyl-N-methylamine has been synthesized in 50% yield from isocaproyl chloride.



EXPERIMENTAL

N-Methylisocaproamide was obtained in 74% yield as a colorless liquid, bp. 142-145°/19 mm., n_D^{23} 1.4417 by the addition of isocaproyl chloride to an ethereal solution of two equivalents of methylamine.

Anal. Calcd for $\text{C}_7\text{H}_{15}\text{NO}$: C, 65.07; H, 11.70; N, 10.84

Found: C, 64.67; H, 11.99; N, 10.46

IR(neat): 2.9(NH), 5.94(C=O) μ ; nmr(CDCl_3): δ 0.92 (6H, d, $(\text{CH}_3)_2$, J = 5cps), 1.30-1.70 (1H, m, CH), 1.56 (2H, m, CHCH_2), 2.25 (2H, t, CH_2CO , J = 7cps), 2.78 (3H, d, CH_3N , J = 4cps), 7.68 (1H, s, NH).

N-Isohexyl-N-methylamine.- A solution of 34.8g.(0.298 mole)

J. A. MOORE

of N-methylisocaproamide in 100 ml. of THF was added to a stirred solution of LAH(17g., 0.45 mole) in 250 ml. of THF under nitrogen in one hour. The resulting mixture was then refluxed for 44 hrs and treated first with 50 ml. of water and then with 200 ml. of 6N hydrochloric acid. Tetrahydrofuran was removed by steam distillation and the resulting solution was made alkaline with 200 ml. of 12N sodium hydroxide and steam distilled until the distillate was neutral. The distilled was acidified with conc. hydrochloric acid and evaporated to dryness. The amine hydrochloride was neutralized with sodium hydroxide and the free amine was extracted with pentane. Fractional distillation using a 90 cm. spinning band column gave 23g. (67%) of the pure amine, bp. $134^{\circ}/741$ mm., n_D^{24} 1.4121.

Anal. Calcd for $C_7H_{17}N$: C, 72.97; H, 14.88; N, 12.16

Found: C, 72.90; H, 15.12; N, 12.17

IR(neat): $3.02 \mu(NH)$; nmr(neat): δ 0.92 (6H, d, $(CH_3)_2$, $J = 5$ cps), 1.10-1.70 (5H, m, $CHCH_2CH_2$), 2.34 (3H, s, CH_3), 2.20-2.60 (3H, m, $NHCH_2$).

The α -naphthylthiourea melted at $103.5-105.5^{\circ}$ (corr.).

Anal. Calcd for $C_{18}H_{24}N_2S$: C, 71.95; H, 8.05; N, 9.32

Found: C, 71.70; H, 8.21; N, 9.19