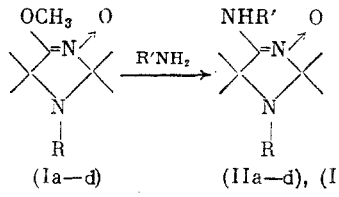
PREPARATION OF α-AMINONITRONES FROM 3-IMIDAZOLINE-3-OXIDE α-METHOXYNITRONES

G. I. Shchukin, I. A. Grigor'ev,
L. A. Vishnivetskaya, and L. B. Volodarskii

UDC 542.91:547.782

α-Methoxynitrones have not been studied extensively [1] but hold promise for the synthesis of α-substituted nitrones such as α-aminonitrones (AN), which have not been readily available. We have found that 4-methoxy-2,2,5,5-tetramethyl-3-imidazoline-3-oxides (MN) (Ia)-(Id) [2] react with NH_3 and methylamine in ethanol at 20°C to give the corresponding AN (IIa)-(IId), (IIIb), and (IIIc) in 70-80% yield. This approach, in contrast to the reductive cyclization of 3-nitroalkyl cyanides in the preparation of pyrrole AN [3], gives a greater scope for the synthesis of AN for other heterocyclic systems.



(Ia—d) (IIa—d), (IIIb, c)

R = O⁻ (a), H (b), CH_3 (c), NO (d); R' = H (II), CH_3 (III)

The elemental composition of the AN synthesized was determined by high-resolution mass spectrometry. The UV spectra of AN (II) and (III) show characteristic absorption at 230-240 nm (log ε ~ 4), while the IR spectra show the νC=N band at 1670 cm^{-1} and νNH bands at 3100-3400 cm^{-1} [4]. The structure of the AN was confirmed by ^1H, ^{13}C, and ^{14}N NMR spectroscopy. The mp of (IIa) is 165-167°C (from ethanol). The mp of (IIb) is 157-160°C (from 3:1 hexane—ethyl acetate). The mp of (IIc) is 190°C (dec.) (from 2:1 hexane—ethyl acetate). The mp of (IId) is 226-229°C (from ethanol). The mp of (IIIb) is 125-126°C (from 3:1 hexane—ethyl acetate). The mp of (IIIc) is 170°C (dec.) (from 3:1 hexane—ethyl acetate).

LITERATURE CITED

1. R. M. Coates and S. J. Firsan, J. Org. Chem., 50, 5198 (1986).
2. G. I. Shchukin, V. F. Starichenko, I. A. Grigor'ev, et al., Izv. Akad. Nauk SSSR, Ser. Khim., 125 (1987).
3. G. D. Buckley and T. J. Elliott, J. Chem. Soc., 1509 (1947).
4. M. L. Bell, R. Oesterlin, K. O. Geolotte, et al., J. Heterocycl. Chem., 14, 1059 (1977).

Novosibirsk Institute of Organic Chemistry, Siberian Branch, Academy of Sciences of the USSR. Translated from Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No. 8, p. 1944, August, 1988. Original article submitted March 30, 1988.

0568-5230/88/3708-1743$12.50