

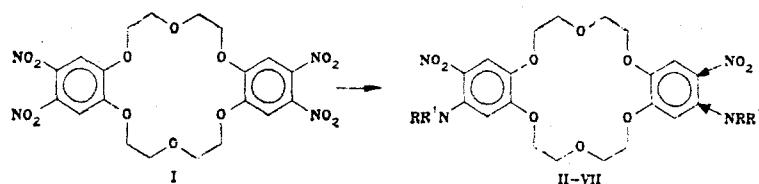
NEW SYNTHESIS OF DIAMINODINITRODIBENZO-18-CROWN-6 DERIVATIVES

É. I. Ivanov, A. A. Polishchuk,
and R. Yu. Ivanova

UDC 547.898.07

We have previously reported [1] that the reaction of tetranitrodibenzo-18-crown-6 (I) with sodium alcoholates in DMSO at room temperature formed a mixture of dialkoxydinitro- and trialkoxynitrobenzene, i.e., the macroheterocycle undergoes scission.

It has been shown that when crown ether I reacts with ammonia or amines in DMSO, compounds II-VII form easily. The previously known synthesis of diamine derivatives like II ($R = R^1 = H$) consists of several steps [2]. A mixture of syn- and anti-isomers of compounds II-VII was synthesized in good yield by heating a solution of the reagents in a boiling water bath for 2-4 h.



II-V $R=H$, VI $R=Me$; II $R^1=H$, III, VI $R^1=Me$, IV $R^1=CH_2Ph$; VII $R-R^1=-(CH_2)_5-$

Compound II: yield 76%, mp 270°. IR spectrum (KBr): 3470, 3360 (NH), 1505 (NO_2), 1130 cm^{-1} (C-O-C). UV spectrum (EtOH): 235, 270, 308, 425 nm. Mass spectrum, m/z: 480 (M^+).

Compound III: yield 78%, mp 240°. IR spectrum (KBr): 3350 (NH), 1505 (NO_2), 1120 cm^{-1} (C-O-C). UV spectrum (EtOH): 238, 274, 315, 428 nm. Mass spectrum, m/z: 508 (M^+).

Compound IV: yield 75%, mp 120°. IR spectrum (KBr): 3320 (NH), 1505 (NO_2), 1115 cm^{-1} (C-O-C). UV spectrum (EtOH): 236, 275, 312, 430 nm. Mass spectrum, m/z: 660 (M^+).

Compound V: yield 72%, mp 112°. IR spectrum (KBr): 3340 (NH), 1505 (NO_2), 1120 cm^{-1} (C-O-C). UV spectrum (EtOH): 245, 274, 316, 430 nm. Mass Spectrum, m/z: 632 (M^+).

Compound VI: yield 82%, mp. 132°. IR spectrum (KBr): 1110 (C-O-C), 1505 cm^{-1} (NO_2). UV spectrum (EtOH): 254, 278, 324, 438 nm. Mass spectrum, m/z: 536 (M^+).

Compound VII: yield 80%, mp 148°. IR spectrum (KBr): 1505 (NO_2), 1120 cm^{-1} (C-O-C). UV spectrum (EtOH): 250, 276, 320, 432 nm. Mass spectrum, m/z: 616 (M^+).

The elemental composition of the synthesized compounds agreed with the calculated values.

LITERATURE CITED

1. É. I. Ivanov, P. B. Terent'ev, and A. A. Polishchuk, Khim. Geterotsikl. Soedin., No. 11, 1572 (1985).
2. H. J. Forster, H. J. Niclas, and N. G. Lukyanenko, Z. Chem., 25, 17 (1985).

A. V. Bogatskii Physicochemical Institute, Academy of Sciences of the Ukrainian SSR, Odessa 270080. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 6, p. 853, June, 1987. Original article submitted July 24, 1986.