NEW MACROHETEROCYCLIC SYSTEMS — 1,4,12,15-TETRATHIA-7,9,18,20-TETRAAZACYCLODOCOSANE DERIVATIVES

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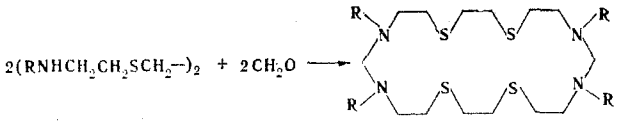
We have accomplished the synthesis of new macroheterocyclic compounds, viz., 1,4,12,15-tetrathia-7,8,18-20-tetrakis(2-carbomethoxyethyl)-7,9,18,20-tetraazacyclodocosane (I) and 1,4,12,15-tetrathia-7,8,18,20-tetrakis(2-cyanoethyl)-7,9,18,20-tetraazacyclodocosane (II), by the reaction of formaldehyde with 1,8-bis(N-carbomethoxyethylamino)- or 1,8-bis(N-cyanoethylamino)-3,6-dithiaoctane, respectively. The reaction proceeds in methanol at 60°C for 6 h under high-dilution conditions.



I R=$CH_2CH_2COOCH_3$; II R=CH_2CH_2CN

Thus a solution of 3.52 g (0.01 mole) of 1,8-bis(N-carbomethoxyethylamino)-3,6-dithiaoctane in 250 ml of methanol and 4 ml of a 40% solution of formaldehyde in 250 ml of methanol were added separately to 1 liter of methanol at 60°C in the course of 6 h. At the end of the reaction the bulk of the methanol was removed *in vacuo*, and the residue was filtered and treated with ether in the cold. The liberated oil was separated and dried *in vacuo* to give 2.2 g (60%) of the macrocycle. IR spectrum: 1050 m, 1200 s, 1370 m, 1440 m, 1735 s, 2840 m, 2930 m, and 2950 m cm^{-1}. The product had R_f 0.36 [Silufol UV-254, benzene–methanol (5:1)].

Compound II [1.94 g (65%)] was similarly obtained in the form of an oil. IR spectrum 920 m, 1070 s, 1140 m, 1200 m, 1420 m, 1460 m, 2250 m, 2870 s, and 2930 s cm^{-1}. The product had R_f 0.40 [Silufol UV-254, benzene–methanol (5:1)]. The results of elementary analysis of I and II for C, H, N, and S were in agreement with the calculated values.

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