FORMATION OF DISPIRO[BIS(1,3-DITHIOLANE)]-1,2';3,2'-(5,5-DIMETHYLCYCLOHEXANE) IN THE REACTION OF 3-METHOXY-5,5-DIMETHYL-2-CYCLOHEXENE-1-THIONE WITH 1,2-ETHANEDITHIOL

L. V. Timokhina, G. M. Panova, and M. G. Voronkov

3-Heterosubstituted 2-cyclohexene-1-thiones synthesized in our laboratory react with nucleophiles at the C³-heterosubstituent bond (AlkO, AlkS, Cl). Thus, the aminolysis of these compounds is a general method for the synthesis of 3-amino-2-cyclohexene-1-thiones [1], while their hydrothiolysis yields dithio analogs of dimedone [2].

In the present work, we report the reaction of 3-methoxy-5,5-dimethyl-2-cyclohexene-1-thione (I) with an S,S-dinucleophile, namely, 1,2-ethanedithiol. In this case, both reaction sites in thioketone I are involved to give previously unreported dispiro[bis(1,3-dithiolane)]-1,2'; 3,2'-(5,5-dimethylcyclohexane) (II). Replacement of the OMe group must be the first step in this reaction followed by addition of the reagent at the C=S bond. Otherwise, the ability would be markedly diminished due to breakdown of the conjugation system.

Dispiro[bis(1,3-dithiolane)]-1,2';3,2'-(5,5-dimethylcyclohexane) (II). A solution of 1.0 g (0.006 mole) thioketone I, 1.7 g (0.018 mole) 1,2-ethanedithiol, and 0.05 g p-toluenesulfonic acid in 10 ml absolute methanol was left for 7 days in an argon atmosphere in the dark. White crystalline II was filtered off and washed with methanol to give 1.2 g (70%) II, mp 162-164°C (from 1-propanol).

PMR Spectrum in CDCl₃: 1.08 (6H, s, 2 Me), 1.90 (4H, s, 4- and 6-CH₂), 2.63 (2H, s, 2-CH₂), 3.25 ppm (8H, s, 4-CH₂-S). Found: C, 48.96; H, 6.90; S 43.59%: Calculated for $C_{12}H_{20}S_4$: C, 49.31; H 6.85; S, 43.84%:

REFERENCES

- 1. L. V. Timokhina, V. A. Usov, Ya. S. Tsetlin, E. O. Tsetlina, and M. G. Voronkov, Zh. Org. Khim., 15, 82 (1979).
- 2. V. A. Usov, L. V. Timokhina, L. I. Lavlinskaya, and M. G. Voronkov, Zh. Org. Khim., 15, 2598 (1979).

Irkutsk Institute of Chemistry, Siberian Branch, Russian Academy of Sciences, Irkutsk 664033, Russia. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 6, pp. 843-844, June, 1998. Original article submitted April 22, 1998.