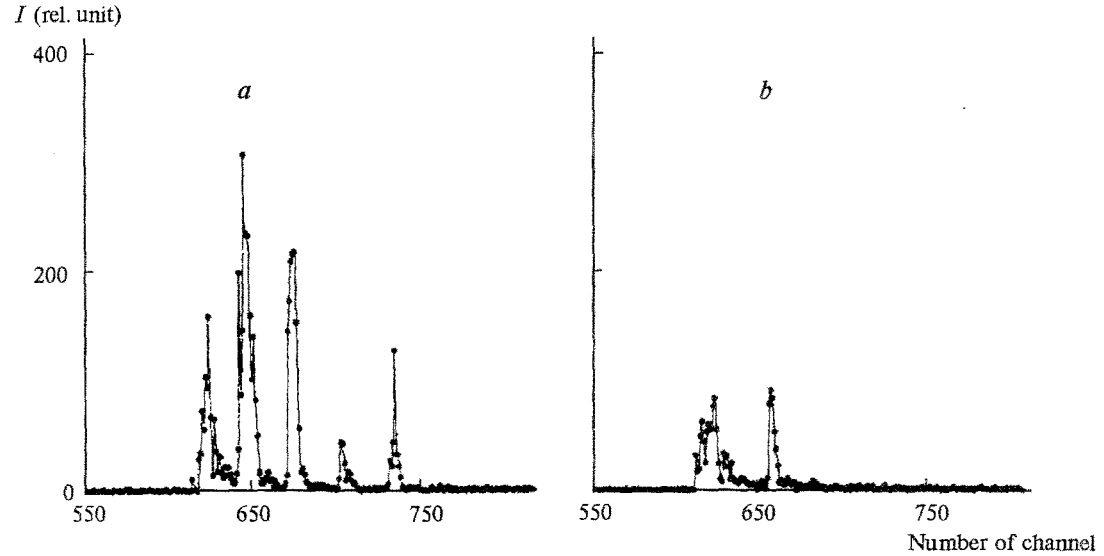

Fig. 1. Light pulses during impact-induced destruction of crystals of the [**1** • $Eu(fod)_3$] complex (*a*) and a mixture of **1** with $Eu(fod)_3$ (*b*) recorded by photon counting on consecutive time intervals. Window width: 1 ms per channel.

This study was financially supported by the Russian Foundation for Basic Research (Project No. 93-03-18165).

References

1. W. Adam, *The Chemistry of Functional Groups: Peroxides*, New York, 1983, 829 pp.
2. G. L. Sharipov, V. P. Kazakov, and G. A. Tolstikov, *Khimiya i khemilyuminestsentsiya 1,2-dioksetanov* [*Chemistry and Chemiluminescence of 1,2-Dioxetanes*], Nauka, Moscow, 1990, 261 pp. (in Russian).
3. A. I. Voloshin, G. L. Sharipov, V. P. Kazakov, and G. A. Tolstikov, *Izv. Akad. Nauk, Ser. Khim.*, 1992, 1056 [*Bull. Russ. Acad. Sci., Div. Chem. Sci.*, 1992, **41**, 828 (Engl. Transl.)].
4. A. I. Voloshin, V. A. Antipin, and V. P. Kazakov, *Khim. Vys. Energ.*, 1994, **28**, 240 [*High Energy Chem.*, 1994, **28** (Engl. Transl.)].

Received March 1, 1995;
in revised form May 15, 1995

Reaction of dithiobisamines with cyclohexene in the presence of phosphorus(v) oxohalides*

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To date there is no information about the use of dithiobisamines as sulfenylating agents. We have investigated the interaction between dithiobismorpholine and cyclohexene in the presence of $POCl_3$ and $POBr_3$. In each case mixtures of the corresponding diastereomeric β,β′-dihalodisulfides were isolated as the products (Scheme 1).

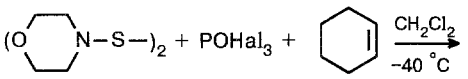
Thus, for the first time dithiobisamines were successfully involved in electrophilic addition to the C=C bond. This method is shown to be more convenient for synthesis of β,β′-dihalodisulfides than the addition of unstable and unpleasantly smelling sulfur dihalides to olefins, which is usually used for this purpose.

* Dedicated to Academician of the RAS N. S. Zefirov (on his 60th birthday).

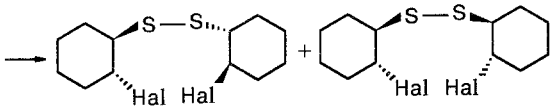
Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 9, p. 1846, September, 1995.

1066-5285/95/4409-1775 $12.50

Scheme 1



Hal = Cl, Br



1a, 2a **1b, 2b**

1: Hal = Cl
2: Hal = Br

^{1}H NMR spectra were registered on a Varian VXR-400 instrument.

Addition of dithiobisamines to cyclohexene in the presence of phosphorus(v) oxohalides. A solution of 2 mmoles of phosphorus(v) oxohalide in abs. CH_2Cl_2 was added with stirring at −40 °C to a solution of dithiobismorpholine (1 mmol) and cyclohexene (2.5 mmol) in the same solvent. After 0.5 h the reaction mixture was heated smoothly to room temperature; then the mixture was passed through a column filter with silica gel (h = 5 cm). When $POCl_3$ was used a chromatographically pure product was isolated; when $POBr_3$ was used the product was further purified by thin layer chromatography on Silufol, eluent — hexane—ethyl acetate (3 : 1).

***trans*-(2,2-Dichlorocyclohexyl)disulfide (1a,b).** Yield 95 %, R_f 0.80. ^{1}H NMR ($CDCl_3$), δ: 4.15 and 4.00 (both m, 2 H, HCCl); 3.21 and 2.95 (both m, 2 H, HCS); 2.4—1.4 (16 H). Found (%): C, 48.43; H, 6.39; Cl, 23.00. $C_{12}H_{20}Cl_2S_2$. Calculated (%): C, 48.15; H, 6.74; Cl, 23.63.

***trans*-(2,2-Dibromocyclohexyl)disulfide (2a,b).** Yield 45 %, R_f 0.85. ^{1}H NMR ($CDCl_3$), δ: 4.3 and 4.15 (both m, 2 H, HCBr); 3.39 and 3.05 (both m, 2 H, HCS); 2.6—1.3 (16 H). Found (%): C, 37.37; H, 5.15; Br, 40.83. $C_{12}H_{20}Br_2S_2$. Calculated (%): C, 37.12; H, 5.19; Br, 41.17.

This work was financially supported by the Russian Foundation for Basic Research (Project No. 94-03-09592).

Received June 7, 1995