

References

- 1 A. M. Richter and E. Fanghänel, *Sulfur Lett.*, 1985, **3**, 25.
- 2 P. G. Degott, *Polymère Carbone-Soufre. Synthèse et Propriétés Electrochimiques. Thèse pour obtenir titre de Docteur*, L'Institut National Polytechnique de Grenoble, 1986, p. 168.
- 3 K. P. Stiehl, A. M. Richter, E. Fanghänel and K. Wiesener, *DDR Patent*, 274709, A1, C1H, 1989 (*Chem. Abstr.*, 1990, **113**, 100925a).
- 4 R. Raap and R. G. Micetich, *Can. J. Chem.*, 1968, **46**, 1057.
- 5 L. Brandsma, *Preparative Acetylenic Chemistry*, Elsevier, Amsterdam–Oxford–New York–Tokyo, 1988, p. 321.
- 6 A. N. Mirskova, S. G. Seredkina and M. G. Voronkov, *USSR Inventors Certificate*, 1204616, C07, 1986 (*Chem. Abstr.*, 1986, **105**, 20846y).
- 7 A. N. Mirskova, A. V. Martinov and M. G. Voronkov, *Zh. Org. Khim.*, 1980, **16**, 2076 [*J. Org. Chem. USSR (Engl. Transl.)* 1980, **16**, 1895].
- 8 B. A. Trofimov, A. S. Atavin, N. K. Gusarova and A. I. Mikhaleva, *USSR Inventors Certificate*, 1819934, C07, 1982 (*Chem. Abstr.*, 1982, **97**, 91733h).
- 9 S. G. Seredkina, V. E. Kolbina, V. G. Rosinov, A. N. Mirskova, V. I. Donskikh and M. G. Voronkov, *Zh. Obshch. Khim.*, 1982, **52**, 2694 [*J. Gen. Chem. USSR (Engl. Transl.)*, 1982, **52**, 2375].

¶ *Reaction of ethylthio(chloro)ethyne with TAA in a KOH/DMSO suspension.* To a KOH (1.39 g, 25 mmol) suspension in 25 ml of DMSO, 1.26 g (16.5 mmol) of TAA and 2 g (16.5 mmol) of ethylthio(chloro)ethyne were successively added, dropwise with stirring. The reaction mixture was stirred for another 1 h and filtered. All the above procedures were performed at room temperature. The filtrate was poured into 70 ml of cold water with ice. The precipitated oil-like product of brown color was separated. The aqueous layer was extracted with diethyl ether, the extract was combined with the above oil-like product, dried over Na_2SO_4 , passed through an Al_2O_3 packed column, the solvent was filtered off and the residue was dried under a vacuum. The product obtained (0.8 g) was a viscous brown oil. $^1\text{H NMR}$ δ : 1.18, 1.26 (m, Me), 2.61 (s, 3H, MeCO) 2.71 (m, 2H, SCH_2). IR (ν/cm^{-1}): 800, 880, 960, 1100, 1180 (C–H), 1250, 1314, 1380, 1442 (Me), 1710 (C=O), 2130 (C \equiv C), 2860, 2914, 2964 (C–H). Found (%): C 41.66, H 5.53, S 40.69. Calc. for $\text{C}_6\text{H}_8\text{S}_2\text{O}$ (%): C 44.98, H 5.03, S 40.02. The product resinified and decomposed during distillation in a vacuum (40–50 °C) in the presence of hydroquinone under argon.

When carried out in a KOH/DMSO suspension at 13–15 °C, the reaction of ethylthio(chloro)ethyne with TAA led to an analogous result.

Received: Moscow, 28th May 1998

Cambridge, 23rd July 1998; Com. 8/04732D