EFFICIENT SYNTHESIS OF DITHIOACETALS OF α-OXOKETENE AND DITHIOFULVENES UNDER PHASE TRANSFER CATALYSIS CONDITIONS

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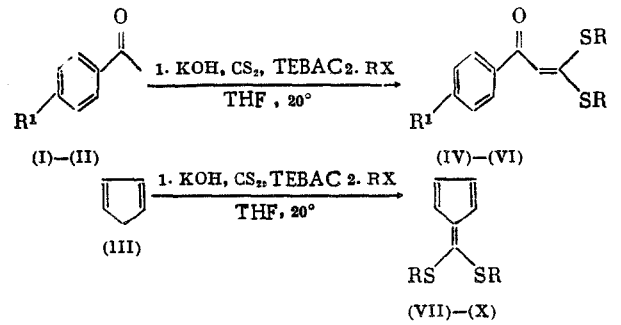
UDC 542.97:547.514.721:546.265.1:547.22

The synthesis of dithioacetals of α-oxoketene and dithiofulvenes is carried out using alkali metal hydrides [1, 2].

We have found that dithioacetals of α-oxoketene and dithiofulvenes are readily obtained under phase transfer conditions. A sample of 40 mmoles alkyl halide is added over 90 min to a stirred mixture of ketene (I), (II), or cyclopentadiene (III), 20 mmoles CS_2, 100 mmoles 85% KOH powder, and 0.5 mmole triethylbenzylammonium chloride (TEBAC) in 10 ml THF at 20°C. Ultraviolet radiation using a UZDN-2T disperser (22 kHz, 400 W) reduces the reaction time to 10 min. The reaction yields are 58% for (IV), 46% for (V), 35% for (VI), 80% for (VII), and 65% for (VIII).



R^1 = H(I), Cl (II); R^1 = H, R = CH_3 (IV); R^1 = H, R = $(CH_2)_2$ (V); R^1 = Cl, R = = CH_3 (VI); R = CH_3 (VII), $(CH_2)_2$ (VIII), CH_2CH_3 (IX), $CH_2C_6H_5$ (X).

The properties of (IV)-(VI) were identical to those described by Thuillier and Vialle [3]. The properties of (VII) and (VIII) were identical to those described by Compper and Kutter [2]. 6,6-Diethyldithiofulvene (IX) was obtained in 47% yield as a dark red oil. PMR spectrum in $CDCl_3$ with TMS as the internal standard (δ, ppm): 1.31 t (6H, CH_3, J = 7 Hz), 3.04 q (4H, CH_2, J = 7 Hz), 6.56 m (4H, CH=CH–CH=CH). 6,6-Dibenzyldithiofulvene (X) was obtained in 71% yield as a dark red oil. PMR spectrum in $CDCl_3$ with TMS as the internal standard (δ, ppm): 4H, CH_2–Ph), 6.47 m (4H, CH=CH–CH=CH), 7.35 m (10H, C_6H_5).

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Institute of Chemistry, Bashkir Branch, Urals Science Center, Academy of Sciences of the USSR, Ufa. Translated from Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No. 1, pp. 213-214, January, 1989. Original article submitted August 10, 1988.

 0568-5230/89/3801-0202$12.50