ONE-STEP SYNTHESIS OF 1-VINYL-2-THIABICYCLO[3.2.0]HEPT-3-ENE FROM VINYLACETYLENE AND HYDROGEN SULFIDE

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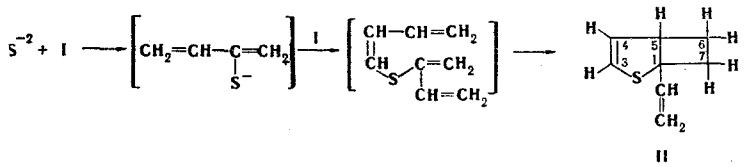
In a study of the reaction of vinylacetylene (I) with hydrogen sulfide in alkaline aqueous organic media we observed that in this case, in addition to the expected addition leading to di(1,3-butadien-1-yl)sulfide, there is also a previously unknown reaction to form 1-vinyl-2-thiabicyclo[3.2.0]hept-3-ene (II) [bp 85° (20 mm), n_D^{20} 1.5415; the results of elementary analysis for C, H, and S were in agreement with the calculated values, and the yield was 26%], apparently via the scheme



II

PMR spectrum, δ, ppm (in CCl_4): 6.11 (q, 3-H), 5.50 (q, 4-H), 3.45 (m, 5-H); J_{34} = 6 Hz, J_{35} = 1.4 Hz, J_{45} = 3 Hz; 2.10-2.60 (m, 6-CH_2, 7-CH_2), 6.14 (q, 1-CH); 5.11 and 5.21 (2q, 1-CH_2); J_{vic} = 10.2 and 17.2 Hz, and J_{gem} = 1.1 Hz. The ^{13}C NMR spectrum (25.2 MHz, TMS, 25°C, $^{13}C-\{^1H\}$ noise decoupling) contains eight signals (δ in parts per million and number of directly attached protons found by the incomplete decoupling with protons): $C_{(1)}$ 61.85, 0; $C_{(3)}$ 126.67, 1; $C_{(4)}$ 123.78, 1; $C_{(5)}$ 55.44, 1; $C_{(6)}$ 27.13, 2; $C_{(7)}$ 35.19, 2; $C_{(\alpha)}$ 140.05, 1; $C_{(\beta)}$ 112.84, 2. The assignment was made on the basis of experiments on selective double $^{13}C-\{^1H\}$ NMR spectroscopy

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