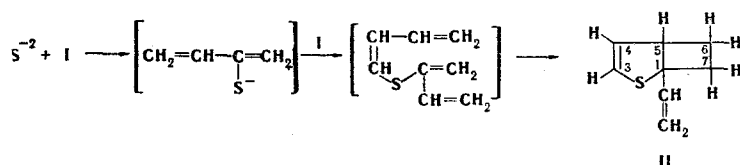


ONE-STEP SYNTHESIS OF 1-VINYL-2-THIABICYCLO[3.2.0]HEPT-3-ENE  
FROM VINYLACETYLENE AND HYDROGEN SULFIDE

B. A. Trofimov, G. A. Kalabin, S. V. Amosova,  
G. K. Musorin, V. V. Keiko, and V. V. Kryuchkov

UDC 547.736.07:543.422.25.4

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



PMR spectrum,  $\delta$ , 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 ( $\delta$  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;  $C(8)$  140.05, 1;  $C(9)$  112.84, 2. The assignment was made on the basis of experiments on selective double  $^{13}C\{-^1H\}$  NMR spectroscopy.

Irkutsk Institute of Organic Chemistry, Siberian Branch, Academy of Sciences of the USSR. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 2, p. 285, February, 1976. Original article submitted July 15, 1975.

This material is protected by copyright registered in the name of Plenum Publishing Corporation, 227 West 17th Street, New York, N.Y. 10011. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, microfilming, recording or otherwise, without written permission of the publisher. A copy of this article is available from the publisher for \$7.50.