PUMMERER REACTION IN SULFOXIDES OF THE SULFOLANE SERIES

N. N. Novitskaya, B. V. Flekhter,
L. V. Spirikhin, and G. A. Tolstikov

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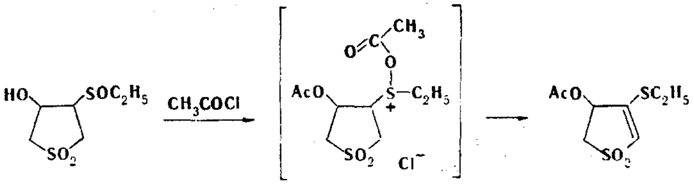
We have observed the unusually facile occurrence of the Pummerer reaction, i.e., the the conversion of 4-hydroxy-3-(1-alkylsulfinyl)sulfolanes (I) to 4-acetoxy-3-(1-alkylthio)-2-sulfolenes (II) under the influence of excess acetyl chloride at 20°C. Thus, trans-I [mp 95-96°C; IR spectrum: 3220 (OH); 1316, 1282, 1148, and 1125 (SO_2); 1008 cm^{-1} (SO)], which was obtained by oxidation of trans-4-hydroxy-3-(1-ethylthio)sulfolane by means of 30% H_2O_2 in acetone, gave sulfolene II [mp 133-135°C; IR spectrum: 1752, 1230 ($OCOCH_3$); 1584 (C=C); 1320, 1296, and 1141 cm^{-1} (SO_2): PMR spectrum ($CDCl_3$): 1.31 (t, 3H, CH_3); 2.04 (s, 3H, Ac); 2.80 (q, 2H, CH_2); 3.29 (dd, 1H, 5-H), J_{55} = 14 Hz, $J_{45\,trans}$ = 3.5 Hz; 3.57 (dd, 1H, 5-H), $J_{45\,cis}$ = 7.5 Hz; 5.80 (dd, 1H, 4-H), $J_{45\,trans}$ = 3.5 Hz, $J_{45\,cis}$= 7.5 Hz; 6.21 ppm (s, 1H, 2-H)] in quantitative yield.



I

II

Under similar conditions, cis-I [mp 144-146°C; IR spectrum: 3220 (OH); 1308, 1280, 1130, and 1109 (SO_2); 1012 cm^{-1} (SO)], which was obtained by oxidation of cis-4-hydroxy-3-(1-ethylthio)sulfolane, gave sulfolene II in 95% yield.

Institute of Chemistry, Bashkir Branch, Academy of Sciences of the USSR, Ufa 450054.
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