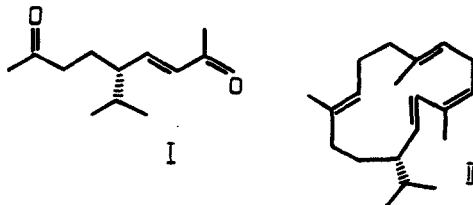


PRODUCTION OF (+)-NORSOLANADIONE

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(S)-5-Isopropylnon-3E-ene-2,8-dione ((+)-norsolanadione) (I) is known as a product of the autooxidation of (+)-solanone, a component of tobacco leaf extract [1]. In small yield and in admixture with other products, the diketone (I), which is of interest as a chiral α,ω -bifunctional synthon block, is formed from the readily available natural diterpene (+)-cembrene (II) [2] on oxidation by the Jones reagent [3].



We have established that the diketone (I) can be obtained easily by the ozonolysis of cembrene at a low temperature using a molar ratio of ozone to cembrene of about 1:0.7. The yield of product amounted to 49% on the cembrene that had reacted, at a degree of conversion of the initial compound of 45%.

A mixture of ozone and oxygen (the output of the ozonizer being 7 mmole of O_3 per hour) was passed at -70°C into a solution of 1.50 g of (+)-cembrene (II) in 150 ml of absolute CH_3OH . The reaction mixture was purged with argon, 0.1 g of Lindlar catalyst ($\text{Pd}\cdot\text{CaCO}_3\cdot\text{PbO}$) was added to it, and it was stirred in an atmosphere of hydrogen at $+25^\circ\text{C}$ until the decomposition of the peroxide compounds was complete (test with an acidified aqueous KI solution). After this, the catalyst was filtered off, and the solution was evaporated to dryness, to give 1.62 g of an oil the chromatography of which on neutral alumina permitted the isolation of 0.82 g of unchanged cembrene (with hexane as eluent) and 0.24 g of (+)-norsolanadione (I) [eluted with hexane-diethyl ether (9:1)] having $[\alpha]_D^{20} +5.68^\circ$ (c 5.02; CHCl_3) and $n_D^{20} 1.4698$ (according to the literature [3]; $[\alpha]_D^{21} +6.05^\circ$). The IR, PMR, and mass spectra of the sample obtained corresponded to those reported in the literature. ^{13}C NMR spectrum (in CDCl_3 , δ scale, ppm): 19.26 q (CH_3), 20.56 q (CH_3), 26.57 t (C-6), 26.97 q (C-1), 30.09 q (C-9), 31.86 d (CH), 41.52 t (C-7), 49.07 d (C-5), 132.51 d (C-3), 149.88 d (C-4), 198.18 s (C-2), 208.17 s (C-8).

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