

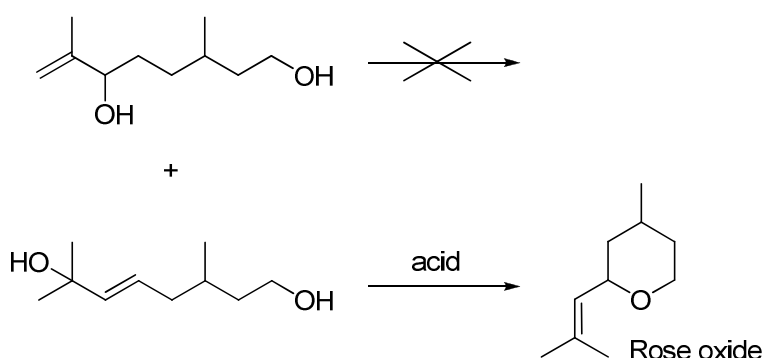
Supporting Information for:

"Dark" Singlet Oxygenation of β -Citronellol, a Key-Step in the Manufacture of Rose Oxide

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Rose oxide via acid-catalyzed cyclization of the diol mixture obtained from DSO of β -citronellol:



Only the tertiary diol in the 1/1 diol mixture cyclizes to Rose oxide, as illustrated above.

Under nitrogen, a reactor was charged with 2.8 L 20 wt-% sulfuric acid and 1.2 L n-heptane. The mixture was stirred well and heated up to 60 °C. Then 1231.5 g of the diol mixture obtained by DSO was added gradually to the mixture within 2 hours. After the addition was complete, the mixture was stirred for 1 hour at 60 °C. The mixture is then cooled to 25 °C. The phases are separated, and the aqueous sulphuric acid layer was extracted with 1.2 L n-heptane.

The combined organic layers were washed with 600 mL 5 wt-% sodium carbonate solution. The phases were separated and the n-heptane layer was concentrated. The slightly yellow/orange residue (raw-Rose oxides) was distilled in vacuum at 70 °C/30-40 mbar.

Yield: 557.2 g Rose Oxide (quantitative, only the tertiary alcohol undergoes cyclization). The cis/trans ratio of the Rose oxide obtained in this way corresponds to 10/1.