The RuO₄-Catalyzed Ketohydroxylation – Part I. Development, Scope and Limitation.

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List of Contents

- S-2 General experimental methods
- S-3 ¹H NMR spectra of the acyloins
- S-16 References

General Remarks.

Infrared spectra (IR) were recorded as a thin film between KBr-plates. Proton (¹H NMR, 400 MHz) and carbon (¹³C NMR, 100.6 MHz) nuclear magnetic resonance spectra were recorded in chloroform(d-1) and referenced to the solvent signal. All signal points are listed on a δ -scale in ppm. Gaschromatographic analysis was performed using a DB1-column (carrier gas: helium). Flash-chromatography was done on silica 60 (230-400 mesh) using head pressure by means of compressed air. Petroleum ether refers to that fraction boiling in the range 35 – 60 °C. Ethyl acetate was purified by distillation over CaCl₂ prior to use. RuCl₃ was obtained from Aldrich. A stock solution was prepared calculating with RuCl₃(H₂O)₂ and dissolving the catalyst (2.44 g, 10 mmol in 100 mL water (0.1 M). The deep brown solution can be stored on the bench for weeks without loss of activity. Olefins **17**ⁱ, **21**ⁱⁱ, **23**ⁱⁱⁱ, **25**^{iv}, **27**^v, **29**^{vii} and **33**^{viii} were prepared according to literature procedures. All other starting material were purchased from commercial suppliers and used without further purification. In cases where the regioselectivity of the oxidation was better than 90 : 10 only the spectral data of the major isomer are reported. Regioselectivities were determined by GC- or NMR-integration of the crude product mixture.



























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