Supplementary Material

Microwave-Assisted oxidation reaction of primary alcohols with sensitive functional groups to aldehydes using Ruthenium Diphosphorus Complexes

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General Remarks for Testing

Toluene was distilled over sodium wire and stored under an inert atmosphere. Xylene was used as purchased. Benzyl alcohol (99.8%) and crotonitrile (99%, mixture of cis and trans) were purchased from Sigma-Aldrich and used without further purification. The internal standard 1,4-dimethoxybenzene (Aldrich, 99%) was dried under vacuum and stored under an inert atmosphere. For substrates containing a methoxy group, 2,6-lutidene (Aldrich, 99%) was used as the internal standard. All alcohol substrates and aldehydes (for calibration purposes) were purchased from Aldrich, Merck, and Fluka unless stated otherwise.

A calibration curve for each substrate-product combination was generated. This involved the $^1$H NMR analysis of a mixture of varying concentrations of a primary alcohol and its respective oxidised product in CDCl$_3$ (250 µL). An internal standard was used for each run at constant concentration (2.5 mg) and set as the reference peak (integration value of 1). Relevant proton peaks of the substrate and product were integrated at the current concentration and a curve of integration area versus concentration (minimum 4 points) was generated. Least squares regression yielded the calibration equation which was used for all catalytic testing, with normal interpolation techniques used when necessary. For further details see Bekiroglu et al.$^1$

All microwave mediated reactions were conducted using a CEM Discover microwave. Temperature measurements were conducted using an infrared temperature sensor situated below the reaction vessel. Reaction times refer to the total hold time at the indicated temperature with the ramp times ranging from 1 to 2 minutes. While there was some variation in the ramp time for each experiment, all reported examples were reproducible using the indicated hold time/temperature. The microwave was calibrated for power and temperature by the distributor.

Reference:
$^1$H NMR spectrum of Compound L1 in CDCl$_3$
$^{13}$C NMR spectrum of Compound L1 in CDCl$_3$
$^1$H NMR spectrum of Compound L2 in CDCl$_3$
$^{13}$C NMR spectrum of Compound L2 in CDCl$_3$
$^1$H NMR spectrum of Compound 5 in CDCl$_3$
$^{13}$C NMR spectrum of Compound 5 in CDCl$_3$
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$^{13}$C NMR spectrum of Compound 14 in CDCl$_3$
$^{31}$P NMR spectrum of Compound 14 in CDCl$_3$