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Reaction monitoring in multiphase systems: Application of coupled in situ spectroscopic techniques in organic synthesis

By Leif Robin Knöpke

Cuvillier Verlag Feb 2012, 2012. Buch. Condition: Neu. Neuware - The benefit of coupling different in situ spectroscopic methods (ATR-FTIR / UV-vis / Ramanspectroscopy) is exemplarily demonstrated by the means of two reactions as application examples: the Lewis acid mediated formal [3+3] cyclocondensation reaction of a diene with aketenacetale and the heterogeneously catalysed asymmetric hydrogenation of imines. For both reactions new reactors (deep-temperature cell, probe autoclave) have been developed enabling in situ measurements under real reaction conditions. For the [3+3] cyclocondensation reaction it was demonstrated that the nature of the formed product depends on the different intermediates formed between the ketenacetal and the acids (complexation or acid catalysed rearrangement reaction). Furthermore the mechanisms of similar reactions (including other Lewis acids like $AlCl_3$, Al_2O_3 and TiO_2 or substrates like N-salicylideneaniline) could be successfully elucidated. The effectiveness of the hydrogenation depends on the specific interaction of the tested ketimines with the phosphoric acid ester used as chiral modifier. This interaction is mainly influenced by the used solvent and leads to the formation of a carbocation (CH_3OH) or a contact ion pair in (CH_2Cl_2 , CF_3CH_2OH). Furthermore, the detected strong adsorption of imines and modifier on the Pt/ Al_2O_3 catalyst affect their efficiency and activity in the reaction. 140 pp. Englisch.


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Reviews

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