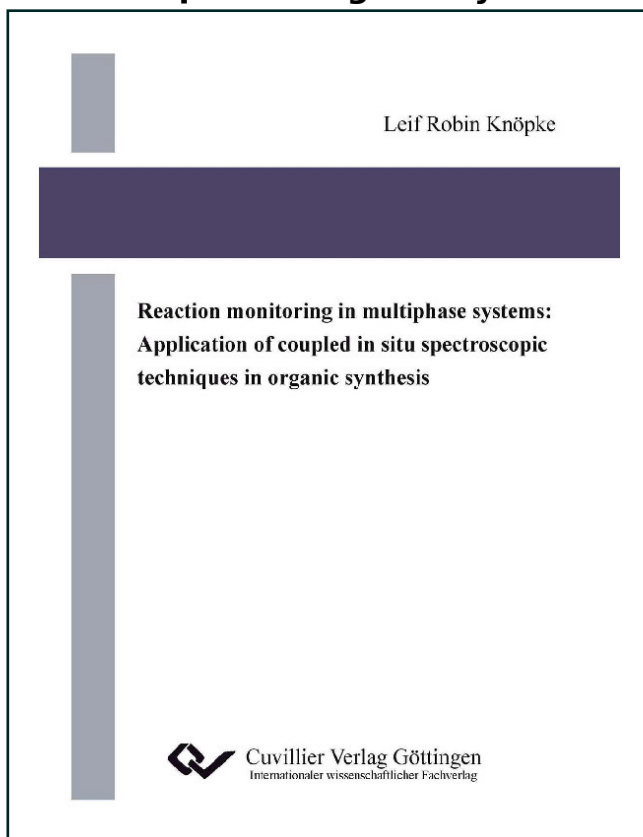




Leif Robin Knöpke (Autor)

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



<https://cuvillier.de/de/shop/publications/94>

Copyright:

Cuvillier Verlag, Inhaberin Annette Jentsch-Cuvillier, Nonnenstieg 8, 37075 Göttingen,
Germany

Telefon: +49 (0)551 54724-0, E-Mail: info@cuvillier.de, Website: <https://cuvillier.de>



TABLE OF CONTENTS

Zusammenfassung	iii
Abstract	v
Selbständigkeitserklärung	vii
Acknowledgements	ix
1. MOTIVATION AND OBJECTIVES	1
2. STATE OF THE ART	5
2.1. Opportunities and limitations of common in situ methods	5
2.2. In situ studies by coupled techniques	10
3. TECHNICAL PART	15
3.1. Development of a reaction cell for the in situ investigations of low-temperature preparation processes	15
3.2. Development of a reaction cell for the in situ investigations of pressure reactions in multiphase systems	17
4. APPLICATION EXAMPLE 1: Elucidating the directing effect of the used Lewis acid on the reaction pathway in formal [3+3] cyclocondensation reactions	23
4.1. The ex situ studies at room temperature	24
4.2. The in situ studies under reaction conditions	32
4.3. Conclusion 1	39
4.4. Transfer of the obtained results to similar systems	39
4.5. Utilisation of aluminium(III)chloride as Lewis acid	40
4.6. Supplementary ex situ investigations	45
4.7. From single phase to multiphase systems: utilisation of solid Lewis Acids	47
4.8. Introduction of N-salicylideneaniline 14 as substrate for the reaction	50
4.9. Influence of the reaction time	59
4.10. Conclusion 2	60



TABLE OF CONTENTS

5.	APPLICATION EXAMPLE 2: Heterogeneously catalysed asymmetric hydrogenation of imines: Reaction monitoring and Mechanistic studies	63
5.1.	Interaction of selected imines with the chiral modifier in liquid phase	65
5.2.	Interaction of selected imines with the catalyst surface	74
5.3.	Interaction of the chiral modifier 22 with the catalyst surface	81
5.4.	Elucidation of the adsorption sites	83
5.5.	In situ studies: Testing of the reaction unit	85
5.6.	Conclusion 3	91
6.	GENERAL CONCLUSION AND OUTLOOK	95
7.	LITERATURE	99
	APPENDIX	A
	Experimental Section Application Example 1	A
	Experimental Section Application Example 2	C
	Selected bond lengths and angles in the bis-chelate complex 8b	F
	ATR-FTIR and Raman spectra of N-salicylideneaniline 14 in CCl ₄ (cf. Figure 25)	G
	ATR-FTIR spectrum of the 1:1-mixture of 14 and 22 in CH ₂ Cl ₂ before the co-adsorption experiments on Pt/Al ₂ O ₃ took place (cf. Figure 41)	H
	Remark to the spectroscopic limitations	I
	Remark to the limitations measuring kinetics	L
	Table of compounds	N
	Supplementary literature	R