

Low temperature NMR spectroscopy with *in situ* visible light illumination

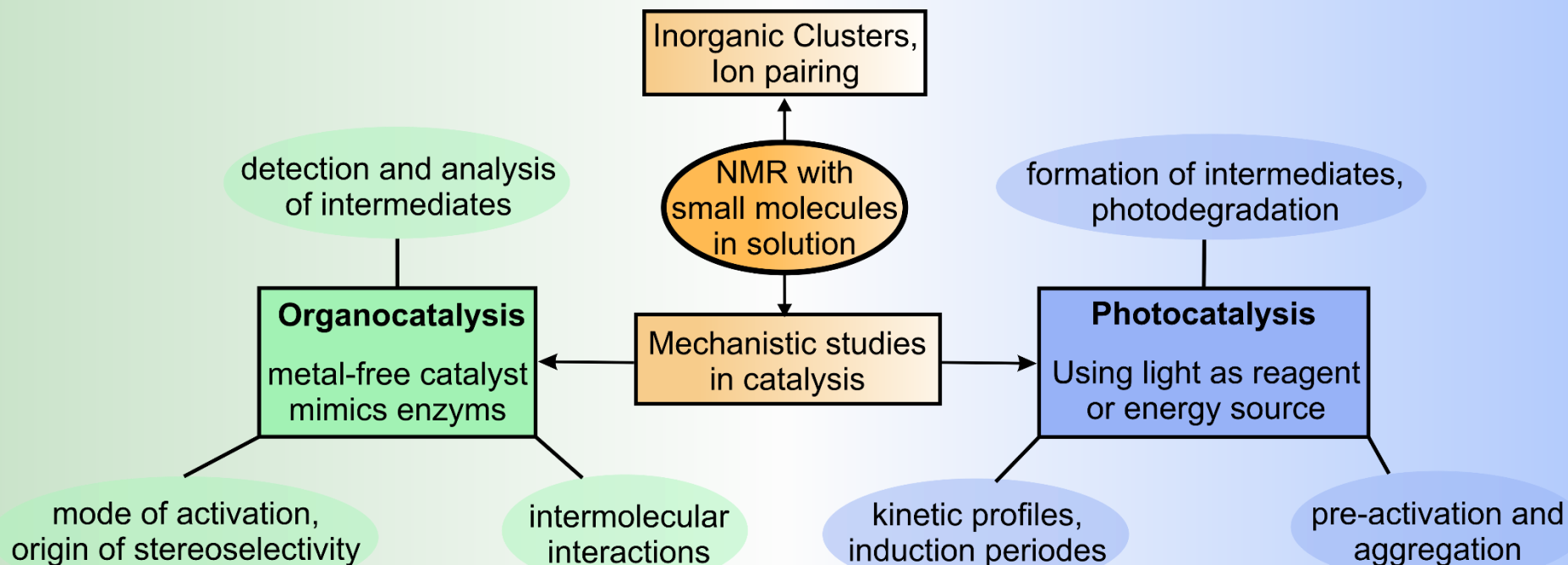
Dr. Johannes Gramüller
Working Group of Prof. Dr. Ruth. M. Gschwind
Faculty for Chemistry and Pharmacy
University of Regensburg

PPNMR

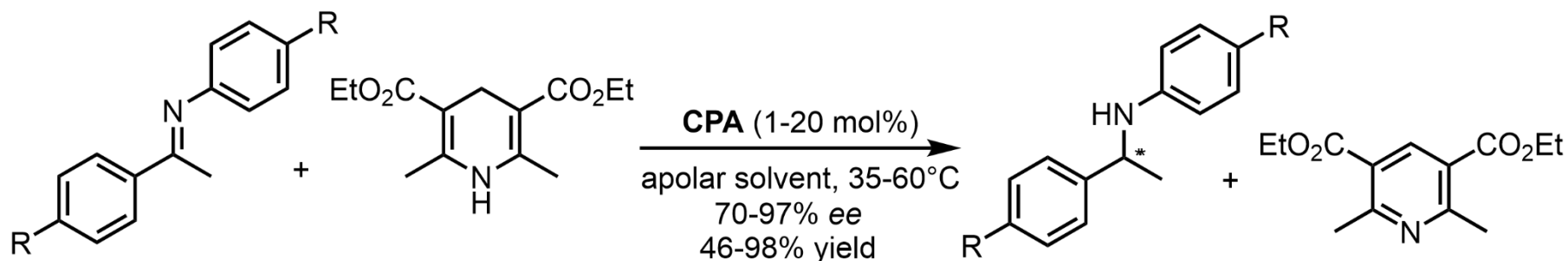
Berlin, 21.03.2023



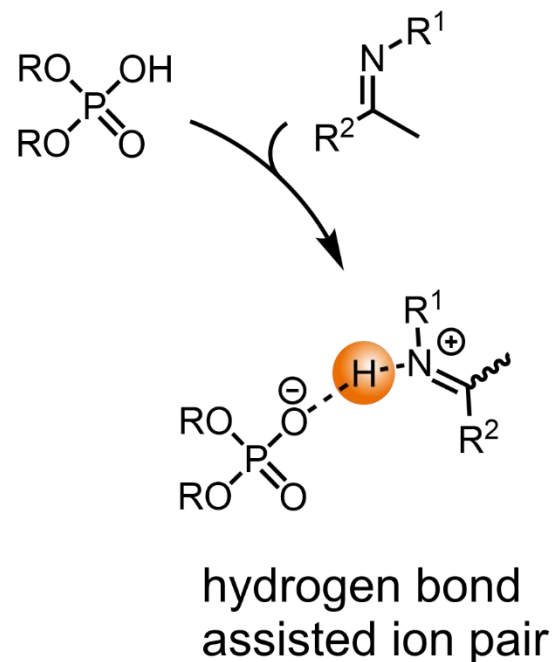
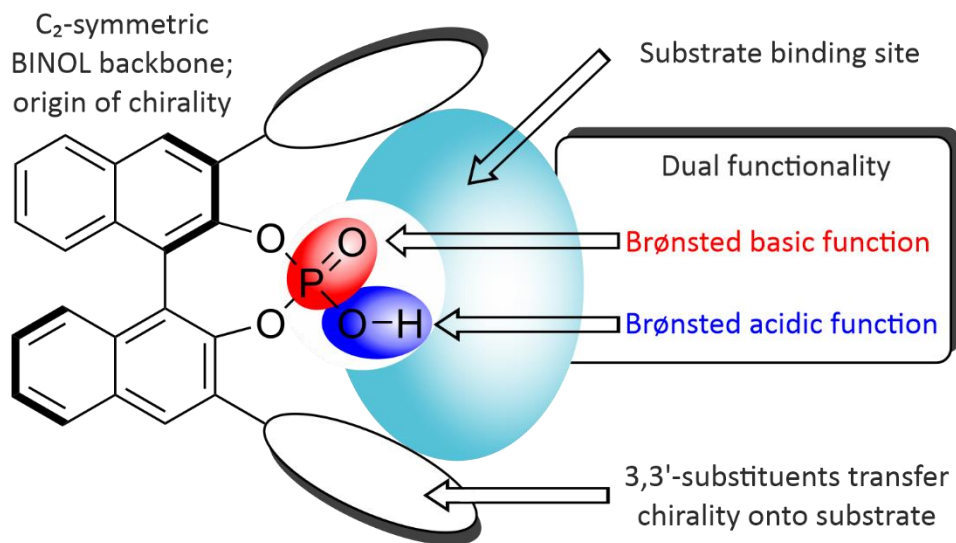
Universität Regensburg



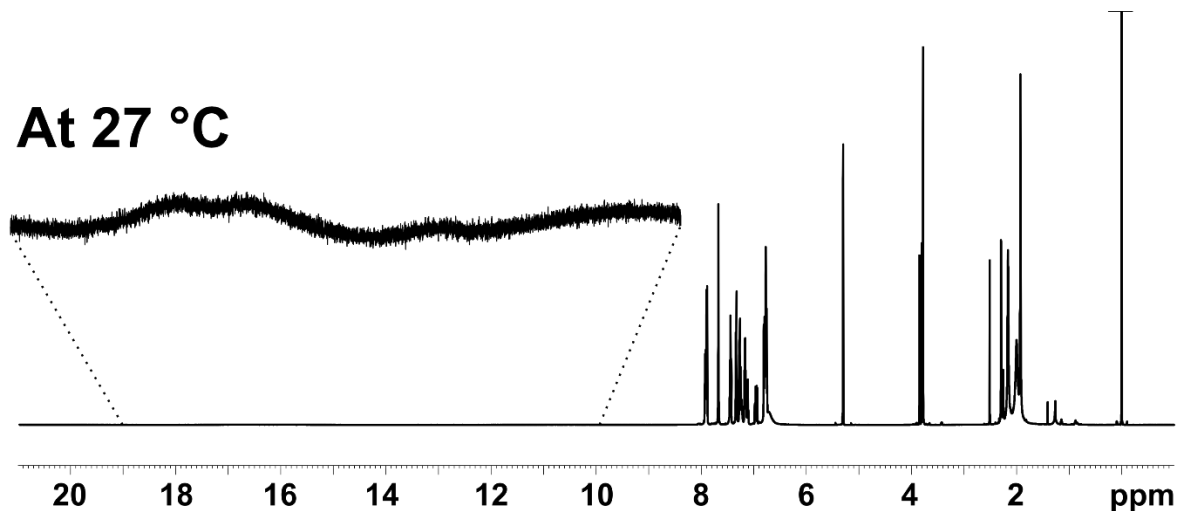
Transfer hydrogenation of imines



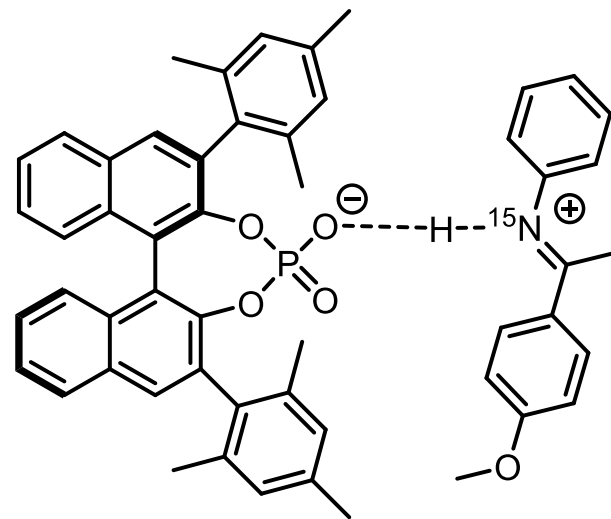
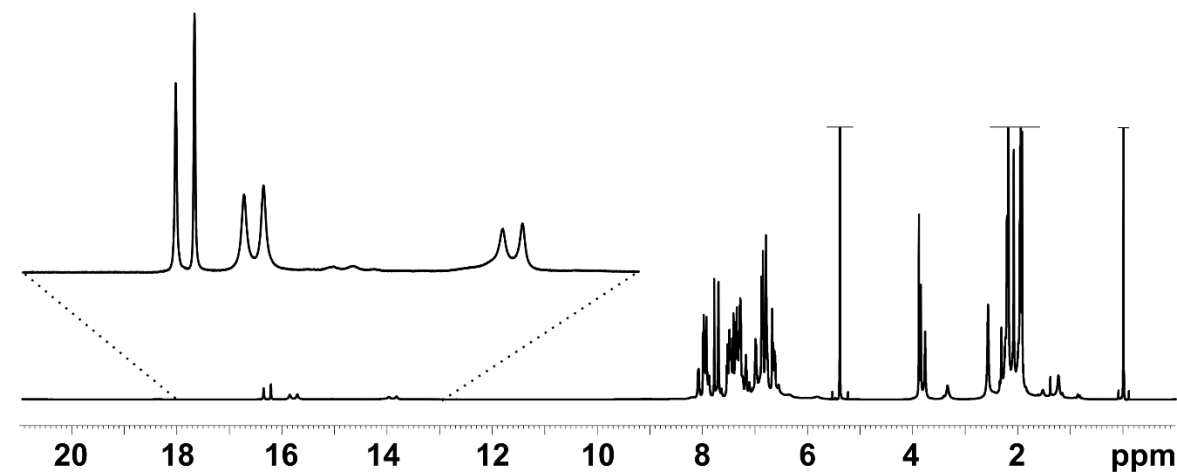
chiral phosphoric acids (CPA)



At 27 °C

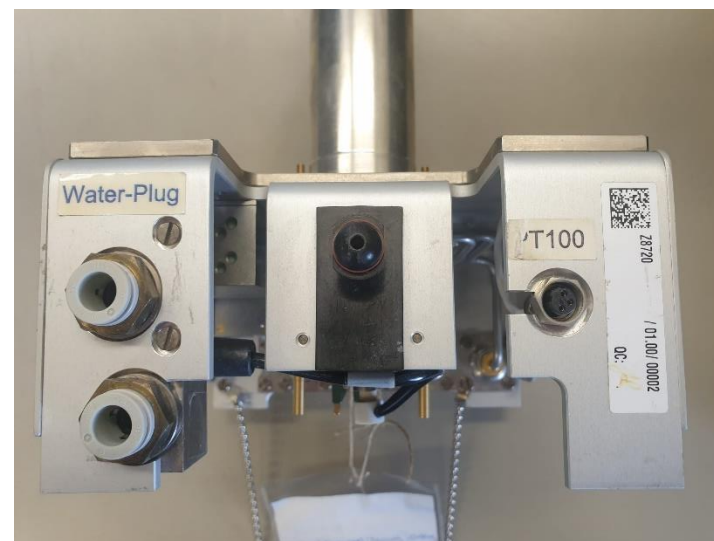


At -93 °C

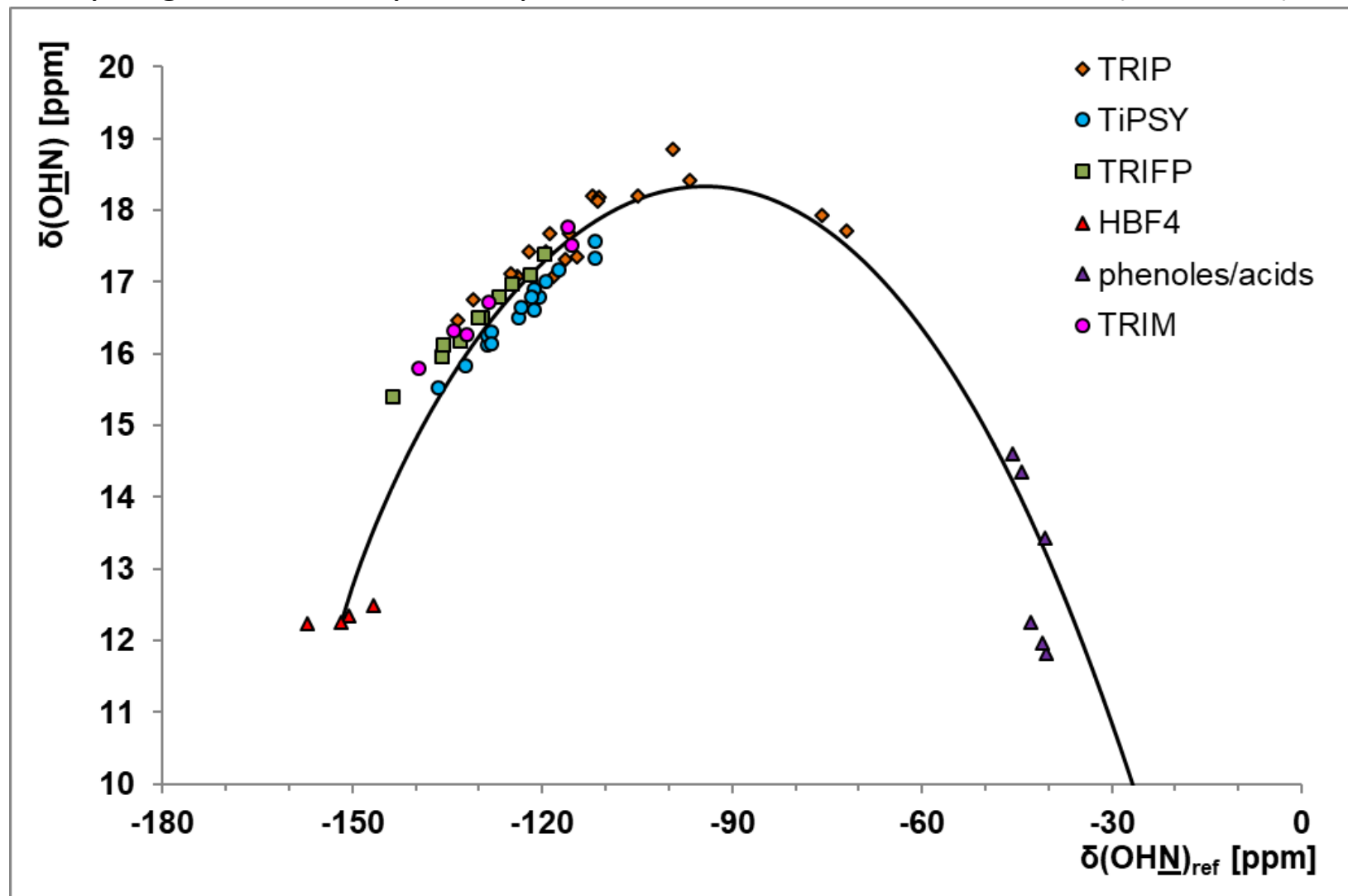


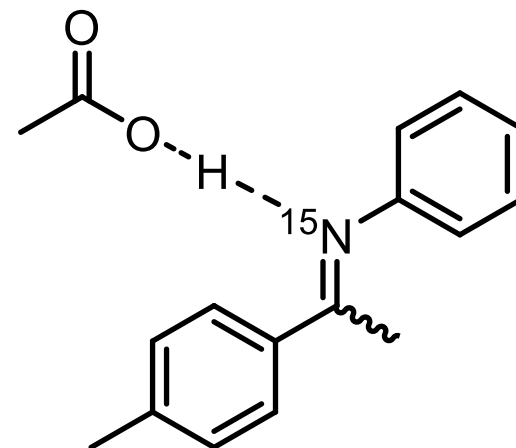
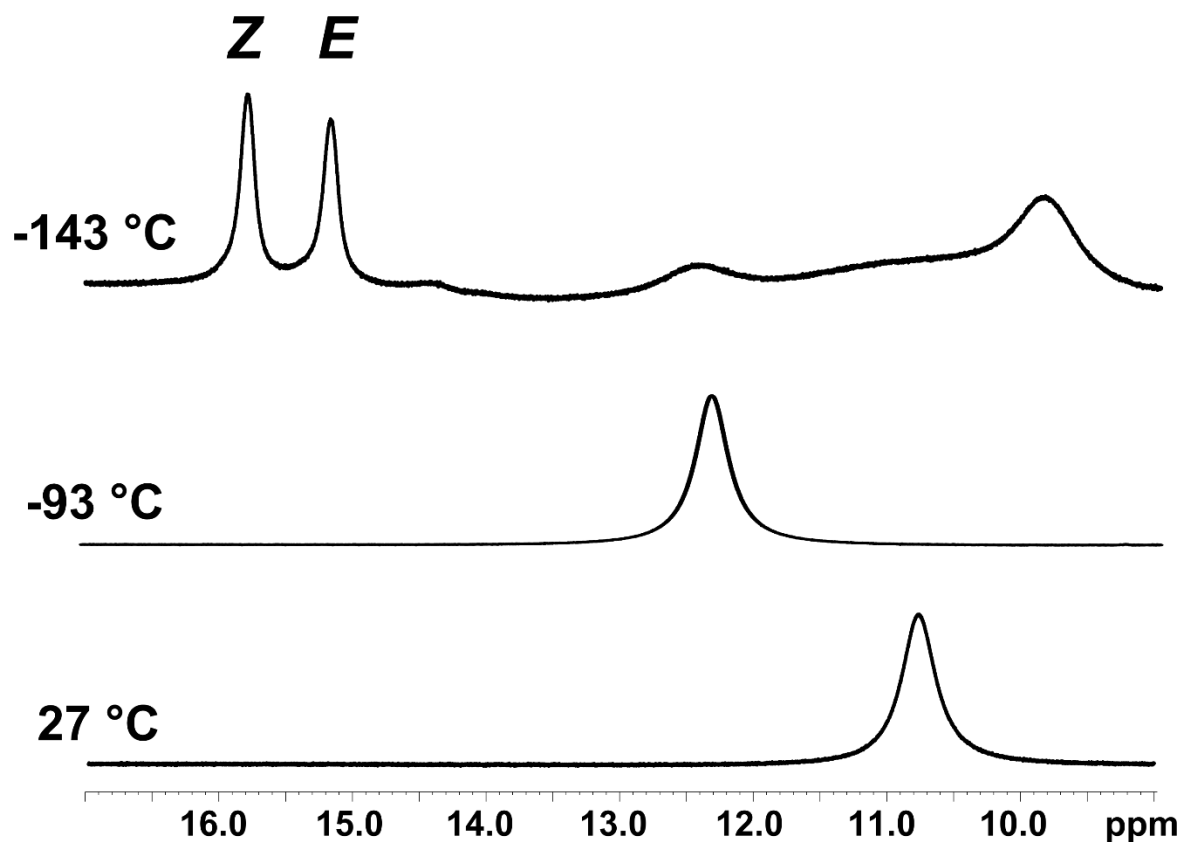
Samples:

1 : 1 ratio of acid and imine

50 mM, CD_2Cl_2 , dry, 600 MHz

Hydrogen bond analysis: Empirical „Steiner-Limbach“ correlation ($\delta^1\text{H} / ^{15}\text{N}$)





In Freonen mixture
(CDF₂Cl + CDFCl₂)

- Synthesis: $\text{CDCl}_3 + \text{SbF}_3 + \text{SbCl}_5 \rightarrow \text{CDF}_2\text{Cl} + \text{CDFCl}_2$
- Condensation on Al_2O_3 to remove HF (1 - 2x)
- Guide over P_2O_5 and KOH to remove water



HiCUBE
PFEIFFER VACUUM

40
40

HP Press 80 mm TC 110, DN 53 150

Ge Ni Al

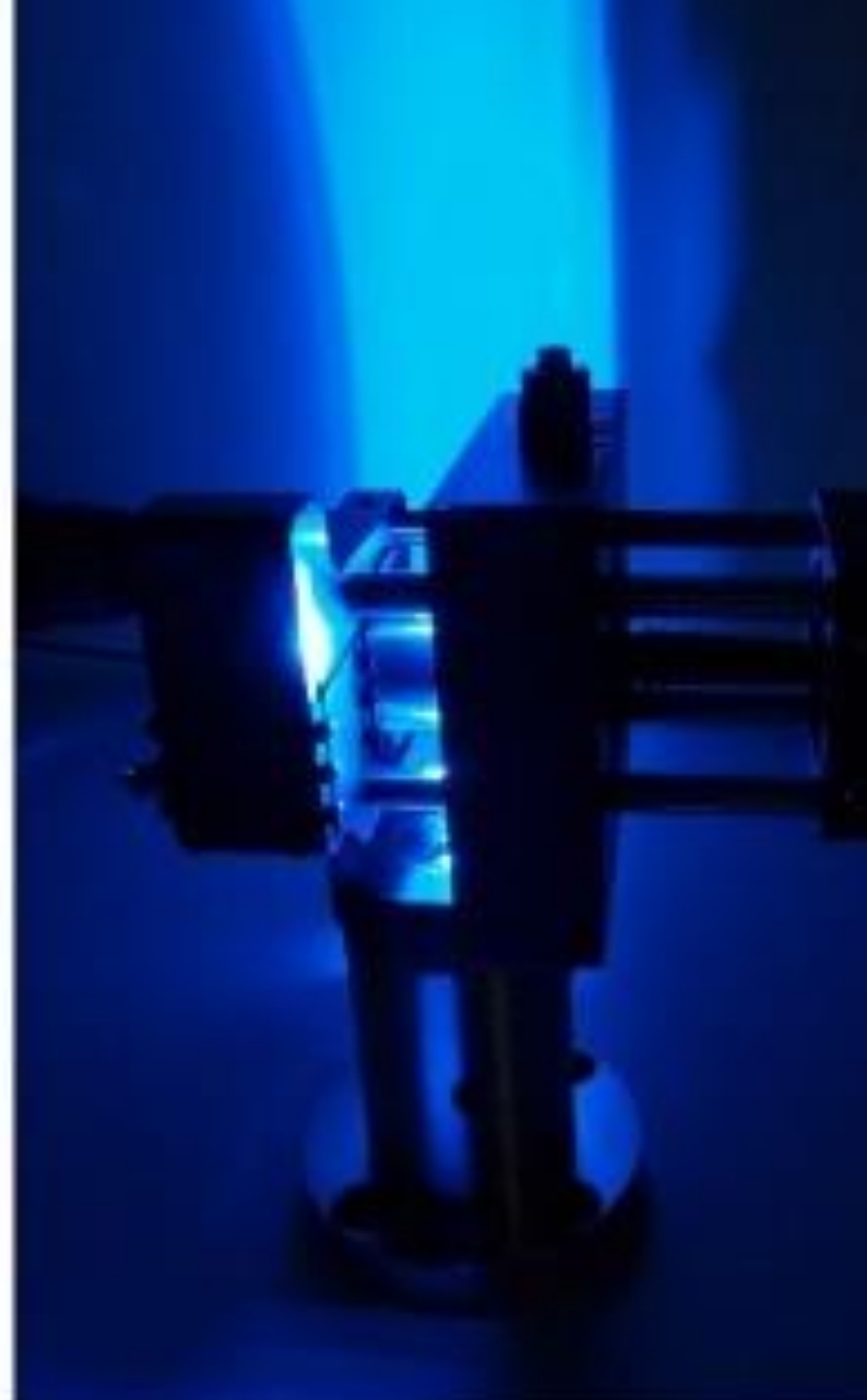
Probencylinder
FRIEDEN

WAGNER

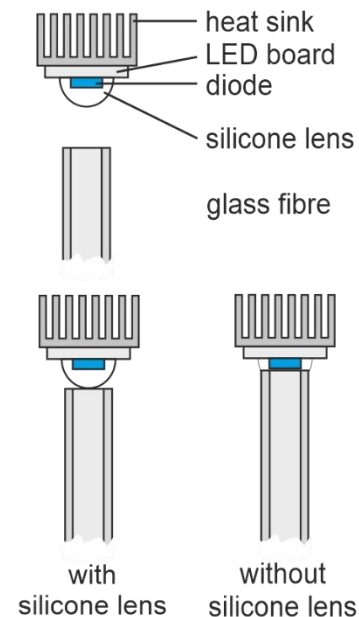
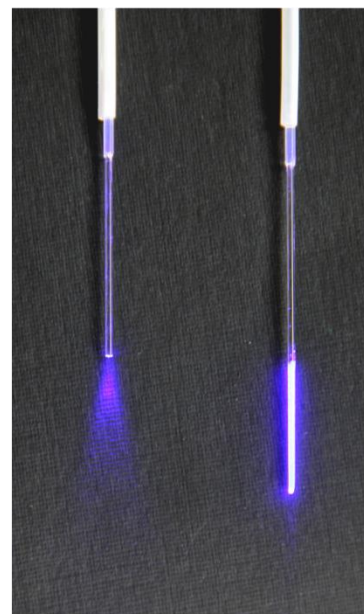
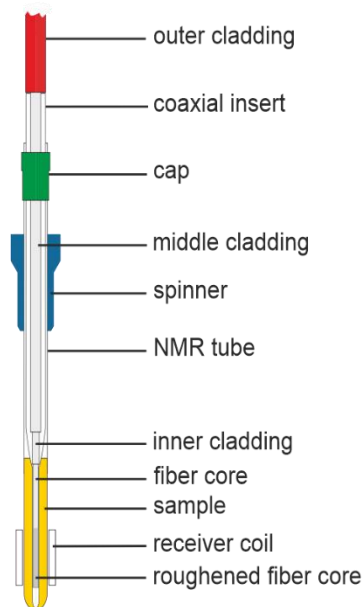
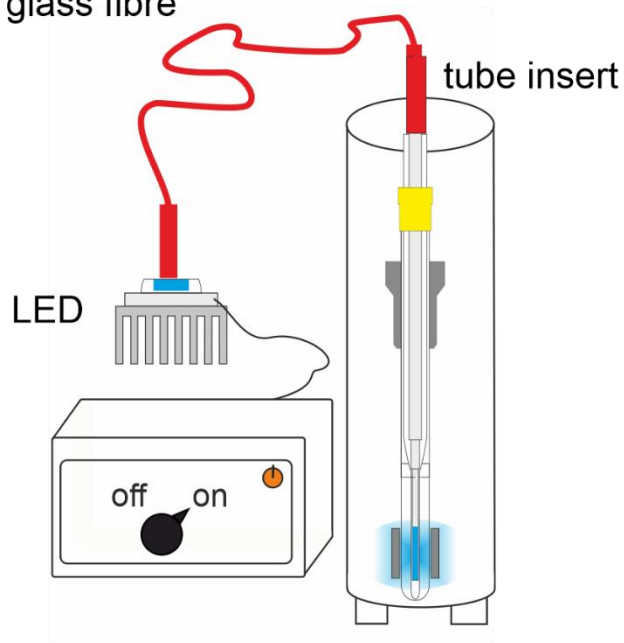
ARTOL 100



Illuminated
NMR



glass fibre

**General:**

- external LED
- glass fibre
- NMR tube insert

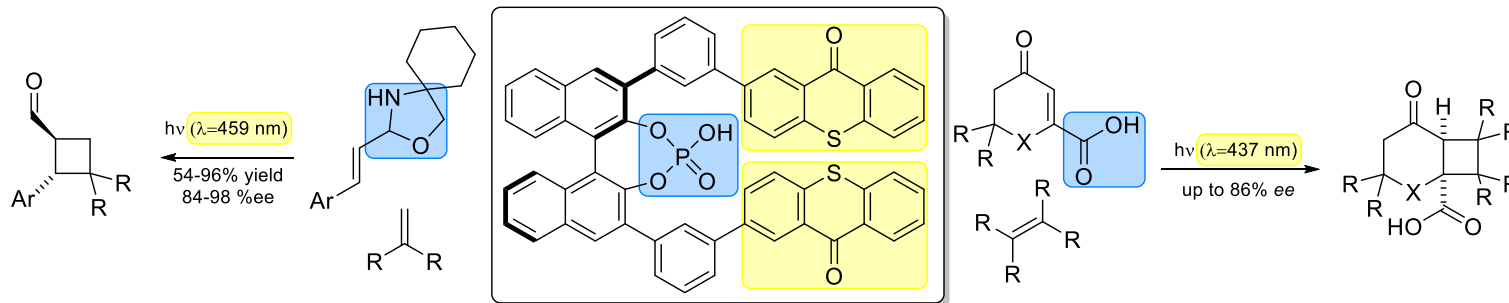
Insert:

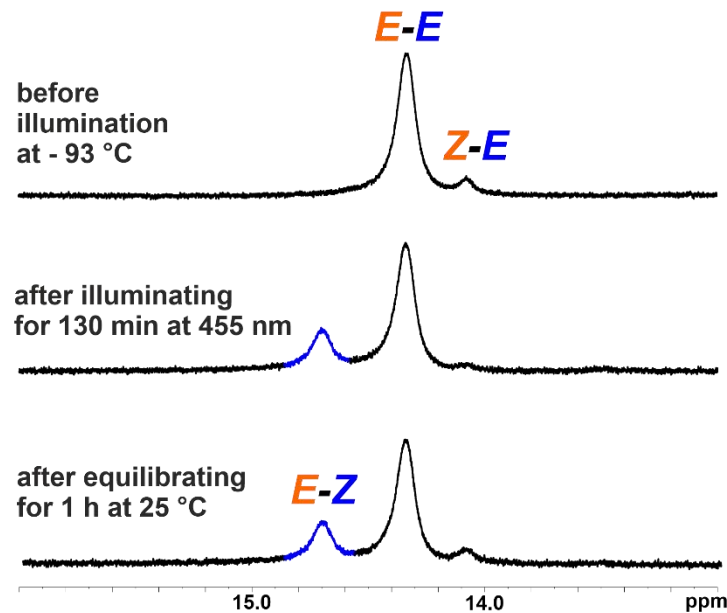
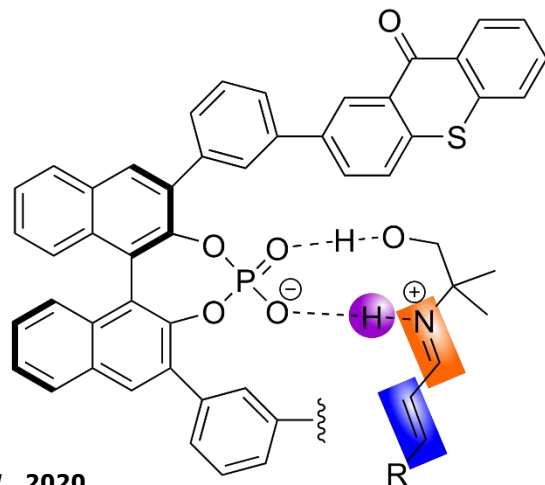
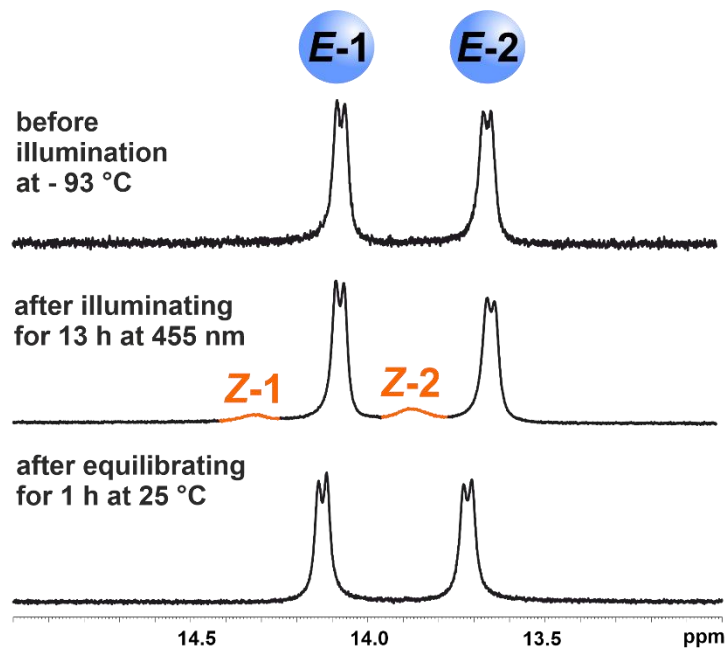
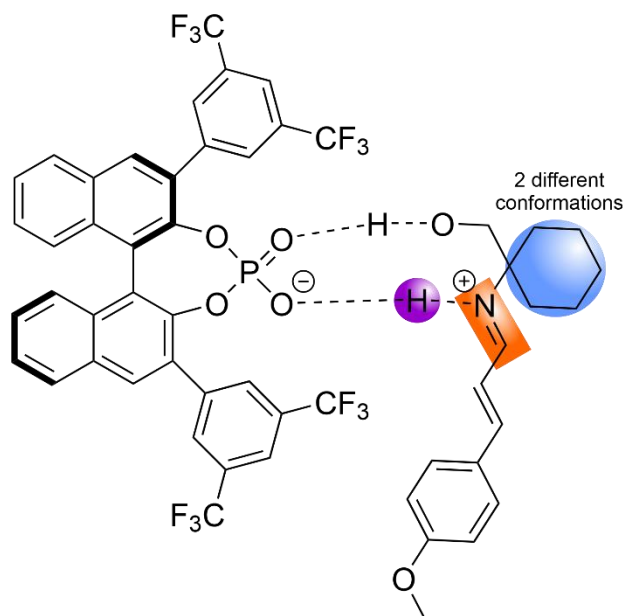
- glass fibre tip
- removed coating
- tube insert

higher light intensities with:

- roughened glass fibre tip
- close contact with LED
- high power light pulses

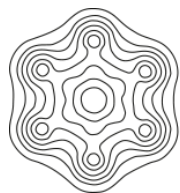
Merging Brønsted acid and photocatalysis: Structural studies and H-bonding







Acknowledgments



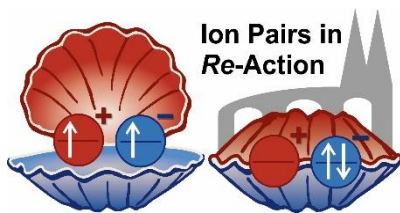
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European
Research
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SPP 1807
Dispersion



GRK
1626



Chemische
Photokatalyse

ASSEMBLY CONTROLLED
CHEMICAL PHOTOCATALYSIS

CRC 325

