UV-vis-NIR spectroscopy in diffuse reflection and FIR-MIR spectroscopy in ATR-technique as valuable tools for thermo- and magnetochemical investigations

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We work with a Perkin-Elmer Lambda 900 UV-VIS-NIR spectrometer with the „Praying Mantis“ ™ accessory allowing the measurement of powder samples in diffuse reflection technique within the temperature range of 110 K to ambient temperature (see Fig. 1a).

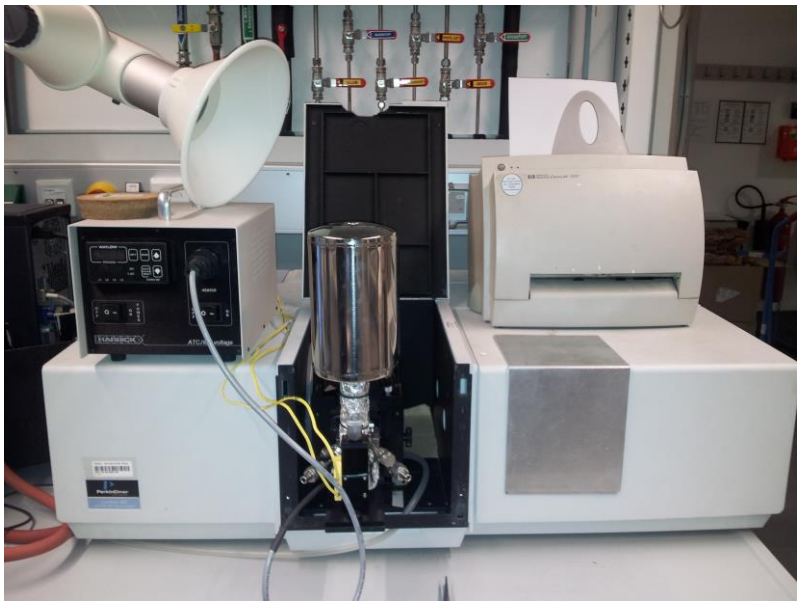

Fig. 1a: UV-VIS-NIR spectroscopy in diffuse reflectance

The first part of this lecture will present our investigations of thermochemical energy storage materials [1] based on *e.g.* transition metal salts forming ammoniates and their *in-situ* characterization by optical spectroscopy of powder samples using the sample chamber as “flow reactor” (see Fig. 1b). The challenges for the measurement set-up will be discussed and possible obstacles and the (partial) solution of problems will be presented.

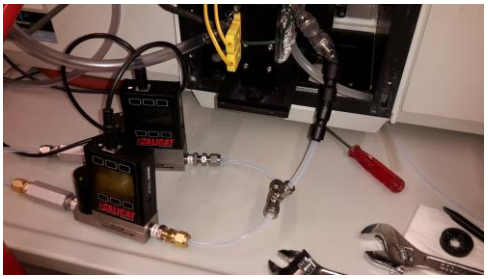

Fig. 1b: Mass Flow Controller for NH_3 and purge gas (N_2) for *in-situ* UV-VIS-NIR spectroscopy in diffuse reflectance

For IR-spectroscopic investigations we work with a Perkin-Elmer Spectrum 400 FIR/MIR spectrometer using a PIKE Gladi-ATR being thermostatable within a temperature range of 100 K to 473K.

The second part will be dedicated to the variable temperature measurements of Fe(II) spin crossover complexes [2].

Fig. 2: Variable temperature FIR-MIR spectroscopy in ATR-technique

These compounds are characterised structurally, magnetically as well as FIR/MIR spectroscopically. Based on quantum-chemical calculations vibrational modes typical for the low spin state of Fe(II) are identified and their measurements at variable temperature is used for a quantitative determination of the spin state ratio at any temperature measured. (see Fig. 2).

References:

[1] M. Deutsch, D. Müller, C. Aumeyr, C. Jordan, C. Gierl-Mayer, P. Weinberger, F. Winter, A. Werner; *Applied Energy*, 183 (2016) 113 – 120.
[2] D. Müller, C. Knoll. M. Seifried, P. Weinberger; *Vibr. Spectr.*, 86 (2016) 198 – 205.