Flow probe for simultaneous *in situ* flow solid-state NMR and UV/Vis spectroscopy using a modified Bruker MAS NMR probe

This operando probe is based on a 7 mm *in situ* flow MAS NMR probe obtained by a Version 2 modification (see Section “flow probe 2”) of a commercial BL-type Bruker MAS NMR probe and 7 mm MAS NMR rotors with a hole at the bottom, earlier utilized for LASER-heated Bruker 7 mm MAS NMR probes. The upper part of the MAS NMR system in **Fig. 1** is a “flow probe 2”. At the bottom, the rotor contains a quartz glass window. At the bottom of the stator, a support for fixing a fiber optics is added (see Fig. 1 in Ref. [1], Fig. 1b in Ref. [2], and Fig. 8 in Ref. [3]). For some applications, a high-temperature 7 mm STD MAS NB NMR probe of type DSI-740 by DOTY Scientific Instruments, USA, was modified in a similar manner ([4] and Fig. 4 in Ref. [5]).

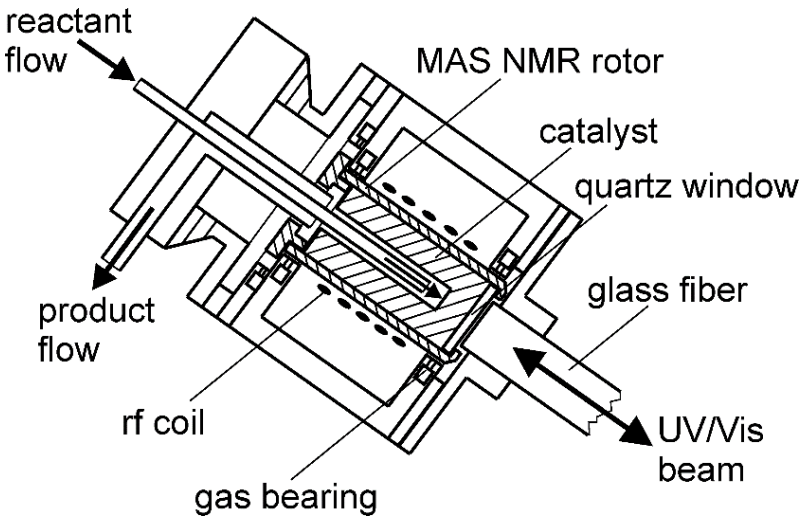

Fig. 1

Before the preparation of the cylindrical catalyst bed using the tool in **Fig. 2**, a size 5.5 x 1.1 mm quartz glass window of type 202-QS delivered by HELLMA (**Fig. 3**) was added at the bottom of the rotor. It is tightly enough that no gas can leave the rotor at the bottom. The preparation and handling of the rotor containing the cylindrical catalyst bed is the same as described in the Section “flow probe 2”.

For recording UV/Vis spectra, an AvaSpec-2048 fiber optic spectrometer and an AvaLight-DH-S deuterium light source were utilized (**Fig. 4**). For *in situ* experiments up to 393 K, a fiber reflection probe FCR-7UV20-3-SR-S1 by AVANTES was applied.

For higher temperatures, the fiber type HPSUV1000A of Oxford Scientific Instruments shown in **Fig. 5** was used (see Section “flow probe 4”). With this equipment, diffuse reflection UV/Vis measurements could be conducted in the spectral range between 200 and 600 nm.

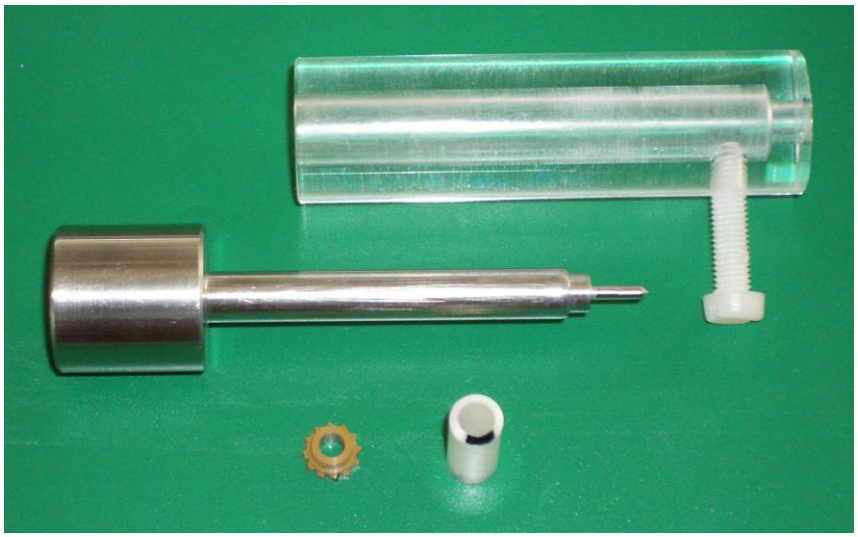

Fig. 2

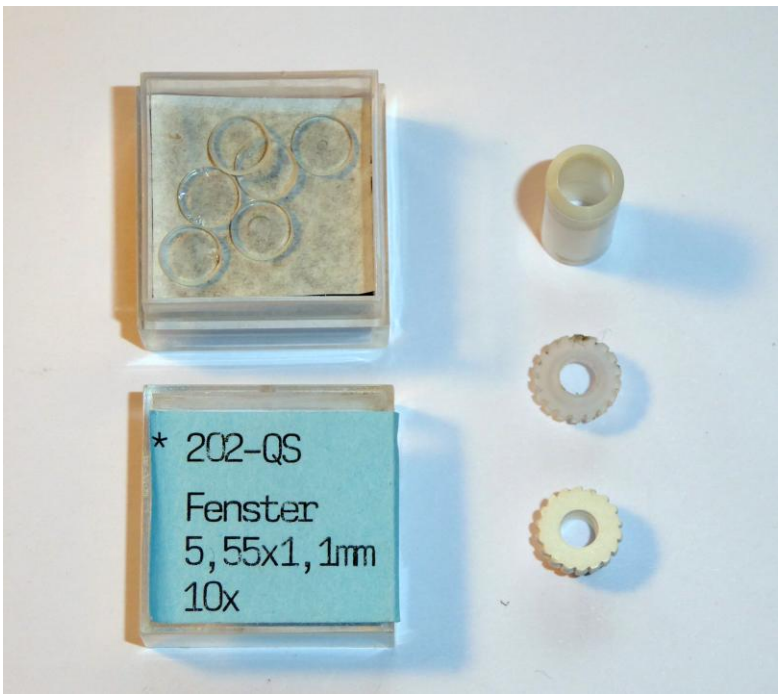

Fig. 3

Fig. 4

Fig. 5

The shape of the fiber support at the bottom of the stator (see **Fig. 6**) depends on the specific type of the MAS NMR probe and the rotor lift system.

Fig. 6

In **Fig. 7**, the installation of the fiber optics at the bottom of the MAS NMR stator is shown (black tube in the centre, reflection probe FCR-7UV20-3-SR-S1). If the fiber optics is not flexible enough to be bent into the magic angle of the MAS NMR probe, a quartz glass rod, which is bent in a suitable angle is added between upper end of the fiber optics and the support at the bottom of the stator.

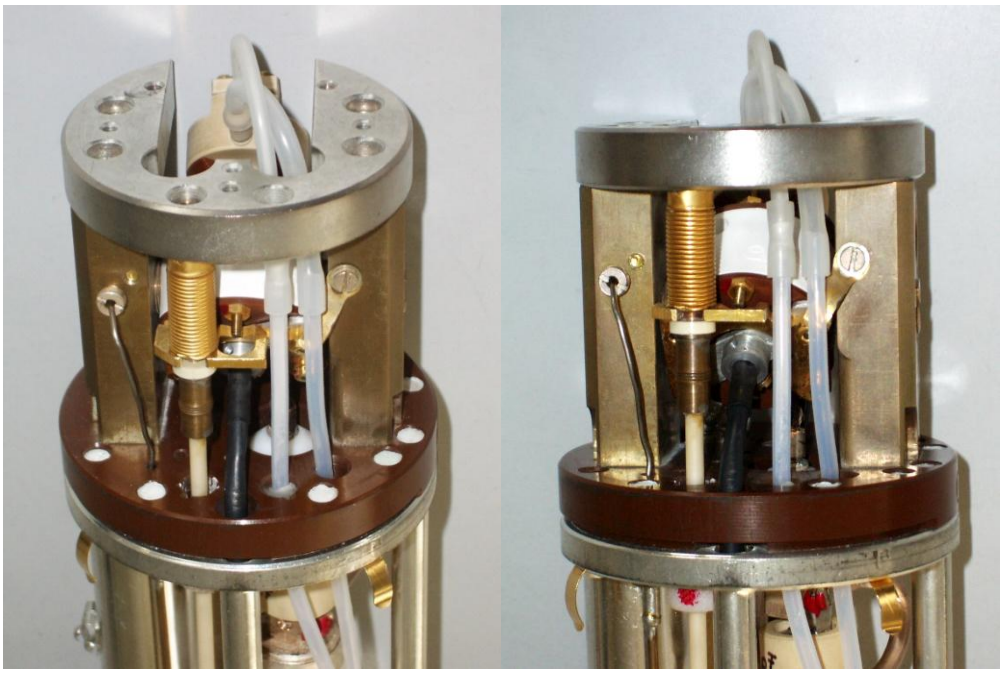

Fig. 7

References:

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[2] Y. Jiang, J. Huang, J. Weitkamp, M. Hunger, *In situ MAS NMR and UV/VIS spectroscopic studies of hydrocarbon pool compounds and coke deposits formed in the methanol-to-olefin conversion on H-SAPO-34*, Stud. Surf. Sci. Catal. 170 (2007) 1137-1144, DOI: 10.1016/S0167-2991(07)80970-0.

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