Flow probe obtained by modification of a 7mm high-temperature MAS NMR probe of Doty Scientific Instruments

For *in situ* MAS NMR studies of heterogeneous reaction systems at temperatures up to 723 K [1-4], a commercial DSI-740 7 mm STD MAS NB NMR probe delivered by Doty Scientific Instruments, Columbia, USA, allowing measurements at temperatures up to 873 K was equipped with an injection system similar to that used for the modification of the Bruker MAS NMR probe described in Section “flow probe 2”.

The DSI-740 probe has a compact stator made by silicon nitride (1 in **Fig. 1**). A 1/16 inch stainless steel tube (2 in **Fig. 1**) is used to conduct the reactant gas to the injection head (3 in **Fig. 1**), which has a shape similar to that shown in Fig. 1 of Section “flow probe 2”. In the first version of the injection system for the DSI-740 probe, this injection head was made by MACOR (glass ceramic) suitable for temperatures up to 1073 K. In the upper part of the injection head, a 2 mm glass tube was fixed by soluble alkali silicate (water glass). The 1/16 inch stainless steel tubes

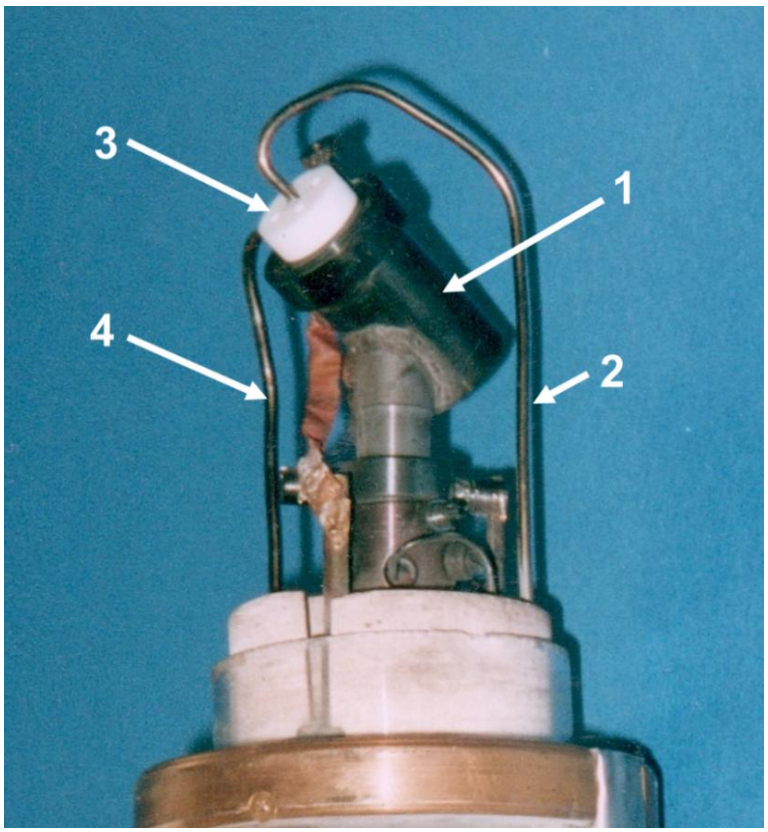

Fig. 1

assigned by 4 in **Fig. 1** is the exhaust tube for the reaction products, which is connected via flexible tubes with an on-line gas-chromatograph (see Fig. 1 in Section “equipment 1”). Also the sealing of the two stainless steel tubes inside the injection head is made by soluble alkali silicate (water glass).

In the second version of the injection system for the DSI-740 probe, the injection head was made by SINTIMID PUR HT of ENSINGER, Germany, suitable for temperatures up to 573 K. This material is easier to handle compared with MACOR, e.g. in the case of making thread holes. **Fig. 2** shows the stator of the DSI-740 probe 1, the reactant gas tube 2, the injection head 3, and the exhaust tube 4. In contrast to the construction shown in **Fig. 1**, the sealing of the tubes inside the injection head was made by high-temperature O-rings and clamping devises containing two small screws. In the subsequent Section “drawings”, the detailed shapes of the support and the additional parts are shown.

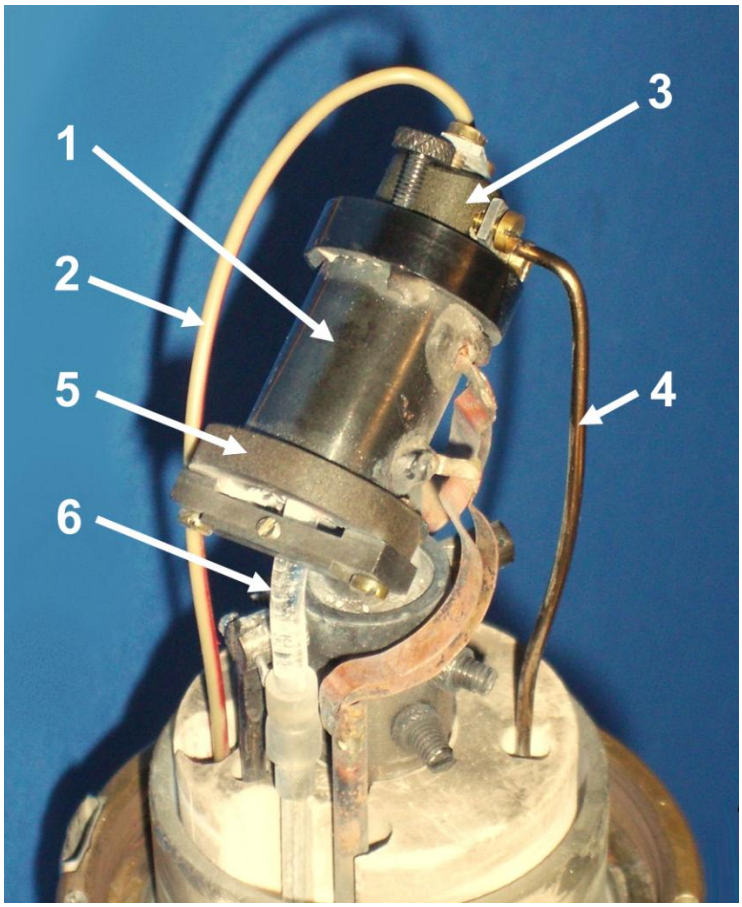

Fig. 2

For *in situ* UV/Vis studies, the stator is equipped at the bottom with a support 5 for fixing a quartz glass stick 6. Also this support was made by SINTIMID PUR HT of ENSINGER, Germany. In a high-temperature flame, the quartz glass stick was bent into the magic angle. This quartz glass stick is connected with a fiber reflection probe. For pressing the shape of the catalyst bed to a hollow cylinder, the tool shown in **Fig. 3** is utilized. At first, the rotor containing a BN rotor insert is loosely filled with catalyst powder without to press it. Subsequently, the rotor is inserted into the acrylic glass part of the tool until the rotor can be fixed by the screw at the bottom. Then, the metal part of the tool is pressed into the catalyst powder inside the rotor. By slow removing this metal tool, a hollow cylinder remains in the catalyst bed, which is required for the insertion of the injection tube.

Fig. 3

If simultaneous *in situ* MAS NMR and UV/Vis spectroscopic studies are planned to perform, the BN rotor insert in **Fig. 3, bottom, middle**, is equipped with a quartz glass windows. In this case, a size 5.5 x 1.1 mm quartz glass window of type 202-QS delivered by HELLMA (**Fig. 4**) is added at the bottom of the BN rotor insert before the preparation of the cylindrical catalyst bed using the tool in **Fig. 3**. The quartz glass

window is placed so tight into the BN rotor insert that no gas can leave the insert 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”.

Fig. 4

For recording UV/Vis spectra, an AvaSpec-2048 fiber optic spectrometer and an AvaLight-DH-S deuterium light source were utilized (**Fig. 5**). For *in situ* UV/Vis studies up to 723 K, a fiber reflection probe HPSUV1000A of Oxford Scientific Instruments was used (**Fig. 6**). With this equipment, diffuse reflection UV/Vis measurements could be performed in the spectral range between λ = 200 and 600 nm.

Fig. 5

Fig. 6

References:

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