Sample tube system for the *in situ* preparation of catalysts in MAS NMR rotors

In this case, the assignment sample tube system means a glass device containing an MAS NMR rotor filled with catalyst, which can be inserted into an activation oven (heater) and simultaneously connected with a vacuum line (**Fig. 1**, Fig. 8 in Ref. [1], Fig. 10 in Ref. [2]). The idea of this device is based on the CAVERN (Cryogenic Adsorption Vessel Enabling Rotor Nestling) system developed by Haw and co-workers (Fig. 1 in Ref. [3]). The device in **Fig. 1** is utilized for the dehydration and activation of catalysts and their subsequent loading with probe molecules or reactants at low temperature. For this purpose, the MAS NMR rotor filled with catalysts is inserted into a small glass tube at the bottom of the device in **Fig. 1**, while the rotor cap is fixed at the bottom of a sealing rod. During the dehydration and activation of the catalyst inside the rotor, the device is inserted into an oven as that shown in Fig. 2, right-hand side, of Section “vacuum line 1”, and connected with the vacuum line. Before starting the dehydration and activation, the sealing rod with the cap (**Fig. 1, top**) is moved upwards, so that the rotor cap is in the room temperature rang of the device, i.e. outside of the oven. After finishing the dehydration and activation of the catalyst, the vacuum valve is closed, the device is disconnected from “vacuum line 1” and connected with “vacuum line 2”, allowing the adsorption of probe molecules or reactants (see Section “vacuum line 2”).

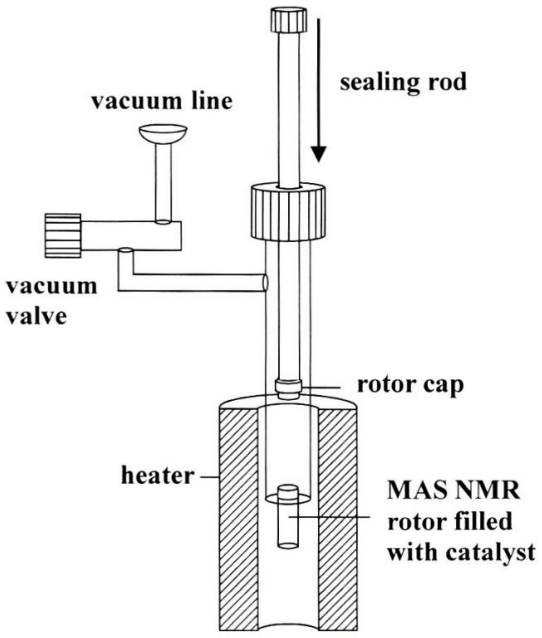

Fig. 1

During the adsorption of probe molecules or reactants, the lower part of device with the rotor containing the catalyst is cooled in liquid nitrogen, if required. After finishing the adsorption of probe molecules or reactants at “vacuum line 2”, the sealing rod with the rotor cap is moved downwards and the rotor cap is pressed on the rotor. Subsequently, the device is opened, the rotor is removed, the cap is additionally pressed into the rotor, and the sealed rotor is transferred into the NMR spectrometer.

The device in **Fig. 2** is made by DURAN glass, the sealing rod by brass. The vacuum valve is of the same type as that used for “sample tube system 1”.

Fig. 2

Three O-rings arranged in three slots of the sealing rod make the device gas tight. These O-rings are moved upwards and downwards with the sealing rod. For decreasing friction between these O-rings and the device, the inner walls of the device are covered with vacuum grease (**Fig. 3**). For the insertion of the rotor into the small tube at the bottom of the device (**Fig. 4, left-hand side**), a common glass tube with an inner diameter slightly larger than the rotor diameter is utilized. By this way, it is easier to insert the rotor into the small device tube at the bottom. Have attention that the rotor does not rapidly fall down into the small device tube. Otherwise, it could be damaged. Therefore, the insertion of the rotor should be performed with the devise in a flat angle and not in vertical position.

Fig. 3

Fig. 4

Reference:

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[2] M. Hunger, J. Weitkamp, *In situ Magnetic Resonance Techniques: Nuclear Magnetic Resonance*, in: B.M. Weckhuysen (ed.), *In situ Spectroscopy of Catalysts*, American Scientific Publishers, Stevenson Ranch, California, 2004, ISBN: 978-1-4020-9678-5, p. 177-218.

[3] J.F. Haw, B.R. Richardson, I.S. Oshiro, N.D. Lazo, J.A. Speed, *Reactions of propene on zeolite HY catalyst studied by in situ variable temperature solid-state nuclear magnetic resonance spectroscopy,* J. Am. Chem. Soc. 111, (1989) 2052-2058, DOI: 10.1021/ja00188a016.