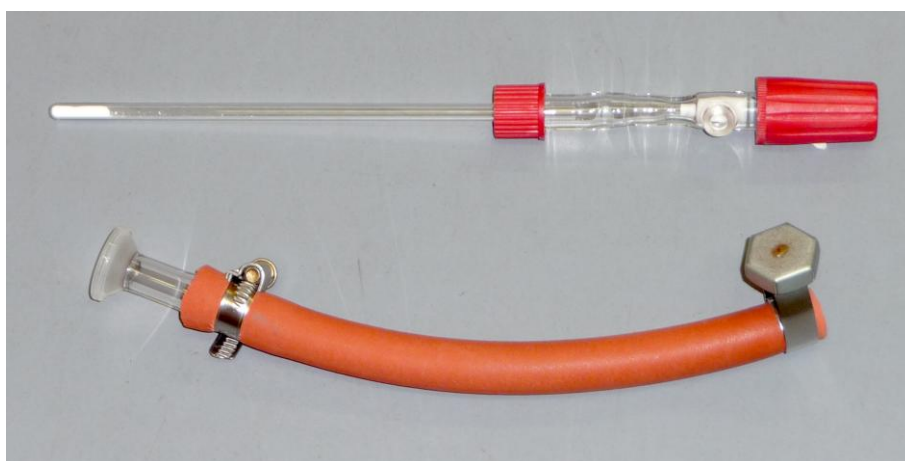


## Sample tube system for the preparation of solid catalysts with changing gas loadings

For static (without MAS) NMR studies and the requirement of changing loadings of molecules on activated catalysts, the sample tube system shown in **Fig. 1** was utilized. This sample tube system can be used, e.g., for PFG NMR studies of molecular diffusion (see e.g. Ref. [1]) and for the sampling of pore systems in solid materials by  $^{129}\text{Xe}$  NMR spectroscopy. The sample tube system in **Fig. 1** consists of a vacuum valve with flexible connector at one side for the insertion of a 6 mm glass tube and a short and cut end for connecting a flexible rubber tube (outer diameter of 18 mm, inner diameter of 8 mm). The cut end at the vacuum valve (**Fig. 2**) is long enough for tightly connecting this rubber tube (**Fig. 3, left-hand side**) during the dehydration and activation of the catalyst samples at a vacuum line, like that described in Section “vacuum line 1”, and for the adsorption of molecules at another vacuum line, like that described in Section “vacuum line 2”. Without the rubber tube, the total diameter of the above-mentioned sample tube system is small enough to fit into the inner room temperature tube of a narrow bore magnet, e.g. for an insertion into a narrow bore PFG NMR probe inside the magnet.

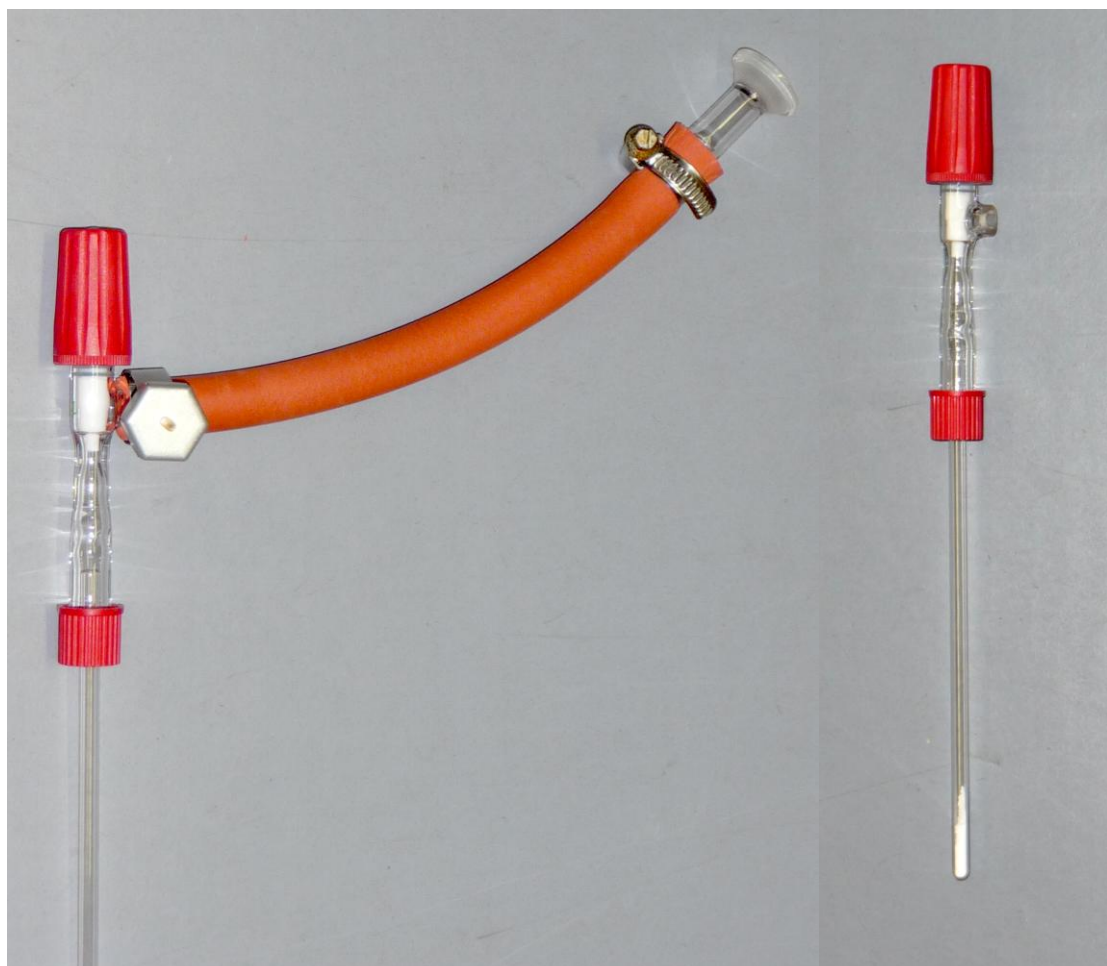


**Fig. 1**



**Fig. 2**

After finishing the loading of the molecules on the activated catalyst, the vacuum valve is closed und the whole sample tube system shown in **Fig. 3, right-hand side**, is inserted into the NMR probe from top of the magnet. This procedure is possible for NMR probes with a radio frequency coil arranged along the z-axis of the magnetic field, such as for the Bruker PFG NMR probe of type Diff-30 [1, 2].



**Fig. 3**

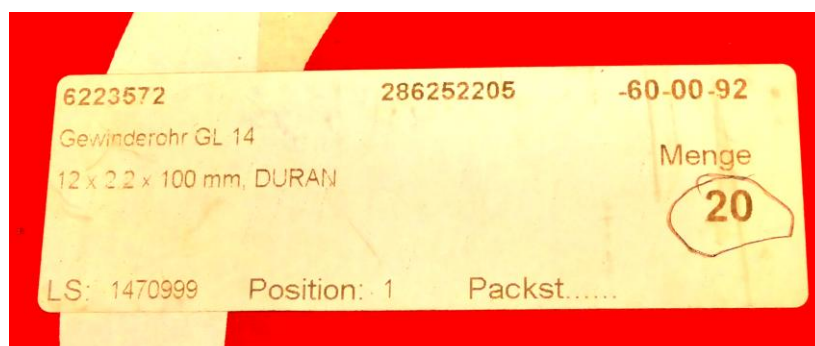
As also described in the Section “sample tube system 1”, the 6 mm glass tubes are made by DURAN glass tubes of SCHOTT-RUHRGLAS (see **Fig. 4**). Commercial DURAN glass tubes are cut in a length of ca. 18 cm and sealed at one of the two sides (**Fig. 1, left-hand side, top**). The length of the tubes must be adjusted in such a manner that the powder samples inside the tubes (dumping height of maximum 2 cm) are in the homogeneous temperature range of the utilized activation oven, if these tubes are connected with a vacuum line. On top of the sample materials inside

the glass tubes, glass wool with a height of ca. 1 cm is added, which hinders that the powder material can leave the tubes under vacuum conditions.



**Fig. 4**

The flexible connectors at the vacuum valves require jointing the threaded tubes shown in **Fig. 4** at one side of the valves. At this side of the valve system, plastic screw caps and O-rings must be added to complete the flexible connectors. The threaded tubes of type GL14 (**Fig. 5**), plastic screw caps for GL14 (**Fig. 6**), and O-rings 12 x 6 mm for GL14 (**Fig. 7**) are delivered by SCHOTT DURAN.



**Fig. 5**



Fig. 6



Fig. 7

For connecting the flexible rubber tubes with a vacuum line, socket members S19 (Fig. 8) are added at one side.

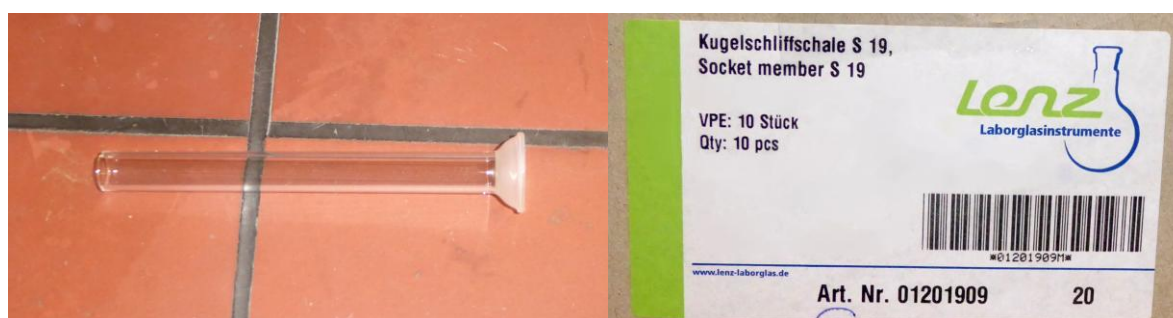


Fig. 8

**Reference:**

- [1] W. Dai, M. Scheibe, L. Li, N. Guan, M. Hunger, *Effect of the methanol-to-olefin conversion on the PFG NMR self-diffusivities of ethane and ethene in large-crystalline SAPO-34*, J. Phys. Chem. C. 116 (2012) 2469-2476, DOI: 10.1021/jp208815g.
- [2] P. Cnudde, E. A. Redekop, W. Dai, M. Waroquier, M. Hunger, L. Li, U. Olsbye, V. Van Speybroeck, *Experimental and theoretical evidence for promotional effect of acid sites on the diffusion of alkenes through small-pore zeolites*, Angew. Chem. Int. Ed. 60 (2021) 10016-10022, DOI: 10.1002/anie.202017025 and Angew. Chem. 133 (2021) 10104-10110, DOI: 10.1002/ange.20201702.