

Sample tube system for dehydration and activation of solid catalysts

For the dehydration and activation of catalyst powders, these materials are filled into glass tubes, which are flexibly connected with a vacuum valve (**Fig. 1**) and a socket, allowing the connection with a vacuum line (Fig. 1 in Section “vacuum line 1”). The valve allows sealing of the sample tubes at the end of the dehydration and activation.

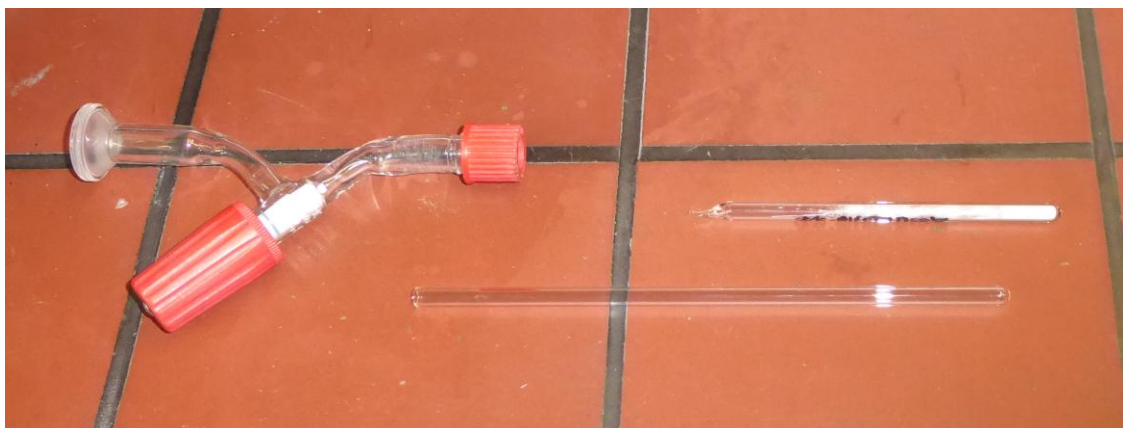


Fig. 1

The sample tubes are made by DURAN glass tubes of SCHOTT-RUHRGLAS with an outer diameter of 6 mm (see **Fig. 2**). Commercial DURAN glass tubes are cut in a length of ca. 18 cm and sealed at one of the two sides (**Fig. 1, bottom**). 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 materials can leave the tubes under vacuum conditions.

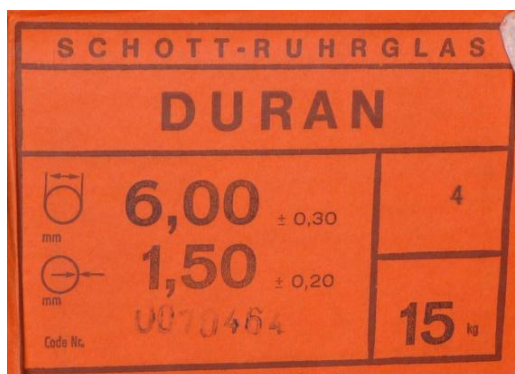


Fig. 2

For connecting the sample tubes with a vacuum line, vacuum valves equipped with a flexible connector at one side and a socket at the other side are used (**Fig. 1, top**). The PRODURAN type vacuum valves are offered by the DURAN GROUP and are delivered as straight valves (**Fig. 3**).



Fig. 3

The flexible connectors require joining 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.



Fig. 4

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

At the second side of the valve systems, socket members S19 are added (**Fig. 8**).



Fig. 8

At the end of the dehydration and activation of the catalyst powders at a vacuum line, such as described in the Section “vacuum line 1”, the vacuum valves at the sample tube systems are closed and the glass tubes are sealed using a gas burner like that shown in **Fig. 9**. Finally, sealed sample glass tubes are obtained like that in **Fig. 1, right**. The gas burner in **Fig. 9** utilizes simple city gas combined with pressed air. The addition of pressed air has the advantage of reaching higher temperatures and makes very sharp flames. Furthermore, this gas burner produces two flames allowing a homogeneous heating of the glass material and sealing easier.



Fig. 9

The samples sealed by this way can be stored for long time until their experimental use. In some cases, the activated catalysts in the sample tubes are immediately loaded with probe molecules, using a vacuum line (see Section “vacuum line 2”). In this case, sealing of the sample tubes is performed after the adsorption step or the loaded samples are directly transferred into MAS NMR rotors inside a mini glove box, like that described in Section “mini glove box”.