

Vacuum line for dehydration and activation of solid catalysts

The vacuum line in **Fig. 1** is made by glass with vacuum valves similar as used for the sample tubes described in Section “sample tube 1”. It has connectors (KS19 with suitable O-ring) for five sample tubes, which are inserted into the activation oven from top. At the right-hand side, there is an additional single connector for special treatments in a separate oven. At the bottom, there are two WEST 2050 or WEST 4400 temperature controllers for the two ovens. The left oven utilized for the simultaneous dehydration and activation of up to five samples was delivered by LabHEAT. On top, it is covered by a self-made thermally isolating metal plat with five holes for sample tubes and one hole for the thermocouple (**Fig. 2**). This oven is used for temperatures up to 723 K, which is the temperature limit of the DURAN tubes used as sample tubes.



Fig. 1



Fig. 2

The vacuum is controlled by an ACTIVE PIRANI GAUGE of type APC-M-NW16 and an ACTIVE PENNING GAUGE of type AIM-8-NW26, both connected to an ACTIVE GAUGE CONTROLLER of EDWARDS. The LEYBOLD vacuum pump of type PT 50 at the left-hand side is connected with the line via a cooling trap. The connection of the pump with this cooling trap requires a flexible metal tube to exclude transfer of vibrations to the glass-made vacuum line. The PT 50 pump system contains a booster pump and a turbo-molecular pump for high vacuum.

The standard activation of solid catalysts starts with an evacuation at room temperature for ca. 10 minutes followed by a temperature ramp from room temperature to $T = 393$ K within 2 hours. At this temperature, the samples are dehydrated for 2 hours. Subsequently, the temperature is increased up to $T = 723$ K within 3 hours and evacuated at this temperature for 12 hours. After this treatment,

the sample tubes are flame-sealed (see Section “sample tubes”) or directly used for adsorption of probe molecules or they are directly transferred into MAS NMR rotors inside a mini glove box purged with dry nitrogen gas (see Section “mini glove box”). Adsorption of probe molecules, such as ammonia, pyridine etc., is performed at the vacuum line described in the Section “vacuum line 2”.

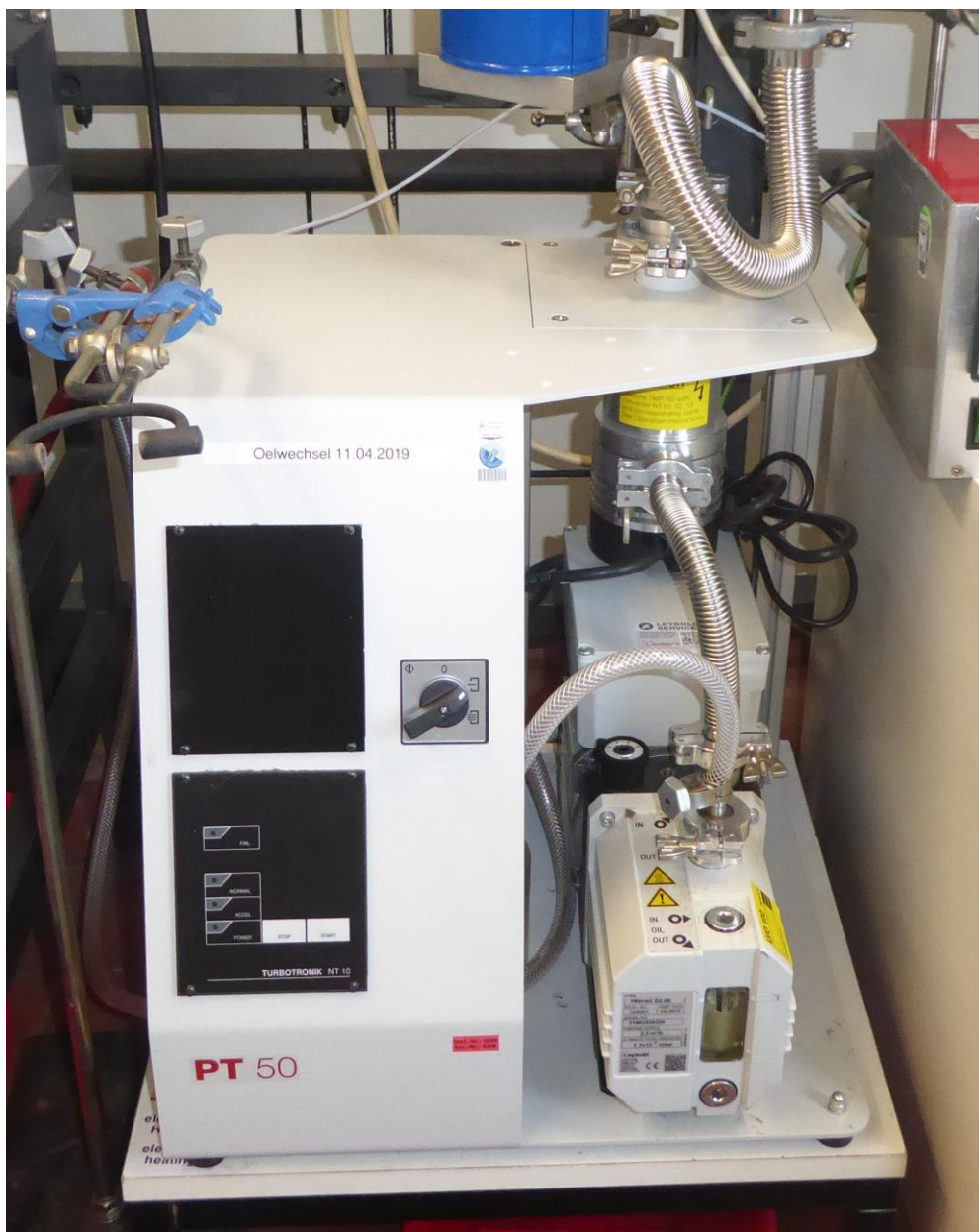


Fig. 3