Set-up for defined steaming of aluminosilicate catalysts and sample transfer

The steaming equipment in **Fig. 1** consists of three parts: The water vapor generator (left), the steaming reactor (middle), and the sample lock (right). The water vapor generator mainly consists of a thermostat with a water bath at stabilized temperatures between T = 399 K and 367 K. A flow of dry nitrogen gas passes the water bath. In this water bath, the nitrogen gas is saturated with water vapor. The water vapor pressure of the nitrogen gas is controlled by the temperature of the water bath. The relationship between the vapor pressure and the water temperature is given in **Table 1**. The steaming of the aluminosilicate samples is performed in the steaming reactor, which is connected to the water vapor generator by a valve at the left-hand side. This valve is used to switch between nitrogen gas loaded with water vapor and dry nitrogen gas at the end of the treatment. The steaming reactor is a quartz glass tube in an oven, which can be heated up to T = 1100 K. To exclude an air contact and uncontrolled hydration of the treated samples after the steaming, the reactor is connected to a sample lock at the right-hand side. By this set-up, the steamed and water-free sample can be transferred into a gas-tight glass container under flowing dry nitrogen gas without air contact.

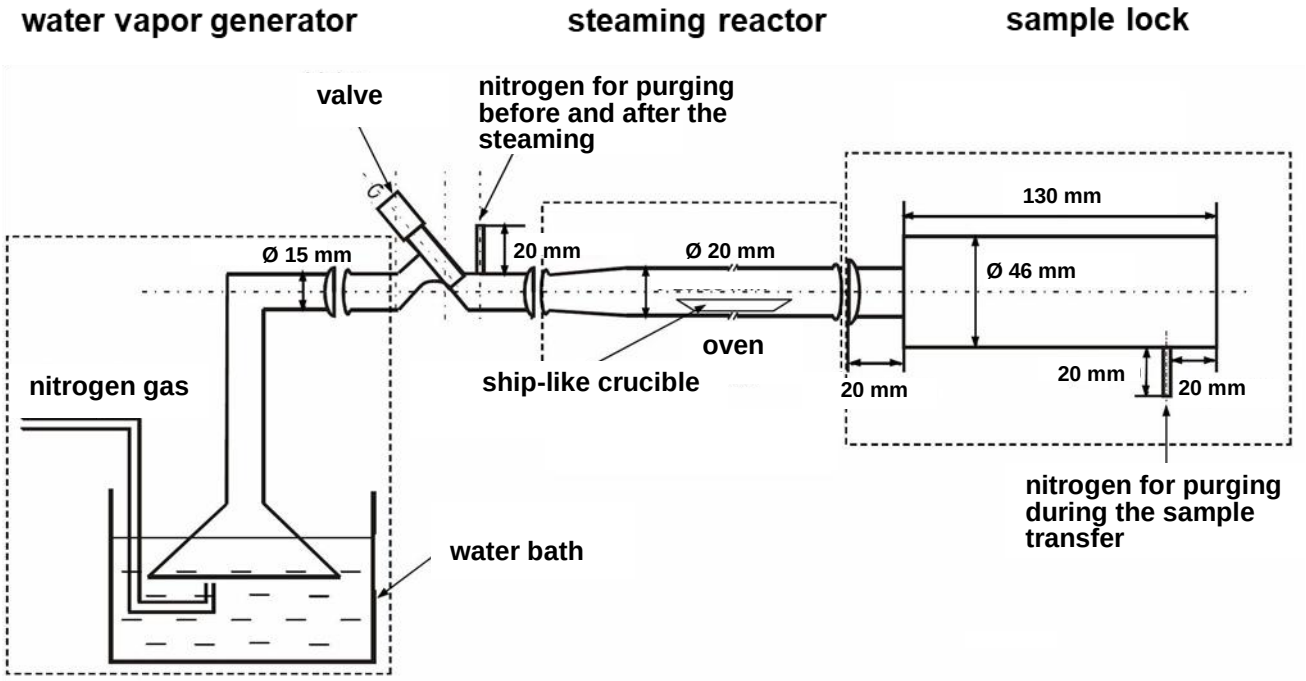

Fig. 1

T_{water} bath / K	299	313	323	333	343	353	363	367
p_{water} vapor / kPa	3.4	7.4	12.4	19.9	31.1	47.4	70.1	81.5

Table 1

For the studies described in Refs. [1], [2], and [3], the dealumination treatment was performed with ca. 2 g zeolite NH_4,Na-Y (cation exchange degree of 93.3%) in a ship-like crucible inside the steaming reactor. For this purpose, the flow of dry nitrogen (200 cm^3/min) was started, and the sample inside the reactor was heated with a rate of 1.6 K/min up to the steaming temperature. At this temperature, at first a calcination was performed for 10 hours leading to a deammoniation of the zeolite and its transformation into the H-form (H,Na-Y). The steaming of the zeolite was carried out at T = 748 or 813 K for 2.5 hours after switching from the gas flow from dry nitrogen to the flow of nitrogen saturated with water vapor. The water vapor pressures of p_{water} = 3.4 to 81.5 kPa were adjusted via the temperature of the water bath according to **Table 1**. After the steaming, the obtained sample was cooled down to room temperature under a flow of dry nitrogen gas. Subsequently, the sample was transferred into the gas-tight glass container inside the sample lock purged with dry nitrogen gas.

Reference:

[1] J. Jiao, S. Altwasser, W. Wang, J. Weitkamp, M. Hunger, *State of aluminum in dealuminated, non-hydrated zeolites Y investigated by multi-nuclear solid-state NMR spectroscopy*, J. Phys. Chem. B 108 (2004) 14305-14310, DOI: 10.1021/jp040081b.

[2] J. Jiao, J. Kanellopoulos, W. Wang, S. S. Ray, H. Foerster, D. Freude, M. Hunger, *Characterization of framework and extra-framework aluminum species in non-hydrated zeolites Y by ^{27}Al spin-echo, high-speed MAS, and MQMAS NMR spectroscopy at B_0 = 9.4 to 17.6 T*, Phys. Chem. Chem. Phys. 7 (2005) 3221-3226, DOI: 10.1039/b508358c.

[3] J. Jiao, W. Wang, B. Sulikowski, J. Weitkamp, M. Hunger, *^{29}Si and ^{27}Al MAS NMR characterization of non-hydrated zeolites Y upon adsorption of ammonia*,

Microporous Mesoporous Mater. 90 (2006) 246-250, DOI: 10.1016/j.micromeso.2005.08.006.