

Quantitative analysis of H₂ isotope sorption in Metal-Organic Framework by cryogenic Thermal Desorption spectroscopy (TDS)

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The cryogenic Thermal desorption spectroscopy (TDS), has been established as a profound technique for quantitative analysis of hydrogen and deuterium through quadrupole mass spectroscopy (QMS). Hence, the precise calibration of QMS using calibration standards is mandatory. Unfortunately, the known calibration standards (titanium hydride, Pd95Y5 alloy) require high temperature conditions (over 800K), which are not available for cryogenic TDS (cryogenic TDS usually allow to operate only below 400 K). In this study, a cryogenic bellows system newly integrated in TDS for low- to high-temperature(20K-1,000K) use is described. This bellow system enables to measure high temperature analysis by detaching cryocooler system. Hence, the combination of techniques allows an in-situ quantitative analysis for hydrogen isotope, and provide very wide temperature range analysis for the investigation of both physisorption and chemisorption. Finally, the performance of the one-step tool setup is illustrated by measurements of a crystalline porous material possessing weak physisorbed site and very strong open metal site.