Evaluation of mass spectra in experiments with deuterium

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For monitoring hydrogen interactions with metals, deuterium is frequently applied as a tracer gas in mass spectrometry as it can be easily resolved from the hydrogen background. In a qualitative analysis, the main peak at mass number 4 may be considered only, while mass numbers 2, 3, 5 and 6 may be ignored. For a quantitative evaluation of reactions where deuterium molecules dissociate, it is important to determine the origin of other peaks, especially mass number 3. To resolve it from an instrumental artefact, a calibration of the instrument by a deuterium flow within reasonable limits is usually performed.

We present the results of calibration procedures of two computer controlled quadrupole mass spectrometer (QMS) instruments mounted on a well outgassed UHV system, realised in a specific way. Each of the QMSs was pumped in line with a turbo molecular pump, but could be also separated from the system by a valve. In a 13 l chamber, pure deuterium, hydrogen or their mixture were prepared and introduced at $1x10^{-2}$ mbar and allowed to leak through a known conductance into the instrument held at high vacuum. The flow changed continuously from 10^{-4} mbar l s^{-1} to 10^{-7} mbar l s^{-1}, typically within one hour. It was determined by monitoring the pressure in the chamber with two calibrated capacitance manometers and by a spinning rotor gauge, mounted close to one of the QMSs. The most valuable data were extracted from the extent and the relative ratio of mass numbers 3, 5 and 6 produced in the QMSs in relation to the previous history.

We made a simple test of the set-up performance after the calibration by the admittance of pure deuterium into the chamber at 10^{-4} mbar while running a hot cathode ionisation gauge (HCG). Deuterium exchange with hydrogen contained in a subsurface layer led in some ten minutes to a noticeable changed proportion of mass numbers 2, 3 and 4 compared to their proportion prior to running the HCG. We could resolve mass number 3 induced by the HCG from the QMS induced background peak. We concluded that with similar QMSs, the extent of deuterium involved in surface reactions can not be determined beyond some limits [1,2]. The origin of this intrinsic inaccuracy lies in their operational principle and remains within some limits indeterminable.

[1] M.G. Rao, C. Dong, JVST A 15, 1312 (1997)
[2] L. Lieszkovszky, A.R. Filippelli, C.R. Tilford, JVST A, 3838 (1990)