



QGA 2.0 : NH₃ Detection in H₂O Vapour

Soft Ionisation for the detection of low concentrations of NH₃ in H₂O Vapour

Introduction

Detection of low levels of NH₃ in H₂O rich gas streams is important in a number of application areas such as:

- ▶ Chemical Industry
- ▶ Energy Production
- ▶ Automotive
- ▶ Refrigeration



When analysing NH₃ in H₂O gas streams the major problem with detection of low levels of NH₃ occurs because of the spectral overlap between the m/z 17 of both water and NH₃. Subtraction of the H₂O background can be performed but increases the uncertainty in the NH₃ concentration results.

One method for deconvoluting the two species is to use soft ionisation. This technique allows users to selectively ionise different gases by setting the ionisation energy for a particular mass. Typically in electron impact ionisation, the excited electrons have energy of the order of 70eV. With Hiden gas analysis systems the electron energy can be altered from 4 to 150eV in 0.1eV increments.

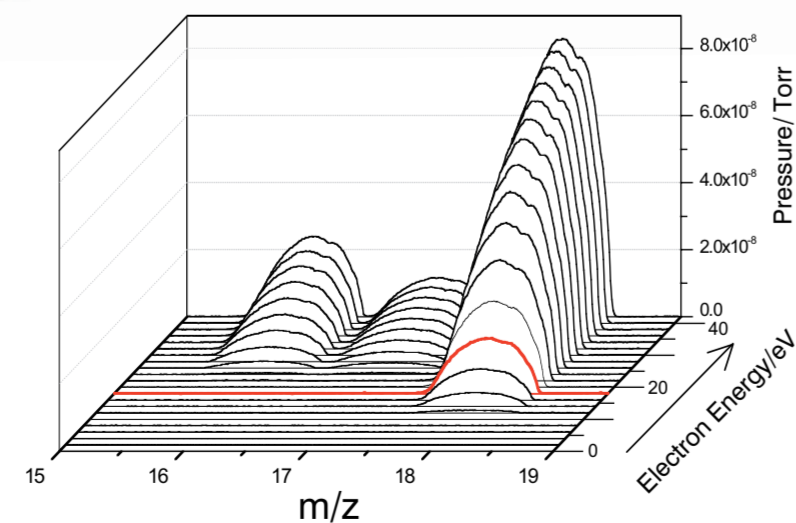


FIGURE 1A : m/z vs Electron energy-H₂O/Air

Application and data analysis: Soft Ionisation of NH₃/H₂O

Figure 1A shows the result of monitoring water vapour in air, scanning between masses 15 and 19 while progressively scanning the electron energy from 10 to 30eV in 1eV increments. This type of scan is easily performed with the Hiden QGA 2.0 using a multi-variant scan as shown in Figure 2.

Figure 1A shows that minimal ionisation of water occurs below 15eV. Above this level, ionisation of H₂O to H₂O⁺ occurs. It can be seen that at 18eV (red) the only ionisation products (m/z 18) detectable when sampling water is H₂O⁺. Ionisation of H₂O to OH⁺ does not occur until around 20eV. Therefore, this difference in ionisation energy can be used to separate OH⁺ and NH₃⁺ if the ionisation threshold of NH₃ to NH₃⁺ is below this level. The ionisation to NH₃⁺ occurs at 11eV. Figure 1B shows the result of scanning between masses 15 and 19 while progressively scanning the electron energy from 10 to 30eV. In this case the Hiden QGA 2.0 is sampling a H₂O/ NH₃ vapour mix. The figure shows ionisation occurring at m/z 17 from around 13eV. Since Figure 1A showed no ionisation at m/z 17 until around 20eV, the species being formed in the ionisation process is NH₃⁺ and therefore NH₃ and H₂O can be separated with the soft ionisation technique using the Hiden QGA 2.0. Continuation of the electron energy scan shows that at 18eV (red) both m/z 17 and 18 can be detected. Therefore, using electron energy of 18eV enables the detection and quantification of NH₃ in the presence of high levels of water.

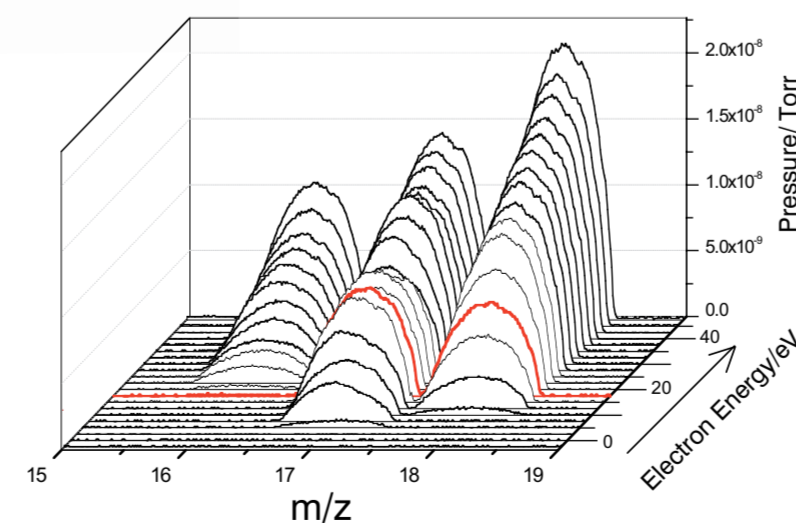


FIGURE 1B : m/z vs Electron energy-NH₃/H₂O/Air mix

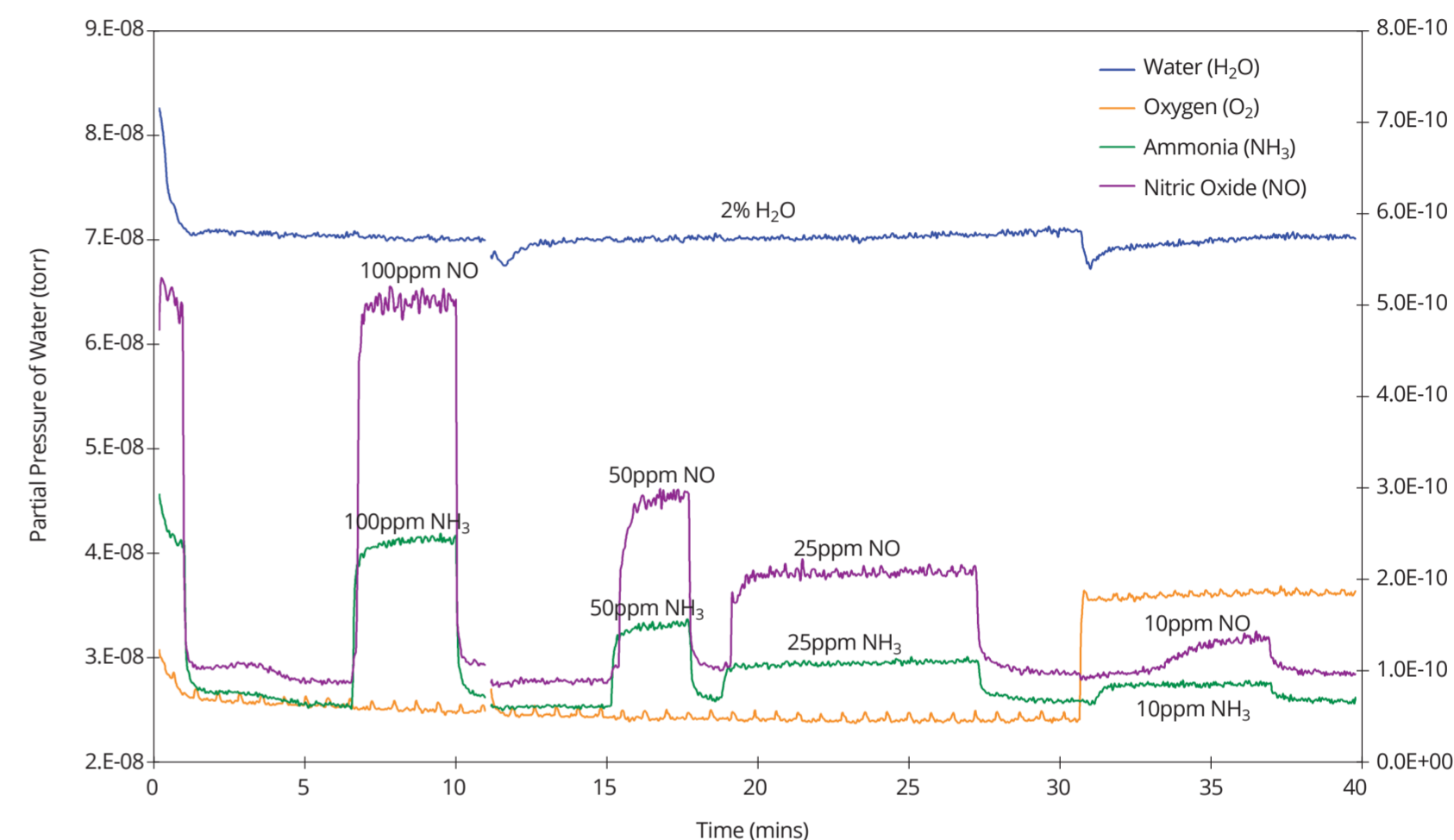


FIGURE 3 : Simultaneous measurement of low ppm levels of ammonia, nitric oxide and oxygen in percentage (2%) concentrations of water

Detection limits

Using the technique described above (i.e. using 18eV as the ionisation energy) the detection limits of this technique were investigated. Here, the QGA 2.0 was connected to a vapour/gas mixing manifold capable of mixing various concentrations of NH₃ in a 2% H₂O stream. The carrier gas used was Argon. Figure 3 shows the results of varying the NH₃ concentration during the experiment between 10 and 100ppm. The results clearly show that detection limits of better than 10ppm NH₃ in water rich gas are achievable.

Conclusion

The data demonstrates the Hiden QGA 2.0 can be used to determine how overlapping species may be separated using soft ionisation techniques. The data also shows how sensitivity remains high even when using reduced electron energy.

Hiden QGA 2.0 software packages allow a variety of different ways to perform soft ionisation scans such as:

- ▶ Multi-variant scans for analysis of changing cracking patterns with electron energy
- ▶ Electron energy scans for determination of ionisation threshold for individual species
- ▶ Global setting of electron energy allowing all masses to be scanned using the same energy
- ▶ Local setting of electron energy allowing different electron energies to be set for individual masses to maximize sensitivity and reduce fragmentation of overlapping species

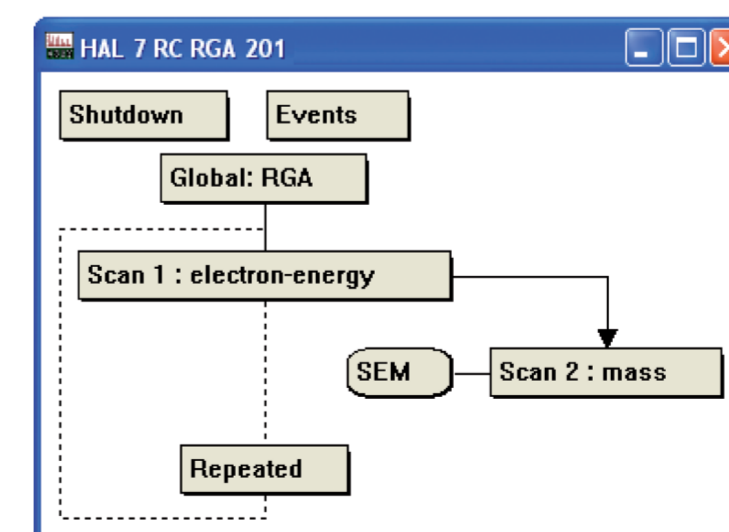


FIGURE 2 : MASoft Multi-variant Scan