Gas Analysis
Application Note 288

NH_3 Detection in H_2O Vapour

Soft Ionisation for the detection of low concentrations of NH_3 in H_2O Vapour

NH_3 Detection Overview.

Detection of low levels of NH_3 in H_2O rich gas streams is important in a number of application areas such as:

- Refrigeration
- Chemical Industry
- Automotive
- Energy Production

When analysing NH_3 in H_2O gas streams the major problem with detection of low levels of NH_3 occurs because of the spectral overlap between the m/z 17 of both water and NH_3. Subtraction of the H_2O background can be performed but increases the uncertainty in the NH_3 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 70 eV. With Hiden gas analysis systems the electron energy can be altered from 4 to 150 eV in 0.1 eV increments

Manufactured in England by:

HIDEN ANALYTICAL LTD
420 Europa Boulevard, Warrington, WA5 7UN, England
t: +44 (0) 1925 445225 f: +44 (0) 1925 416518
e: info@hiden.co.uk w: www.HidenAnalytical.com

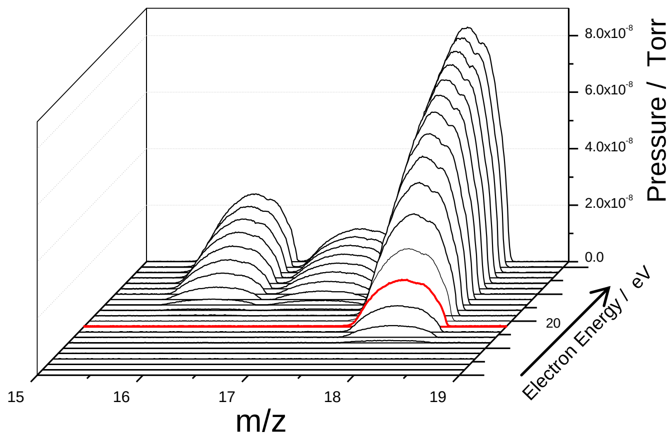

Figure 1 A: m/z *vs* Electron energy-H_2O/Air

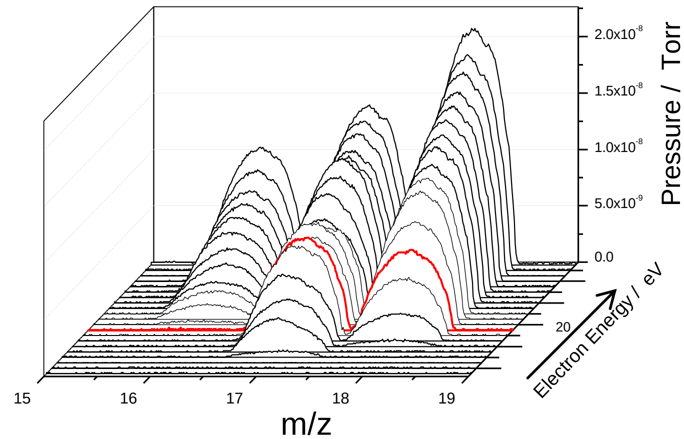

Figure 1 B: m/z *vs* Electron energy-NH_3/ H_2O/Air mix

Soft Ionisation of NH_3/H_2O

Figure 1 A (data plotted using Origin®) shows the result of scanning between masses 15 and 19 while progressively scanning the electron energy from 10 to 30 eV in 1 eV increments. This type of scan is easily performed in MASsoft using a multi-variant scan as shown in Figure 2. In this case the gas sampling is performed by the Hiden HPR-20 gas analysis system monitoring water vapour in air.

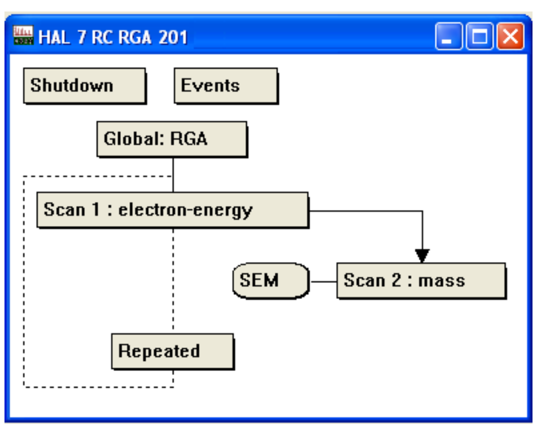

Figure 2: MASsoft Multi-variant Scan

Figure 1A shows that minimal ionisation of water occurs below 15 eV. Above this level, ionisation of H_2O to H_2O^+ occurs. It can be seen that at 18 eV (red) the only ionisation products (m/z 18) detectable when sampling water is H_2O^+.

Ionisation of H_2O to OH^+ does not occur until around 20 eV. Therefore, this difference in ionisation energy can be used to separate OH^+ and NH_3^+ if the ionisation threshold of NH_3 to NH_3^+ is below this level. Theoretically, the ionisation to NH_3^+ occurs at around 11 eV.

Figure 1 B shows the result of scanning between masses 15 and 19 while progressively scanning the electron energy from 10 to 30 eV. In this case the HPR-20 is sampling a H_2O/NH_3 vapour mix. The figure shows ionisation occurring at m/z 17 from around 13 eV. Since Figure 1 A showed no ionisation at m/z 17 until around 20 eV we can establish that the species being formed in the ionisation process is NH_3^+ and means that NH_3 and H_2O can be separated using the soft ionisation technique.

Continuation of the electron energy scan shows that at 18 eV (red) both m/z 17 and 18 can be detected. Therefore, using electron energy of 18 eV enables the detection and quantification of NH_3 in the presence of high levels of water.

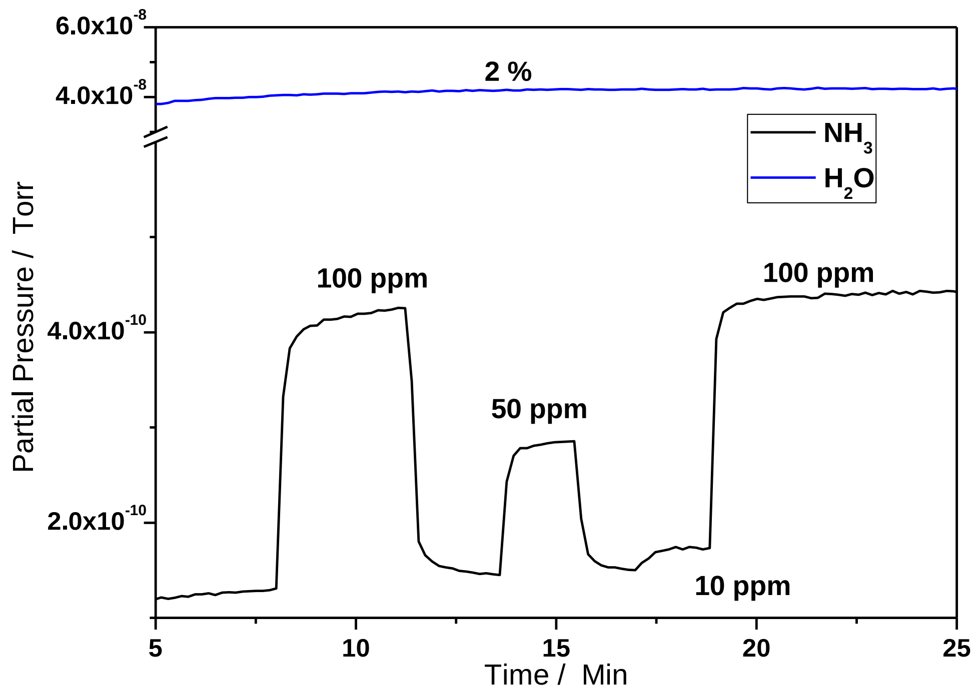

Figure 3: Detection limits of NH_3 in 2% H_2O

Detection Limits

Using the technique described above (*i.e*. using 18 eV as the ionisation energy) the detection limits of this technique were investigated. Here, the HPR-20 was connected to a vapour/gas mixing manifold capable of mixing various concentrations of NH_3 in a 2 % H_2O stream. The carrier gas used was Argon.

Figure 3 shows the results of varying the NH_3 concentration during the experiment between 10 and 100 ppm. The results clearly show that detection limits of better than 10 ppm NH_3 in water rich gas are achievable.

Conclusion

The data presented in this application note demonstrates how the Hiden HPR-20 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.

The Hiden MASsoft software package allows a variety of different ways to perform soft ionization scans such as:

- Multi-variant scans for analysis of changing cracking patterns with electron energy.
- Electron Energy scans for determination of ionization 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.