Gas Analysis
Application Note 252

HPR-20

Detection and Analysis of trace impurities in light gases

Summary

A Hiden HPR-20 - QIC (quartz inert capillary) has been specially modified for the analysis of very low levels of gas and vapour impurities in light gases. This application note describes the changes in pumping configuration made, the preparation for sampling, the measurements performed and the detection limits for a range of gases using the Hiden HPR-20 QIC. The data highlight the ability of the HPR series QMS to perform extremely sensitive measurements and follow trends over time.

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Impurities Analysis

Hiden mass spectrometers are versatile instruments for gas analysis and enable real-time, on-line analysis of gas / vapour mixtures not provided by conventional off-line methods, for instance gas chromatography. However, sensitivity varies depending on the system being examined. In a continuous flow situation, it is difficult to continuously monitor very low levels of impurities.

To obtain parts per billion (ppb) sensitivity levels, 8 to 9 decades difference in signal must be obtained. Hence for a bulk gas flow sampled with the HPR20 - QIC at 10^{-5} Torr levels, then any impurities are present at 10^{-13} to 10^{-14} Torr level. However, this is within the minimum signal level of a secondary electron multiplier (SEM) detector of 2 x 10^{-14} Torr. Hence this kind of application requires a Triple Filter Gauge with Faraday and SEM detectors. This standard configuration allows both the bulk gas and impurities to be monitored simultaneously. If only light elements are to be analysed, a low mass range gauge (e.g. 200 amu), ensuring that the highest RF frequency can be used for maximum sensitivity.

However, to analyse these very low signals, the background signal must be minimised by effective pumping. To address this particular application a change in the pumping configuration was made. The standard Hiden HPR-20 layout was altered by adding a second Turbomolecular drag pump. A dry membrane pump was additionally used as a backing pump. This is shown in schematic form in Figure 1. Turbo pump 2 was also equipped with a purge facility, so that a neutral gas could dilute the bulk gas and improve pumping performance. The capability for lower vacuum pressures was therefore provided, coupled with elimination of any possible oil back-streaming problems, by use of the membrane pump.

Figure 1: Schematic of Pumping configuration.

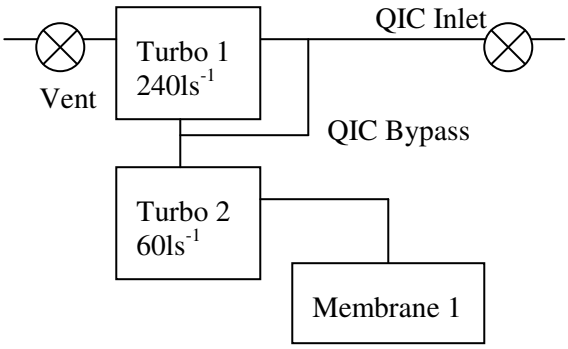
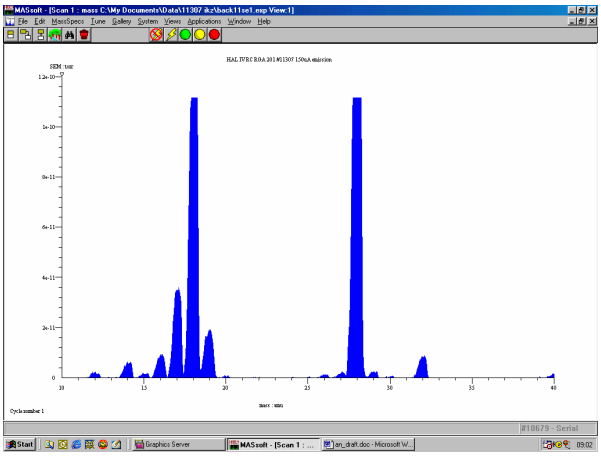


Figure 2: Profile Scan of Background levels



Prior to all analyses the system was baked at 200°C for 16 hours (overnight) to remove any adsorbed gases. This was followed by a profile scan to identify all remaining residual gases, giving the results shown in Figure 2. From this analysis the background contributions for water and oxygen, the major impurities of interest for the intended application, were determined. The water signal was relatively low at approximately 4 x 10^{-10} Torr and the oxygen contribution was even lower at 9 x 10^{-12} Torr. Other atmospheric gases were at similarly low levels, indicating a satisfactory level from which to perform

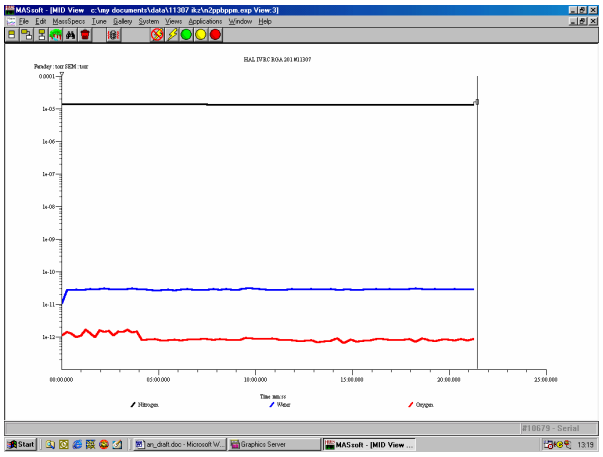
the gas flow experiments.

Firstly an analysis of flowing N_2 was performed. The capillary inlet needle valve was adjusted until the pressure reading from the chamber Penning gauge was 4 x 10^{-6} Torr. This was equivalent to approximately 1 x 10^{-5} Torr at the source as determined by MASsoft. This source pressure will depend on the bulk gas being analysed, as the source will have a slightly different sensitivity for each.

Where possible soft ionisation was used to facilitate species discrimination and ensure accurate identification. Further details can be found in Hiden Application Note 250.

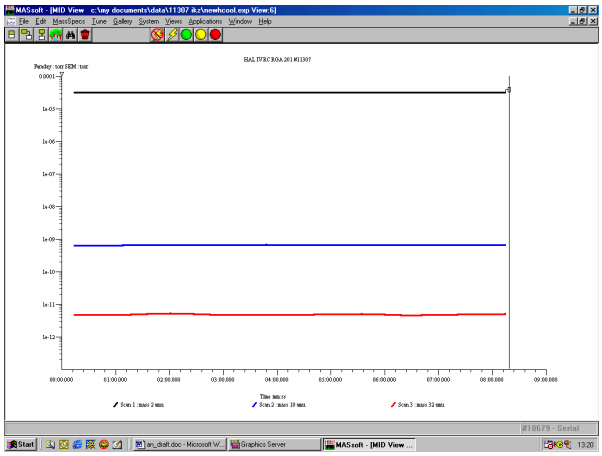
A graph of the impurities is shown (on a logarithmic scale) in Figure 3. Water was detected at a level of 2.1 ppm, which was satisfactory. However, the lower limit of oxygen detected was a more disappointing 68.3 ppb.

Figure 3: Raw Data of H_2O and O_2 content in N_2.



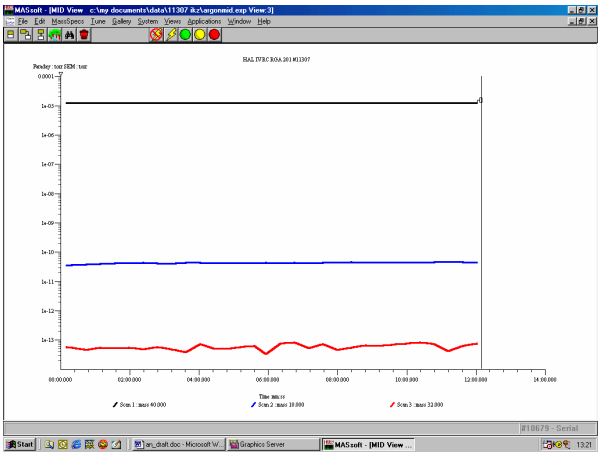
It appeared that this oxygen was in the gas, as changes in pressure had a corresponding effect of the impurity level. Hence a different gas was selected for further investigation. Hydrogen is of particular interest due to it being a light gas. Gas was flowed to allow the gauge to stabilise with this different bulk gas and levels of oxygen and water again measured. Figure 4 shows the content levels of these impurities. As can be seen the initial measurement could only achieve 35ppm water and 75ppb oxygen, again reflecting problems with gas purity.

Figure 4: Raw Data of H_2O and O_2 content in H_2.



The third gas to be analysed was Argon which was again introduced to the gauge. Figure 5 shows the content levels of the impurities of interest. This was the most promising result with levels of 3.5 ppm and 4.9 ppb being measured for water and oxygen respectively.

Figure 5: Raw Data of H_2O and O_2 content in Ar.



Not only was the ability of low level detection demonstrated, but also detection of small changes in the concentration of oxygen could also be

performed. An oxygen trap was placed in-line with the gas flow, prior to the HPR20 – QIC to allow the oxygen level to be effectively increased and decreased. When this experiment was run, changes of ±20 ppb were detected depending on whether the oxygen trap was inline, or bypassed. Clearly this demonstrated sensitivity to the impurity levels at these low concentrations.

Results for the three gases analysed are summarised in Table 1.

Table 1: Comparison of H_2O and O_2 impurity levels.

Gas / Pressure	**H_2O (18) Pressure**	**ppm**	**O_2 (32) Pressure**	**ppb**
N_2 / 1.39×10^{-5}	2.9×10^{-11}	2.1	9.5×10^{-13}	68.3
Ar / 1.24×10^{-5}	4.3×10^{-11}	3.5	6.0×10^{-13}	4.9
H_2 / 3.35×10^{-5}	1.2×10^{-9}	35	2.5×10^{-12}	76

Hence the different gases show a range of content levels. For the lowest level of detection, a profile scan was performed. The peak for the oxygen could clearly be distinguished from the background. Hence the resolution was better that ±5 ppb.

One possible improvement if different gases are being analysed and changes between them are frequent is to select a different source for the gauge. A standard enclosed source was used to collect the data above in Table 1.

An open source significantly lowered the time required for conditioning between different gases. This is because there is an improvement in pumping on the source, which outweighs the better gain from the enclosed course. The compromise is that the gas throughput is higher, so this may be an issue if only small amounts of gas are available for study, or in a production environment where yield is important.

Data using an open source is seen in Table 2.

Table 2: Comparison of H2O and O2 impurity levels.

Gas / Pressure	**H_2O (18) Pressure**	**ppm**	**O_2 (32) Pressure**	**ppb**
Ar / 7.53×10^{-6}	4.0×10^{-11}	5.1	6.3×10^{-14}	8.0

Detection limits are comparable to the enclosed source.

Conclusions

With the appropriate instrument configuration for effective pumping and resolution and using the MASsoft software, levels of impurities in light gases can be achieved in the low ppb range. Following a profile scan, where the signals of interest can be identified, monitoring in MID mode is a convenient way to follow the level of the impurity over time.

The ion source can be selected to have either good sensitivity and a small throughput, or comparable sensitivity and faster conditioning to different gases.