Hiden Analytical Ltd.
420 Europa Boulevard
Warrington WA5 7UN
England

T +44 [0] 1925 445 225
F +44 [0] 1925 416 518
E info@hiden.co.uk
W www.HidenAnalytical.com

NO_x Detection

The detection of NO and NO_2 using the HPR-20

Summary

The Hiden HPR-20 gas analysis system has been used in a variety of applications in the fields of catalysis and thermal analysis. One important aspect of catalysis research is the reduction of NO_x in such applications as car exhausts and power plants. This research involves many challenges in meeting current emission legislation whilst keeping catalyst cost low. Several NO_x reduction technologies have emerged in previous years, some of which have now been commercialized including selective catalytic reduction (SCR) with NH_3, urea and hydrocarbons, and NO_x storage and reduction (NSR). NSR technology consists of two cyclic steps that occur on a lean NO_x trap (LNT) catalyst. The LNT catalyst readily stores NO_2 as compared to NO [1,2]. For this reason, NO should be oxidized to NO_2 to achieve an acceptable level of NO_x storage.

This application note describes the measurement of NO_2 using the m/z 46 peak and the NO/NO_2 ratio in a research application using the Hiden HPR-20. The data highlights the ability of the QIC series gas analysis systems to deconvolute and quantify low levels of NO in high concentrations of NO_2.

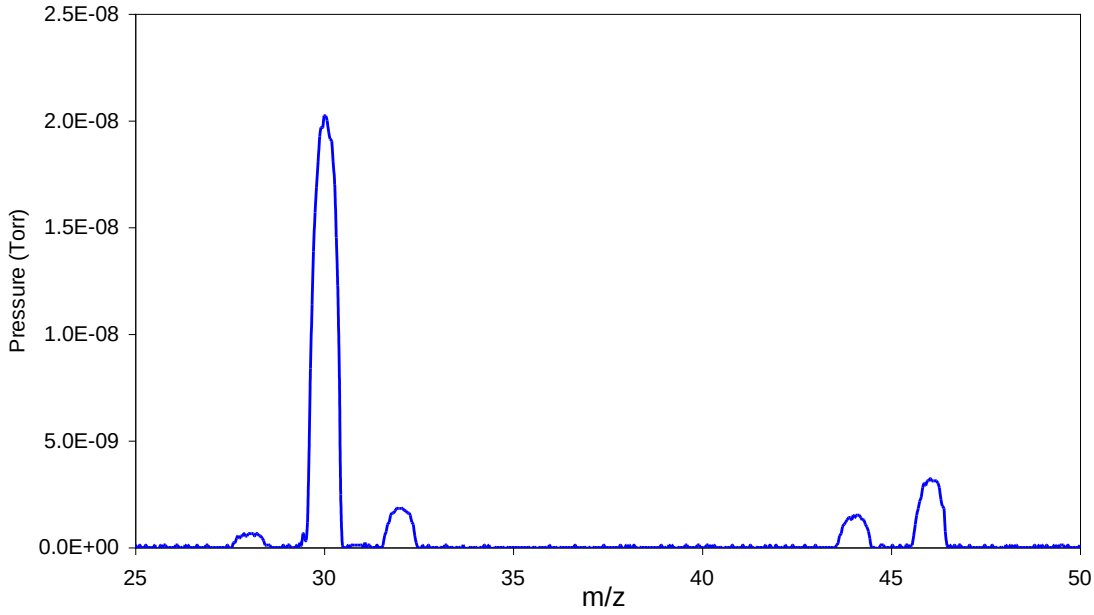

Figure 1: Profile scan for 10000ppm NO_2.

Introduction

The Hiden HPR-20 gas analysis system is configured for continuous analysis of gases and vapours.

The Hiden QIC quartz-lined sampling interface operating up to 200°C provides fast response times of less than 300 millisecond for most common gases and vapours, including water vapour. For this particular application note, the HPR-20 system had the following specifications:

i) QIC capillary inlet with 2 metre heated capillary, operating up to 160°C
ii) Quadrupole probe with 100 amu mass range capability.
iii) Triple-stage mass filter
iv) Corrosion resistant pumping system

Test Data

Figure 1 shows the result of measuring NO_2 in a carrier gas of He. It clearly demonstrates that a distinct peak at m/z 46 can be measured using the HPR-20 system.

This is important as it is known that NO_2 rapidly and easily fragments to NO^+ making analysis of any NO_x mixtures containing NO and NO_2 difficult to deconvolute thereby increasing the uncertainty in the NO and NO_2 concentration results. Assuming the major peak at m/z 30 represents 100%, the peak at m/z 46 is equivalent to 15% giving a 46/30 ratio of 1:6.6.

Another potential problem of the facile fragmentation of NO_2 to NO^+ is the measurement of low concentrations of NO in a high background of NO_2 (a vital measurement in developing NO_x reduction technologies such as the LNT catalyst). The NO_2 produces a high background at m/z 30, which could potentially mask any signal due to the NO in the gas mixture. Figure 2 shows an example MID scan for a range of different NO concentrations measured at m/z 30 with a background level of 1000ppm NO_2. It can clearly be seen that, for the Hiden HPR-20 system, detection limits better than 12ppm are achievable.

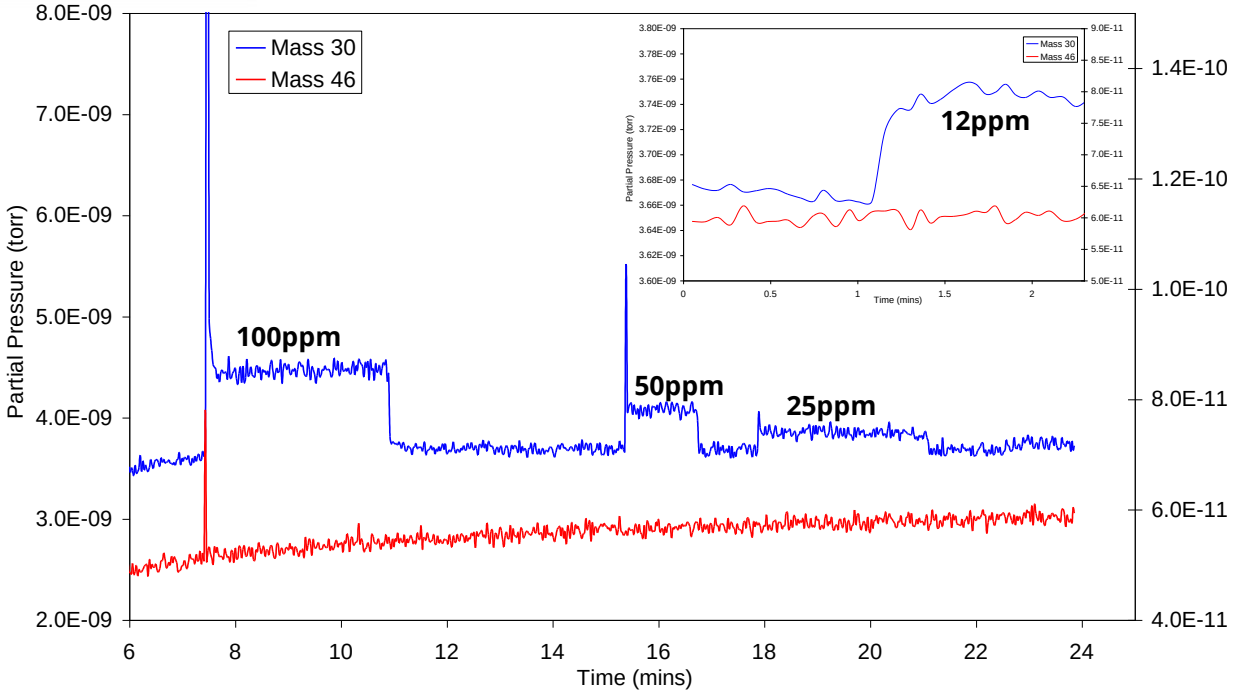

Figure 2: Detection Limit of NO in 1000ppm NO_2 (Inset: Expansion of detection of 12ppm NO in 1000ppm NO_2). Primary Axis represents Partial Pressure at m/z 30, Secondary Axis represents Partial Pressure at m/z 46.

Correlating the NO peak signal intensity with the NO concentration level provides quantitative information on the NO detection levels as shown in Figure 3. The high degree of linearity indicates a high level of confidence in the detection of ppm levels of NO in a high background of NO_2.

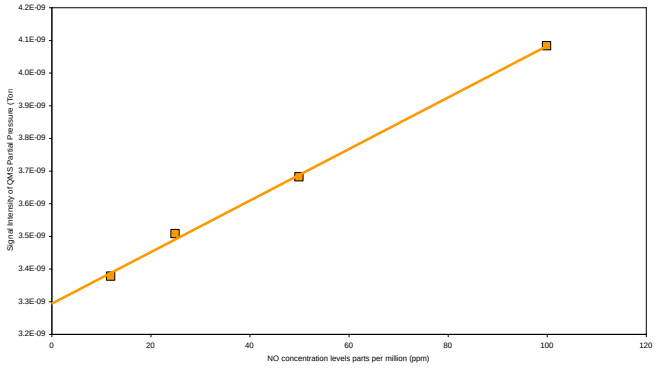

Figure 3: Relationship between partial pressure for m/z 30 and NO concentration (Note: The background at m/z 30 due to NO_2 has been subtracted).

Conclusions

- The Hiden HPR-20 shows high sensitivities for NO_2 detection using the m/z 46 peak despite a large degree of fragmentation.
- Low levels of NO detection in a high background of NO_2 can be achieved without the requirement for other additional or tandem techniques.
- The specific data as detailed in this application note shows detection levels better than 12ppm of NO are attainable – a significant benefit of the Hiden QMS system.

References

[1] S. Erkfeldt, E. Jobson, M. Larrson, *Top. Catal.*, 2001, 16/17, 127.
[2] W.S. Epling, L.E. Campbell, A. Yezerets, N.W. Currier, J.E. Parks II, *Catal. Rev.*, 2004, 46, 164