APPLICATION NOTE



Analysis of NMP in AVTUR by GC Heart-Cut

- No Sample Preparation/Extraction
- No Jet Fuel matrix interference
- High Sensitivity, Linearity, Accuracy and Precision
- Analysis within 10 minutes

Keywords: NMP, AVTUR, GC Heart-Cutting, Deans-Switching, FID-detection.



INTRODUCTION

n-Methyl-Pyrolidone (NMP) is known in the petrochemical business for its excellent extraction properties. It is widely used in various product processes for lubes, reformates and other products. Even though not regulated by method or government, product properties may be affected by traces NMP, and as such it is a parameter that may be monitored by industries using NMP on a large scale. With the risks involved a particular market of interest is jet fuel, which is addressed in this note.

Solution

PAC has developed a solution for determining NMP in Jet Fuel, without the need for the usual sample preparation. The system may also be used for extracts from other matrices.

The core of the system is a Deans heartcutting setup, which allows combining with the PAC FAME in Jet analytical system.

Accuracy and Precision

Figure 1 shows a chromatogram of an AVTUR sample that was spiked with NMP at a level that is close to the established limit of quantification of 0.9 mg/kg. Analysis of this validation sample yielded a result of 1.12 mg/kg which only deviates by 0.09 mg/kg to the gravimetric target value of 1.21 mg/kg. Repeatability on the 1.21 mg/kg validation sample is shown in figure 2. The RSD (n=10) found was 3.2%. At higher NMP content this percentage will drop to even lower values.

1 mg/kg NMP in AVTUR Matrix 11.5 11.4 11.3 11.2 11.1 NMP 11 10.9 10.8 10.7 10.6 10.5 0 2 4 6 10 8

Figure 1. Chromatogram of 1 mg/kg NMP in AVTUR Matrix



Figure 2. Precision of Analysis at 1 ppm level





Linearity

As shown in figure 3, linear behavior is achieved over at least a range of 1 - 1000 mg/kg NMP. The peak for 1000 mg/kg still shows good symmetry and does not show any retention shift.

Matrix Interference

Injection of several AVTUR samples did not show any matrix interference with the analysis of NMP. This is depicted in figure 4 where the chromatograms of NIST samples 1617A and 1616B, as well as a jet fuel taken from a FAM round robin, are shown. No peaks are visible where normally NMP would elute.



Figure 3. Calibration Curve of NMP in AVTUR



Figure 4. Chromatograms of several AVTUR Samples

| LOQ (10*N) | Below 1.0 mg/kg |
|-------------------------|------------------|
| Quantification Range | 1.0 – 1000 mg/kg |
| Matrix | AVTUR fuel |
| Precision | Below 5 % at LOQ |
| Analysis Time | < 10 minutes |
| RT Stability | Below 0.1 % RSD |

CONCLUSION

Gas chromatography using Deans switch heartcutting technology provides a rugged, fast and reliable way of determining NMP-traces in aviation turbine fuels. The high selectivity of the 2nd dimension column towards NMP allows for very broad heart-cuts on the 1st dimension column which adds to the stability of the system

Sample treatment can be eliminated and the system can be calibrated on a single external standard, making it a more user friendly solution that may even be combined with PAC's unique Fame in Jet solution.

AC Analytical Controls® has been the recognized leader in chromatography analyzers for gas, naphtha and gasoline streams in crude oil refining since 1981. AC also provides technology for residuals analysis for the hydrocarbon processing industry. Applications cover the entire spectrum of petroleum, petrochemical and refinery, gas and natural gas analysis; ACs Turn-Key Application solutions include the AC Reformulyzer ®, DHA, SimDis, NGA, Hi-Speed RGA and Customized instruments.



00.00.170 2012/1 - Copyright 2012 PAC L.P. All rights reserved