Determination of Trace Level Total Nitrogen content in aromatic hydrocarbons according ASTM D7184

analyzer

TN

7000

Introduction

The Total Nitrogen content in liquid hydrocarbons and aromatics like benzene plays an important part in the manufacturing process of these chemicals and is essential for adherence to stringent pollution laws. The Total Nitrogen content has also been found to be critical to the effectiveness of catalysts and shortens their lifespan, which affects the cost of operations dramatically. This causes problems for petrochemical plants which have moved into the production of light products whereby catalysts play an important role in those processes. Aromatics and olefins are particularly susceptible to nitrogen effects when they are saturated by a hydro-treating process.

This application note describes the principle of operation and performance of the TSHR 7000 Total Nitrogen Analyzer for the determination of Total Nitrogen in liquid aromatic hydrocarbons according to ASTM D7184 methodology.

Experimental

Calibration of the TN 7000 analyzer was achieved using nitrogen standards that were produced by dissolving pyridine in xylene. A set of standards covering the range 0 to 1 mg/kg were prepared by serial dilution of stock standard and were then used to provide calibration of the nitrogen detector. A set of aromatic and liquid hydrocarbon samples were analyzed and the total nitrogen content determined based on the prepared calibration line.

The TSHR HR 7000 series auto sampler was used in conjunction with the TN 7000 analyzer to directly inject the samples into the injection port and furnace. Data acquisition and peak analysis was fully automated by the TSHR Athena software. System parameters were set by Athena software in accordance with ASTM D7184 method and presented in Table 1.

Presented in Figure 2 is the nitrogen calibration line for the TN 7000 analyzer. The calibration fit shows excellent linearity, with $R^2 = 0,99973$
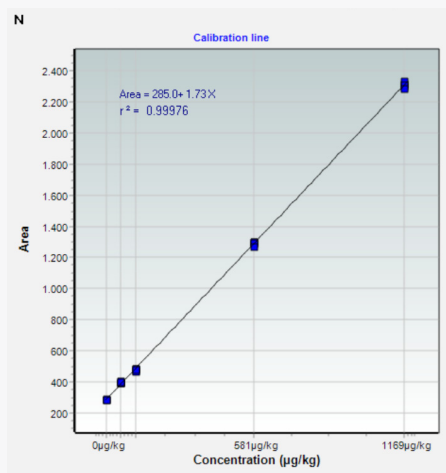

Figure 1:
TS 7000 with HR 7000 set-up

Parameter	System value
Argon carrier flow	75 ml/min
Oxygen primary flow	300 ml/min
Oxygen turbo flow	100 ml/min
Ozone generator flow	100 ml/min
Temperature Inlet	500 °C
Temperature furnace I	850 °C
Temperature furnace II	1000 °C
Injection speed	0.8 ul/s
Sample volume	80 ul

Results

Presented in Figure 2 is the nitrogen calibration line for the TN 7000 analyzer. The calibration fit shows excellent linearity, with $R^2 = 0,99973$

Theoretical TN conc (ug/kg)	Measured TN conc (ug/kg)
0	0,56
58,3	63,8
116	111
581	581
1161	1161

Figure 2: Linear calibration line for the TSHR TN 7000 Total Nitrogen Analyzer

Sample	Conc. (ug N/kg)	SD (ug N/kg)	RSD (%)
Cumene	197	11	5,4
Benzene	366	11	3,0
Toluene	756	6,1	0,8
p-Xylene	<30	5	
Hexane	1211	30	2,4
Heptane	1270	23	1,8
Methanol	147	3,7	2,5
Iso-Propanol (IPA)	427	14	3,3

Table 2: Nitrogen performance data obtained from several (aromatic) samples using TN 7000 Analyzer. Five repetitions of 80 uL were performed for each of the samples.
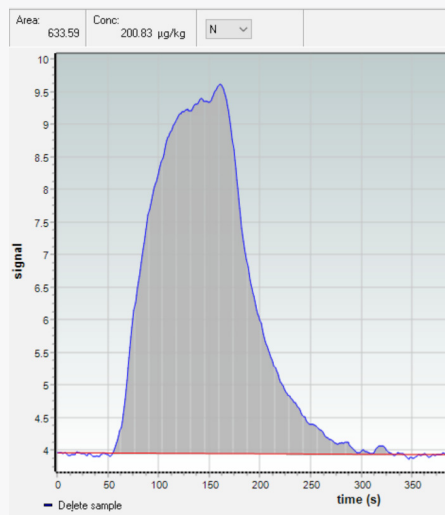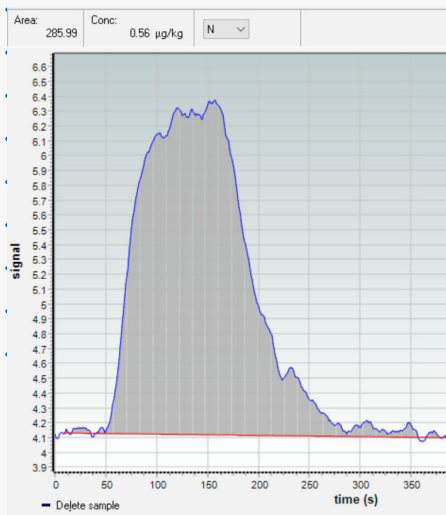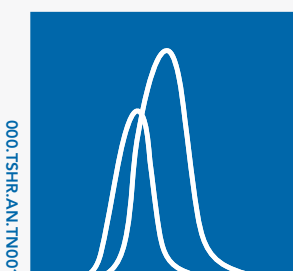
Conclusion

The analysis of trace level total nitrogen content in aromatic and liquid hydrocarbons is a routine analysis which can be adopted to the TSHR TN 7000 model with good repeatability performance, which are in compliance with ASTM D7184 method.

The availability of high quality gases, chemicals and a nitrogen free environment are very important factors which might influence the performance and accuracy of the analysis. Careful preparation of calibration standards are of paramount importance and will influence the precision of the test data.

The presented results in this Application Note shows excellent data and in full compliance with ASTM D7184 method.

References

[1] ASTM D7184 – 15 Standard Test Method for Ultra Low Nitrogen in Aromatic Hydrocarbons by Oxidative Combustion and Reduced Pressure Chemiluminescence Detection.

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