BRUKER

Chemical Analysis

Low Level Oxygenates Analyzer

- Trace Analysis of Oxygenates in Hydrocarbon Matrices

think forward

Gas Chromatography

The determination of sub to high ppm levels of ethers, alcohols, aldehydes and ketones in different hydrocarbon matrices is a recurring challenge in the petroleum refining and petrochemical industry. The Bruker Low Level Oxygenates Analyzer is an easy to use 'turnkey' solution to meet this analysis challenge.

Key Benefits include:

- Factory optimized to ensure excellent analysis results
  The system is configured, optimized and tested at the factory to analyze oxygenated components at concentrations as low as 100 ppb in streams with a final boiling point of up to 250 °C and no interference from the hydrocarbon matrix. An optimized system for the analysis of oxygenates at trace level.

- A powerful GC solution, yet extremely easy to operate
  The 450-GC and Galaxie™ Chromatography Data Handling Software, is a very powerful combination and is key to the system's ability to generate reliable results. The system can be used sucessfully without a high level of operator skill.

- A flexible, economical analysis solution
  The analysis system is configured to accommodate gas, liquid and liquified petroleum gas (LPG) streams. With both a gas and a liquid sampling valve, both substances can be analyzed by any hardware reconfiguration.

- A cost effective, time-saving solution
  All Bruker GC analyzers not only include proven GC hardware and software but are also pre-loaded with analysis methods and documentation specific to the method required. All operational procedures are fully documented.

- A comprehensive, single vendor solution
  The hardware, software, application optimization, documentation, installation and support services are all provided by Bruker, allowing analysis to be carried out quickly and efficiently by one dependable supplier.

Figure 1: The analyzer incorporates the 450-GC

Low Level Oxygenates Analyzer

Oxygenated compounds can be present in hydrocarbon streams for a variety of reasons. For example, methanol is added to crude oil to reduce the formation of hydrates during transportation and storage. Unfortunately, the presence of methanol in downstream operations is highly problematic.

Clean-up processes like hydro-treating are used in an attempt to remove oxygenated compounds. But even at trace levels (sub ppm), oxygenates quickly degrade or destroy expensive process catalysts, (e.g. polymer production).

Although there are a number of industry standard methods for the analysis of oxygenated compounds in use today (ASTM D4815 or D5599, EN 13132 or 1601 and DIN 51413-7), none are suitable for the analysis of oxygenates at sub ppm levels. To meet this need, the Low Level Oxygenates Analyzer has been developed.

Analyzer Overview

The Low Level Oxygenates Analyzer is designed and optimized to quantify ppm and sub ppm levels of ethers (e.g. DME, MTBE, ETBE, DIPE), alcohols (e.g. methanol, ethanol, propanol), ketones (e.g. acetone, MEK) and aldehydes in various hydrocarbon matrices. In general, all oxygenated components with a boiling point of up to 100 °C can be analyzed. The sample can be a gas, LPG or liquid under ambient conditions with a final boiling point up to 250 °C.

The system is comprised of a Bruker 450-GC configured with gas and liquid sampling valves, two high performance capillary analysis columns, digitally controlled pneumatics including a 'fluidic' switch and Flame Ionization Detector (FID). An optional 'pressure station' can be added to eliminate the possibility of losing sample due to evaporation when analyzing LPG.

The 450-GC is controlled via the Galaxie™ Chromatography Data Handling Software, which acquires data, processes it and generates analyses reports. The gas or liquid sample is injected via a gas or liquid sampling valve onto the first of two columns (CP-SIL 5CB and LOWOX™). A fluidic switch is positioned between the two columns. The 'lighter' fraction (containing the oxygenates) is separated from the rest of the stream components on the CP-SIL 5CB column.

The 'heavier' components are then back-flushed tovent. The fraction containing the 'lighter' components is transferred onto the second column (LOWOX) using the fluidic switch. The LOWOX column is used to separate the individual oxygenated components from the bulk hydrocarbons. The LOWOX column was specially developed in the 1990s for the trace analysis of oxygenates in hydrocarbon streams. It offers unequalled selectivity for oxygenated compounds.

Due to its high selectivity, large amounts of sample can be introduced onto the LOWOX column which, through the use of the sensitive FID, enables the system to achieve the required low detection levels for oxygenated compounds.

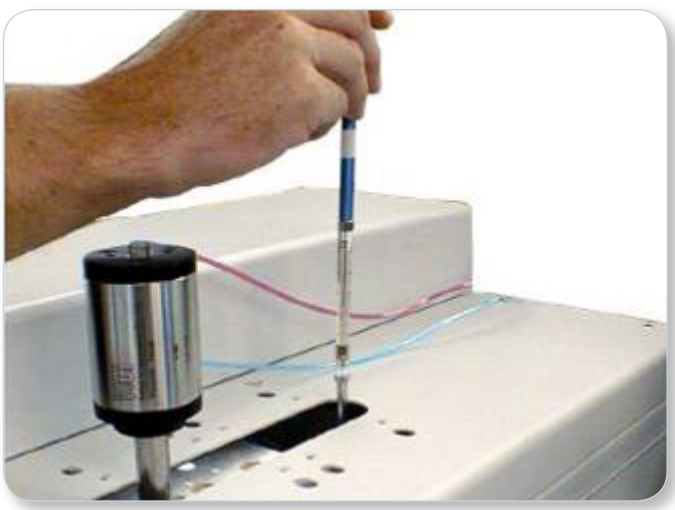

Figure 2: The analyzer includes both gas and liquid sample valves for maximum flexibility.

Analysis of Oxygenates at sub-ppm Levels

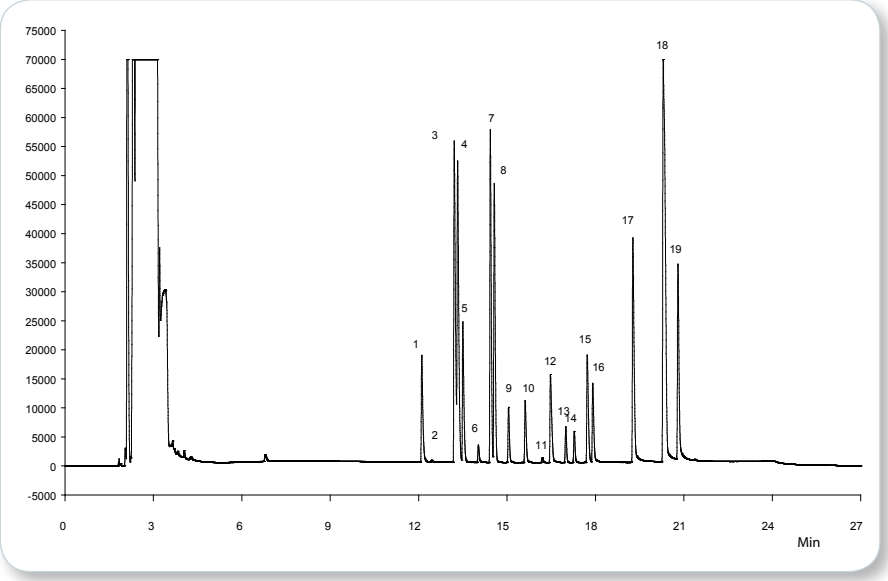

Figure 3: A typical chromatogram showing a wide range analysis of a liquid sample stream

Calibration is performed using several concentrations of a multi-component standard mixture with varying known/ measured amounts of each oxygenated compound.
The system automatically generates a calibration curve for each analyte. The samples are analyzed, each of the oxygenates are automatically quantified and a report of the results is printed at the end of each analysis cycle. (Typical performance results are depicted in Table 1.)

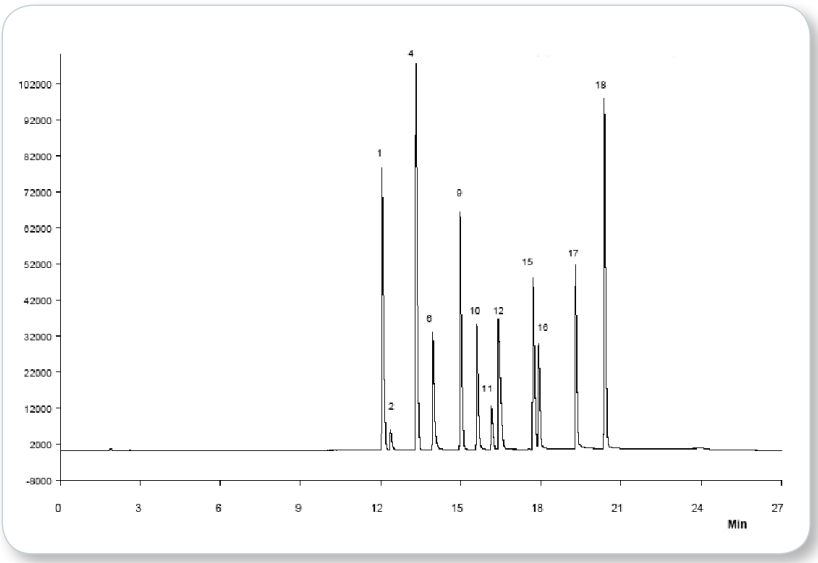

Figure 4: Typical chromatogram showing a MTBE range analysis of a gas stream.

Peak Identification

1. Diethylether
2. Acetaldehyde
3. Ethyl tert. Butyl ether
4. Methyl pert. Butyl ether
5. Diisopropylether
6. Propanal
7. Tert. amyl methyl ether
8. Propylether
9. Isobutyraldehyde
10. Butyraldehyde
11. Methanol
12. Acetone
13. Isovaleraldehyde
14. Valeraldehyde
15. 2-Butanone
16. Ethanol
17. 1-Propanol
18. Tert. butanol
    2-Methyl 1-propanol
19. 1-Butanol

Typical Performance

Table 1. Typical repeatability in the analysis of oxygenates in a gas stream

	ETBE	MTBE	Methanol	Aceton	MEK	Ethanol	1-Propanol
Run 1	579554	490771	59388	224339	338840	129250	224083
Run 2	578838	491908	59310	224407	339195	129779	225111
Run 3	585890	481217	60135	225063	340116	131457	227596
Run 4	579313	491673	59663	224583	339338	130075	225695
Run 5	578500	492449	59897	224743	339606	131011	227058
Run 6	577796	493989	59766	224583	339482	130628	226651
Run 7	579844	490743	59879	224593	339622	130645	226309
Run 8	580253	487207	59800	224570	339470	130874	226918
Run 9	580592	484805	59856	224539	339556	130808	226284
Run 10	578693	487272	59946	224530	339893	131131	226630
N	10	10	10	10	10	10	10
Mean	579927	489203	59764	224595	339512	130566	226234
Std. Dev.	2142.60	3775.30	238.60	187.10	334.20	637.80	975.60
RSD %	0.37	0.77	0.40	0.08	0.10	0.49	0.43

Table 2. Typical analyzer performance.

Minimum Detection Level	100 ppb/Weight
System Linearity	100 ppb to 500 ppm
Typical RSD %	For gas streams; < 2 % for MTBE, methanol and ethanol. For liquid streams; < 2.5 % for MTBE, methanol and ethanol.

Chemical Analysis Solutions

GC quadrupole mass spectrometers

The Bruker 300-MS series GC/MS systems stand at the pinnacle of versatility for quadrupole mass spectrometer systems. Both the 300-MS and 320-MS are configurable as either single-quadrupole, or triple-quadrupole systems.

The 300-MS delivers the performance you've come to expect from an industry leader in quadrupole innovation. It features an 800 Da mass range, superior negative ion sensitivity, and unmatched robustness in its performance class. The 320-MS delivers femtogram sensitivity, 2000 Da mass range, and a wide array of chromatographic and ionization configurations to uniquely match your needs - all in less than 72 cm. (28 in.) of linear bench space! In minutes, our 300-MS series systems can be changed from EI to CI modes of operation. Easily, our 300-MS and 320-MS are the most sensitive, robust, and flexible quadrupole GC/MS systems currently available.

ICP mass spectrometers

Choosing an ICP-MS for your elemental analysis needs has never been easier with the Bruker 800-MS Series. The 810-MS is the instrument of choice for routine analysis with industry leading sensitivity and intuitive Web-integrated ICP-MS Expert software. The 820-MS features Bruker's novel collision reaction interface (CRI), providing interference-free analysis and allowing you to tackle any application with ease. With a vast range of accessories, Bruker has the solution to all your ICP-MS application requirements.

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Bruker Daltonik GmbH

Bremen · Germany
Phone +49 (421) 2205-0
Fax +49 (421) 2205-103
sales@bdal.de

Bruker Daltonics Inc.

Billerica, MA · USA
Phone +1 (978) 663-3660
Fax +1 (978) 667-5993
ms-sales@bdal.com

www.bruker.com/chemicalanalysis