

Low level oxygenates analyser

- ASTM D7423, ASTM D7754, UOP 960
- Robust, modular, high uptime
- For liquid samples; option for gas and LPG

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GAS offers custom configured GC analysers for many application fields for over 50 years. GAS analysers are designed to meet many standardised methods from GPA, ASTM, UOP, ISO, EN and others. The efficient configurations are based on proven GC technology, resulting in robust, highly productive instruments with an optimal return on investment.

The low level oxygenates analyser from GAS analyses oxygen containing components like alcohols and ethers at (sub) ppm level. The presence of these components in hydrocarbon feedstock reduces catalyst lifetime, even at low ppm level. Analysis at low levels is also demanded by environmental requirements.

ASTM D7423, UOP 960

ASTM D7423 and UOP 960 cover the analysis of oxygen containing components with boiling points up to 100 $^{\circ}$ C in hydrocarbon streams with final boiling point below 250 $^{\circ}$ C. The concentration range is 0.1-1000 ppm.

Figure 1 shows the instrument diagram. A highly selective capillary column (Lowox) separates the components of interest from the hydrocarbon matrix. The system is equipped with a SSL module (liquid sample injector) with backflush option for removing components with boiling point above 100 °C using an Rtx-1 pre-column. Figure 2 shows a 100 ppm calibration standard and figure 4 shows the chromatograms of a pygas sample. Figure 7 demonstrates the repeatability.



Figure 1

Lowox analyser with modular injector and detector modules. The liquid injector comes with an integrated backflush option, so the use of additional valves is not needed.

LOWOX CALIBRATION STANDARD (100 ppm for each component)

Dimethylether Acetaldehyde Methanol Ethanol Propylether Propionaldehyde iso-propanol **T-Butanol** Propanol Methyl Ethyl Ketone Diethylether Iso-Butanol N-Butanol Iso-Butanal Buteraldehyde Ethyl Tert Butyl Ether **Di-Isopropylether** Methyl Tert Butyl Ether





Figure 3 Chromatogram: Pygas sample



Figure 4 Trace 1600 GC with InstantConnect injector and detector technology & AS 1610 autosampler

ASTM D7754

Besides ASTM D7423 and UOP 960, the GAS low level oxygenates analyser covers ASTM D7754 as well, for samples containing 1-15% ethanol, with the use of a longer pre-column.

Trace GC 1600

The low level oxygenates analyser is based on Thermo Trace GC 1600 with modular injector and detector technology, providing low maintenance costs and high uptime (figure 4,5). Automated liquid sample injection is provided by AI 1610, AS 1610 or TriPlus RSH autosamplers.

Options for gas and LPG samples

Figure 1 explains the basic setup for liquid samples. In case of gas samples, the SSL is replaced by a gas sampling valve (GSV). For liquefied gas samples, a LSV (Liquid sampling valve) is added to the SSL, and a Sample Securitiser is available to secure the liquid state in the valve. Figure 6 shows an analyser setup for injecting gas, liquefied gas and liquid samples.

GC-MS

High yield catalysts are extremely prone to poisoning by feedstock impurities like oxygenated components. Therefore a GC-MS based solution is available for substantial increase in identification and sensitivity compared to GC-FID analyses. The gain is particularly significant when SIM mode is used. ISQ mass spectrometer conveniently combines full scan and SIM in a single run for both increased sensitivity and reliable identification.



Figure 5 Swift exchange of injector and detector



Figure 6

Lowox analyser equipped for gas (using GSV), liquefied gas (using LSV and Sample Securitiser) and liquid (using SSL) samples.

Specification

| Standardised method: | ASTM D7423, UOP 960 | | | | | |
|------------------------|--|--|--|--|--|--|
| | ASTM D7754 (for samples containing 1-15% v/v ethanol) | | | | | |
| Application: | Analysis of oxygenated components (alcohols, ethers, ketones) in liquids | | | | | |
| Configuration: | Single channel instrument based on Thermo Trace 1600 GC series using FID detec | | | | | |
| Injection: | SSL (Liquid Injection), GSV (Gas Sampling Valve) or LSV (Liquid Sampling Valve); | | | | | |
| | all three injection techniques can be combined | | | | | |
| Optional: | Automated liquid sample injectors Al 1610, AS 1610 and TriPlus RSH | | | | | |
| | Gas sampling valve (GSV) | | | | | |
| | Liquid sampling valve (LSV) $\&$ Sample Securitiser for highly quantitative injection of | | | | | |
| | liquefied gas samples | | | | | |
| | Mass Spectrometer for enhanced identification and sensitivity | | | | | |
| Tubing: | Sulfinert [®] tubing for inert sample path | | | | | |
| Analytes: | see table (page 2). | | | | | |
| Calibration standards: | 1, 10, and 100 ppm per individual component in n-hexane | | | | | |
| Dynamic range: | 0.1-1000ppm (FID) | | | | | |
| Sample requirements: | Liquid, liquefied gas or gas sample | | | | | |
| Analysis Time: | 25 minutes | | | | | |
| Minimum Detectability: | 0.1 ppm for all individual components. | | | | | |
| Repeatability: | 2% RSD (n=10) | | | | | |
| Data systems | Chromeleon, OpenLab | | | | | |
| | | | | | | |

| ETBE | MTBE | DIPE | TAME | PE | MEK T-Butanol, Iso | | 1-Butano |
|-----------|-----------|-----------|-----------|-----------|--------------------|-----------|-----------|
| Area | Area | Area | Area | Area | Area | Area | Area |
| 217590.00 | 219186.00 | 225544.00 | 234268.00 | 221104.00 | 197462.00 | 668182.00 | 352884.00 |
| 218929.00 | 223651.00 | 228403.00 | 238190.00 | 225201.00 | 199992.00 | 674969.00 | 364892.00 |
| 216047.00 | 220135.00 | 223283.00 | 232977.00 | 220184.00 | 195684.00 | 661960.00 | 356955.00 |
| 223290.00 | 227834.00 | 233581.00 | 243023.00 | 229025.00 | 201593.00 | 684831.00 | 353251.00 |
| 215944.00 | 218017.00 | 224411.00 | 233341.00 | 219821.00 | 194399.00 | 658748.00 | 339042.00 |
| 216799.00 | 220695.00 | 227555.00 | 235189.00 | 221814.00 | 194438.00 | 661578.00 | 348803.00 |
| 218518.00 | 220454.00 | 227493.00 | 236573.00 | 223679.00 | 193629.00 | 661017.00 | 351983.00 |
| 223242.00 | 224438.00 | 232150.00 | 241893.00 | 228986.00 | 197467.00 | 675404.00 | 353714.00 |
| 215976.00 | 215619.00 | 222285.00 | 232897.00 | 219837.00 | 190374.00 | 646960.00 | 347844.00 |
| 222034.00 | 224530.00 | 232040.00 | 241757.00 | 227848.00 | 195446.00 | 664451.00 | 354400.00 |
| 215944.00 | 215619.00 | 222285.00 | 232897.00 | 219821.00 | 190374.00 | 646960.00 | 339042.00 |
| 223290.00 | 227834.00 | 233581.00 | 243023.00 | 229025.00 | 201593.00 | 684831.00 | 364892.00 |
| 218836.90 | 221455.90 | 227674.50 | 237010.80 | 223749.90 | 196048.40 | 665810.00 | 352376.80 |
| 2974.18 | 3624.26 | 3916.83 | 3966.46 | 3776.53 | 3234.66 | 10573.04 | 6631.43 |

Figure 7 Repeatability of analysis of low level oxygenates components. 100 ppm calibration standard, liquid injection

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