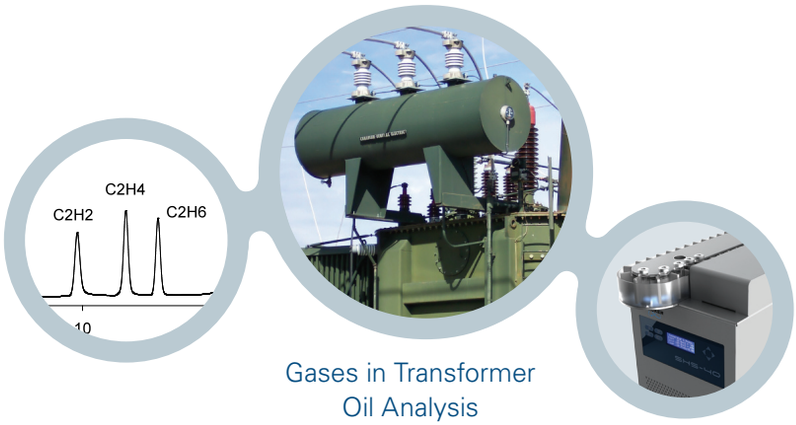


Gases in Transformer Oil Analysis

Application Notes #283028

Transformer Oil Gas Analysis with the Bruker TOGA Analyzer equipped with the Bruker Headspace Sampler.

Insulating fluids, generally mineral oils, are used in transformers. Under normal, mild conditions there is very little decomposition.

Occasionally however (localized or general) overheating of the oil occurs and decomposition products are formed. If the concentration of these gases reaches a critical point, the chances of catastrophic transformer failure are high.

The ASTM D 3612 method[1] describes in detail three different routes.

A. Vacuum Extraction.
The gases are extracted from the oil via a vacuum extraction device and analyzed via gas chromatography.

B. Stripper Column Extraction.
Dissolved gases are extracted from a sample of oil by sparging the oil with the carrier gas on a stripper column containing a high surface area bead. The gases are then flushed from the stripper column into a gas chromatograph for analysis.

C. Headspace Sampling.
An oil sample is brought into contact with a gas phase (headspace) in a closed vessel purged with Argon.

As a result, a portion of a gas dissolved in the oil is transferred to the Headspace.

This application note describes Method C.

Instrumentation

Gas Chromatograph
- Bruker TOGA Analyzer based on 450-GC

Headspace Sampler
- SHS-40 Headspace Analyzer

GC control and data handling
- Compass CDS software

Figure 1. TOGA analysis, TCD channel.

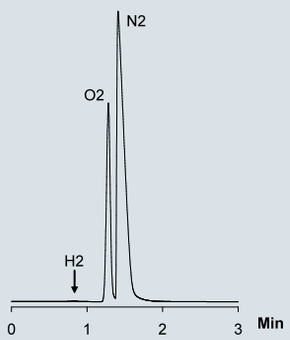


Figure 2. TOGA analysis FID channel.

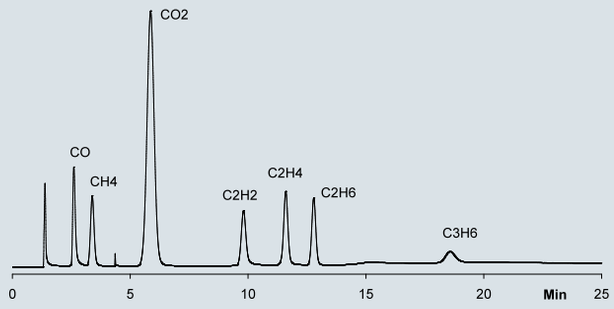
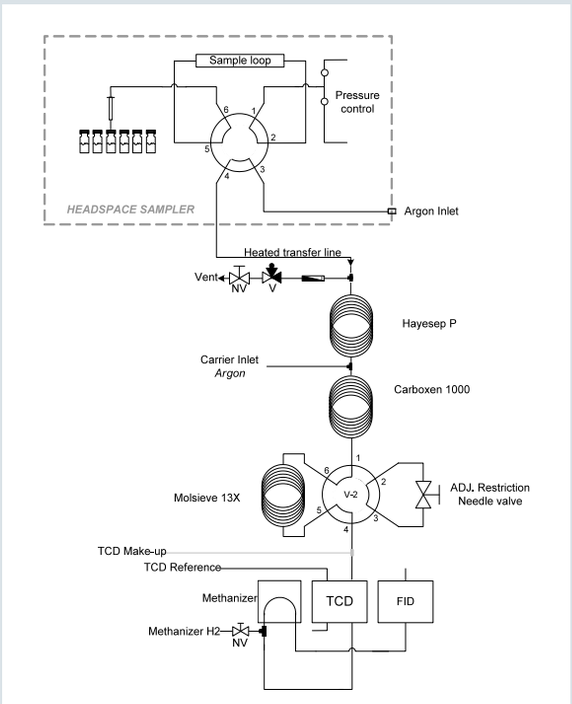


Figure 3. Schematic overview hardware.



Materials and Reagents

"True North" DGA Oil Standard by Morgan Schaffer:

Hydrogen	88 ppm
Oxygen	11163 ppm
Nitrogen	40368 ppm
Methane	96 ppm
Carbon monoxide	89 ppm
Carbon dioxide	123 ppm
Ethylene	90 ppm
Ethane	92 ppm
Acetylene	84 ppm

Sample Preparation

The Morgan Schaffer Calibration Standard is carefully transferred into the headspace vial.

The gases are extracted from the oil by means of a headspace sampler and injected onto a short Hayesep P precolumn and then to a micro packed Carboxen-1000 column. The fraction containing Hydrogen, Oxygen, Nitrogen, Carbon Monoxide, and Methane will elute direct from the Carboxen-1000 column to the micro packed Molsieve column. Hydrogen, Oxygen and Nitrogen are detected by the TCD. Carbon Monoxide, and Methane are detected by the FID, after passing the Methanizer. When the Molsieve column is bypassed, Carbon Dioxide and the C_2-C_3 isomers are eluting from the Carboxen-1000 column and detected by the FID after passing the Methanizer. The back flush time is set to completely elute the C_3 isomers. C_4 and higher are back flushed.

Extraction, Method Parameters:

Conditions

Table 1. Column oven settings.

Safety Class 1

Rate (°C/min)	Step (°C)	Time (min.)
Initial	50	5.0
10.0	75	0.0
20.0	220	10.25
	Total Time	25.0

Table 2. TCD, FID, Methanizer settings.

TCD	Ar reference flow	10 mL/min
	Temperature	200°C
	Filament temperature	254°C
	Carrier gas	N_2 /Ar
	Total Time	25.0
FID	Temperature	300°C
	Ar makeup flow	20mL/min
	H_2 flow	10mL/min
	Air flow	300mL/min
Methanizer	Temperature	400°C

Table 3. Valves.

Time (min)	(1) Gas Sampling Valve	(2) Series bypass	Sample	Event A
Initial	Fill	Series	OFF	OFF
3.0	Fill	Series	OFF	ON
4.2	Fill	Bypass	OFF	ON

Results and Discussion

Chromatograms of both TCD and FID channels are shown in Figure 1 and Figure 2. The complete hardware configuration (SHS-40/TOGA) is shown schematically in Figure 3.

Repeatability is tested by analyzing multiple samples from the same source. Data can be found in Table 4.

Table 4. Repeatability data.

Run	N_2 Peak Area	CH_4 Peak Area	CO_2 Peak Area
1	692201	609	369764
2	696712	606	365757
3	669175	584	361535
4	678626	592	361783
5	709715	577	364403
6	702775	576	376105
7	724545	607	393602
n	7	7	7
Average	696249.9	593.0	370421.3
St.Dev.	18640	14.4	11414
RSD (%)	2.68	2.43	3.08

A graphic representation of the data is shown in Figure 4 and Figure 5.

Figure 4. Repeatability results of a Transformer Oil.

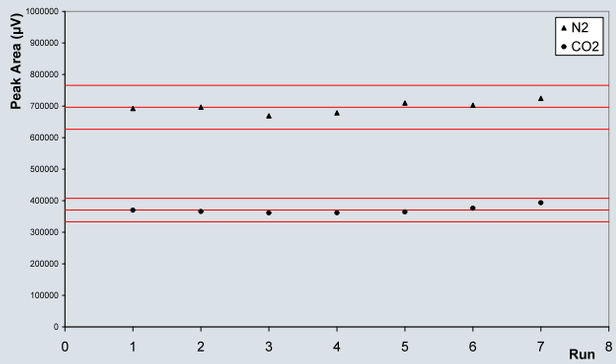
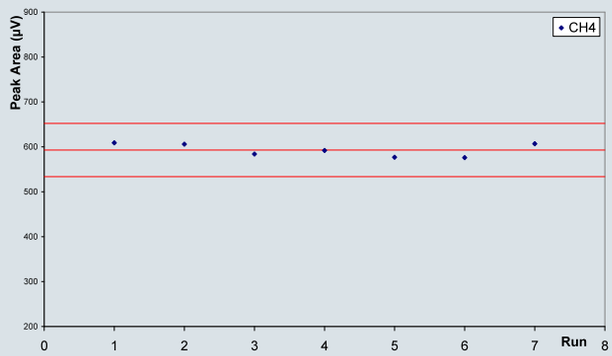


Figure 5. Repeatability results of a Transformer oil.



Besides the analytical result also the window specified in the ASTM D 3612 method is presented.

From the data presented in Table 4, Figure 4 and Figure 5 it is clear that the repeatability of the system is well within the window specified by the ASTM D 3612.

Conclusion

Full separation of all components of interest, easy and reliable quantification results with very good repeatability was achieved.

The analysis of dissolved gases in transformer oil according to ASTM D 3612, Method C, can be performed perfectly with the Bruker Transformer Oil Gas Analyzer (TOGA Analyzer) in conjunction with the Bruker SHS-40 Automated Headspace Sampler.

References

1. ASTM Standard D 3612 - 02, "Analysis of Gases Dissolved in Electrical Insulation Oil by Gas Chromatography. Method C", ASTM International, West Conshohocken, PA, www.astm.org

Bruker Daltonics Inc.

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

Fremont, CA · USA
Phone +1 (510) 683-4300
Fax +1 (510) 490-6586
ms-sales@bdal.com

www.bruker.com

Bruker Daltonik GmbH

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