Transformer Oil Gas Analysis with the Scion TOGA Analyzer Equipped with a Headspace Sampler

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Introduction

Insulating fluids, generally mineral oils, are used in transformers. Under normal, mild conditions, there is very little decomposition. Occasionally, however, localized or general heating 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 for transformer gas analysis.

A. Vacuum Extraction

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

B. Stripper Column Extraction[2]

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[3]

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

Scion TOGA Analyzer
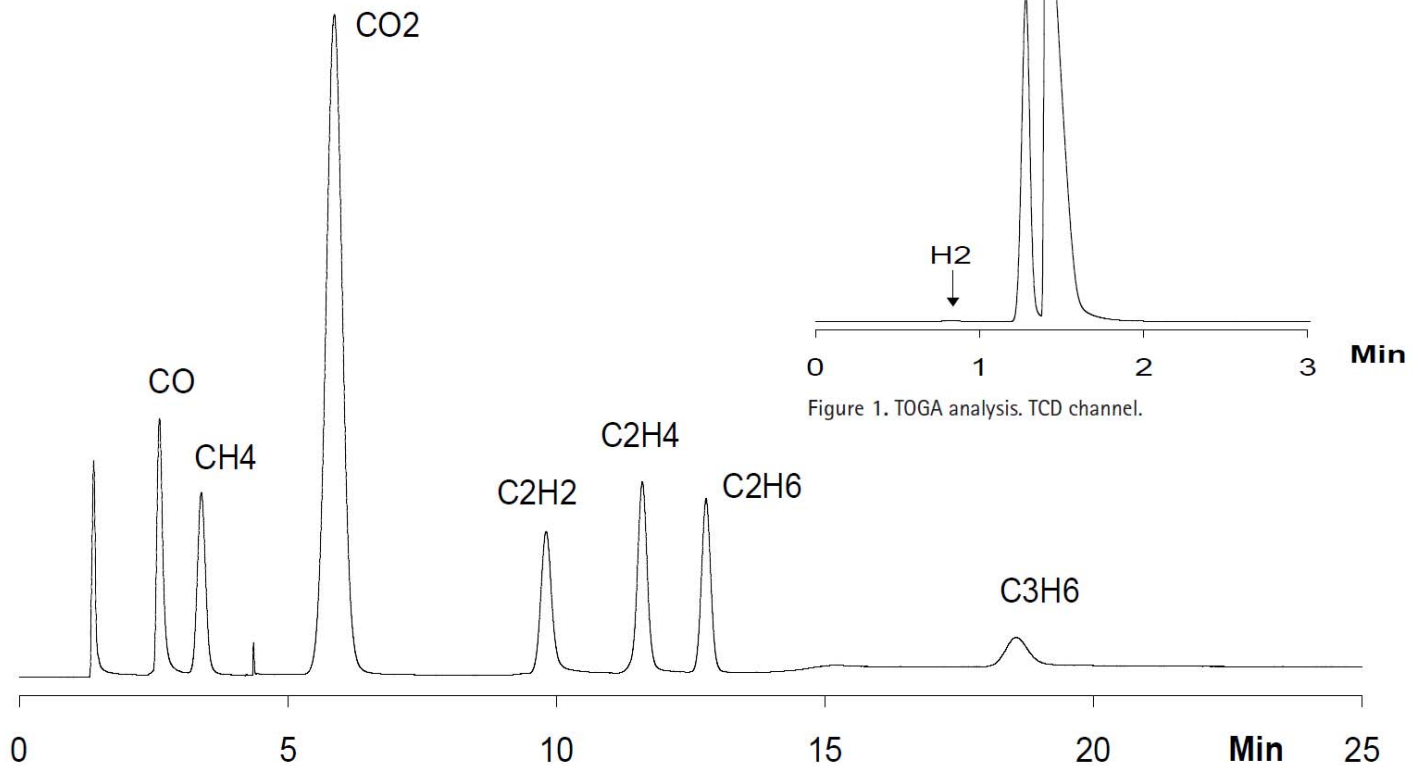Scion 456 -GC Gas Chromatograph
Headspace sampler in sample loop mode

Figure 1. TOGA analysis. TCD channel.

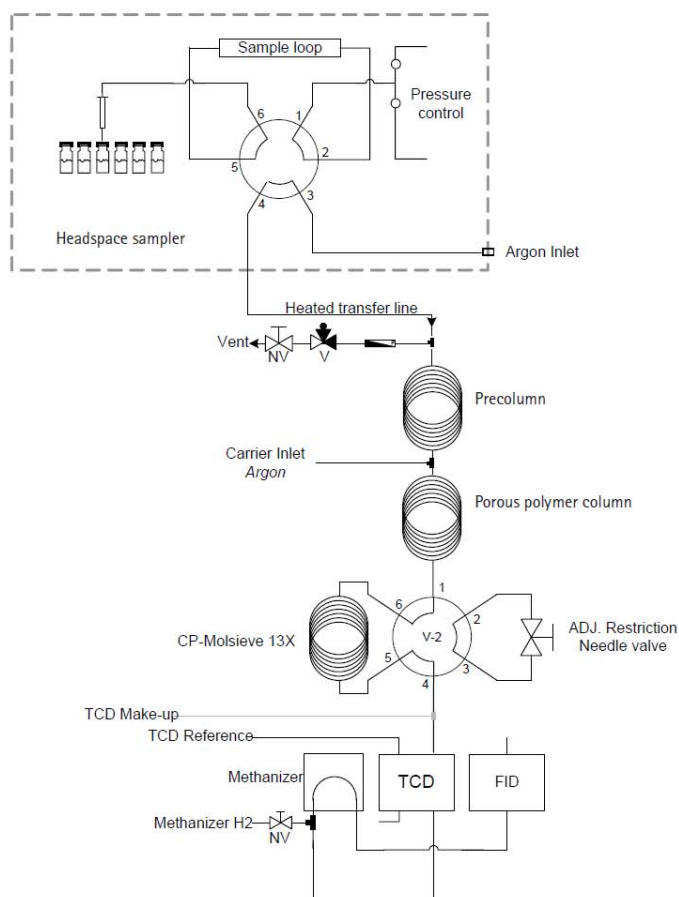Figure 1. TOGA analysis. FID channel.

Software
Compass ™ Software

Figure 3. Schematic overview of the hardware.

Materials and Reagents
Commercial oil standard containing:

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 calibration standard is carefully transferred into the headspace vial. The gases are extracted from the oil by means of a headspace autosampler and injected onto a short porous polymer precolumn and then to a micro packed spherical carbon molecular sieve column.

The fraction containing hydrogen, oxygen, nitrogen, CO and methane will elute directly from this column to the micro packed Scion CP-Molsieve™ column. Hydrogen, oxygen and nitrogen are detected by the TCD. CO and methane are detected by the FID, after passing the methanizer. When the CP-Molsieve column is bypassed, CO_2 and the C2–C3 isomers elute from the porous polymer column and are detected by the FID after passing the methanizer. The back flush time is set to completely elute the C3 isomers. C4 and higher are back flushed.

Extraction, Headspace Parameters

Table 1. Column oven settings.

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
FID	Temperature	300 °C
	Ar makeup flow	20 mL/min
	H_2 flow	10 mL/min
	Air flow	300 mL/min
Methanizer	Temperature	400 °C

Table 3. Valves.

Time (min)	(1) Gas Sampling Valve	(2) Series bypass	(3) Sample	(4) 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 Figures 1 and 2. Repeatabilty was tested by analyzing multiple samples from the same source (Table 4). A graphic representation of the data is shown in Figures 4 and 5.

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

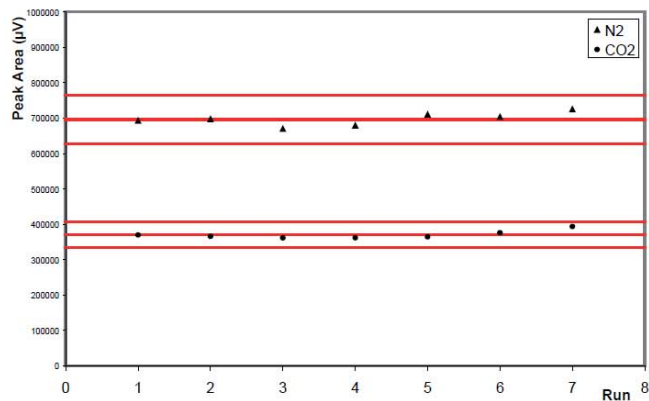Figure 4. Repeatability results for methane from analysis of a transformer oil.

Figure 4. Repeatability results for nitrogen and carbon dioxide from analysis of a transformer oil.

As well as the analytical result, the limits specified in the ASTM D 3612 method are shown. 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 limits specified by ASTM D 3612.
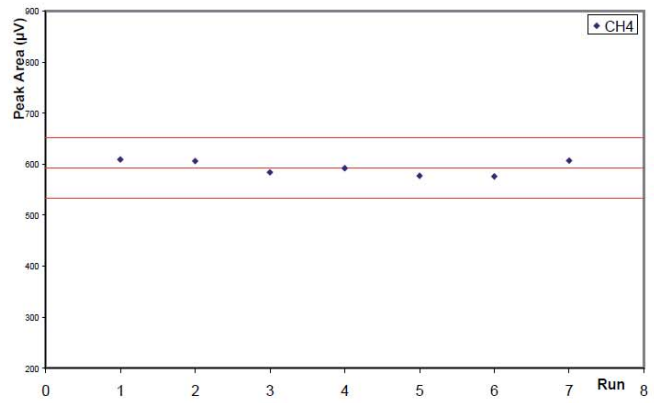
Conclusion

Full separation of all components of interest, and easy and reliable quantification, results in very good repeatability using the Scion Transformer Gas Oil Analyzer. The analysis of dissolved gases in transformer oil according to ASTM D 3612, method B, can also be performed perfectly with the Scion TOGA with a headspace sampler.

References

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

[2]Duvekot, C. Analysis of Dissolved Gas in Transformer Oil by Gas Chromatography using a Stripper Column. Scion Application Note SI-01298.

[3]Duvekot, C. Gas Chromatography, TOGA Analysis to ASTM D3612. Scion Application Note 68.

These data represent typical results.
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Application Note SI-01364

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