Transformer Oil Gas Analysis via Headspace Sampling (ASTM D3612)

Application Note

AN0030

INTRODUCTION

Insulating fluids, generally mineral oils, are used in transformers. Under normal, mild conditions, there is very little decomposition. However, occasionally localised or general heating of the oil occurs and decomposition products are formed. If the concentration of these gases reach a critical point, the chances of catastrophic transformer failure are high. ASTM D3612 describes in detail three different routes for transformer gas analysis.

During Vacuum Extraction gases are extracted from the oil via a vacuum extraction device and analysed using gas chromatography (GC). Stripper Column Extraction details the extraction of dissolved gases from a sample of oil by sparging the oil with the carrier gas, onto a stripper column containing a high surface area bead. The gases are then flushed from the stripper column into a GC for analysis. The final method is Headspace Sampling in which an oil sample is brought into contact with the headspace in a closed vessel sparged with argon. As a result, a portion of gas dissolved in the oil is transferred to the headspace. This application note describes the final method; Headspace Sampling.

EXPERIMENTAL

The SCION TOGA Analyser comprised of a SCION 456 GC with FID and TCD detectors. A headspace sampler in sample loop mode was also used. Figure 1 details the schematic overview of the TOGA Analyser.
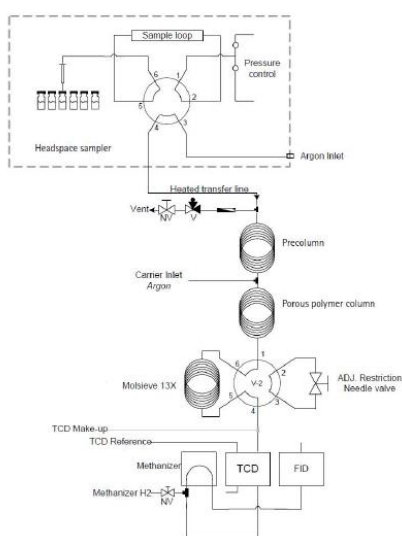

Fig 1. Schematic operation of the TOGA Analysis

Table 1 details the analytes of interest obtained from an injection of a commercial oil standard.

Table 1. Commercial oil standard components

Analyte	Conc (ppm)
Hydrogen	88
Oxygen	11163
Nitrogen	40368
Methane	96
Carbon Monoxide	89
Carbon Dioxide	123
Ethylene	90
Ethane	92
Acetylene	84

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 onto a micro packed spherical carbon molecular sieve column. The fraction containing hydrogen, oxygen, nitrogen, CO and methane will elute directly from this column onto the packed SCION Molsieve column. Hydrogen, oxygen and nitrogen are detected by the TCD. CO and methane are detected by the FID, after passing the methaniser. When the molsieve column is bypassed, CO_2 and the C_2-C_3 isomers elute from the porous polymer column and are detected by the FID (after passing through the methaniser). The back flush is set to completely elute the C_3. C_4+ are backflushed. Tables 2a and 2b detail the method parameters used throughout this application note.

Table 2a. Analytical conditions of the GC

Condition	
Oven	50°C (5 mins), 10°C/min to 75°C, 20°C/min to 220°C
TCD	200°C, Filament 254°C, Air 10mL/min, Carrier N_2/ Ar
FID	300°C, Ar makeup 20mL/min, H_2 10mL/min, Air 300mL/min
Methaniser	400°C

Table 2b. Valve settings

Time (min)	GSV	Series Bypass	Sample	Event A
Initial	Fill	Series	OFF	OFF
3.0	Fill	Series	OFF	ON
4.2	Fill	Bypass	OFF	ON

RESULTS

Chromatograms of both TCD and FID are shown in Figures 2 and 3.
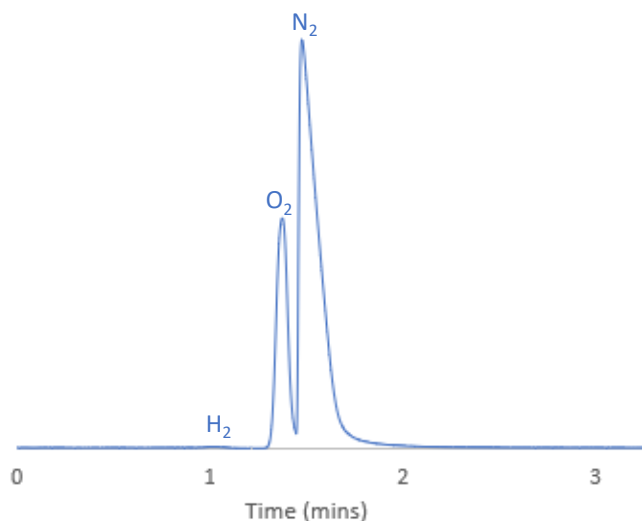

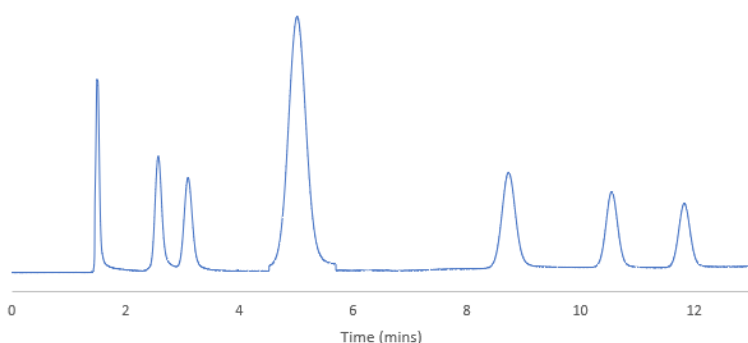
Fig 2. TOGA Analysis; TCD channel

Fig 3. TOGA Analysis; FID channel

Repeatability was tested by analysing multiple samples from the same source. Table 3 shows the repeatability data whilst Figures 4 and 5 show graphic representation of the repeatability of the TOGA analyser.

Table 3. Peak area repeatability values

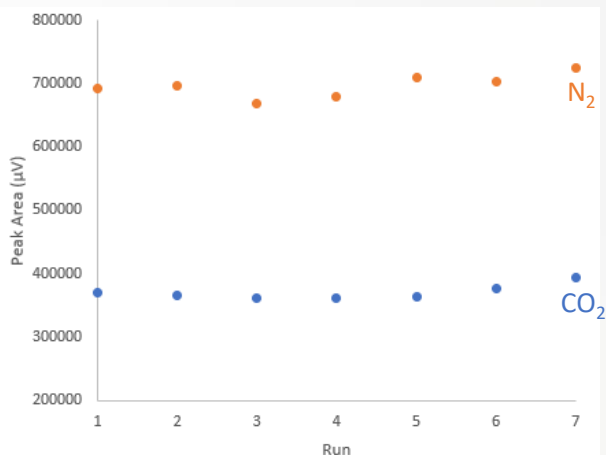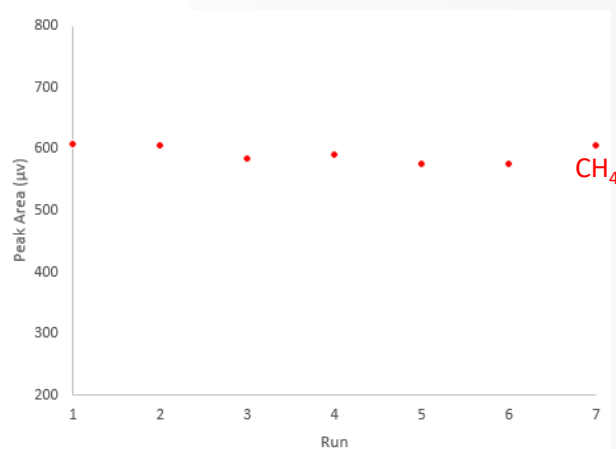| Run	N_2	CH_4	CO_2
1	692201	609	369764
2	696712	606	365757
3	669175	584	361535
4	678626	592	361783
5	709715	577	364403
6	702775	576	376105
7	724545	607	393602
Ave	696249.9	593	370421.3
RSD %	2.68	2.43	3.08

Fig 4. Repeatability data N2 and CO2

Fig 5. Repeatability data CH4

The repeatability data shown above is well within the defined limits specified by ASTM D3612 (N_2 <5%, CO_2 <4%, CH_4 <4%).

CONLUSION

Full separation of all components of interest with easy and reliable quantification results in very good repeatability using the SCION Transformer Gas Oil Analyser. The analysis of dissolved gases in transformer oil according to ASTM D3612, method C, can also be performed perfectly with the SCION TOGA analyser with headspace sampler.