LCGC north america
solutions for separation scientists

Analysis of Electrical Insulating Oils Using Static Headspace Sampling

M. Taylor, Arnel Inc.

The analysis of dissolved gases in insulating oil is commonly used for the detection of transformer degradation. The addition of method C to ASTM method D3612-02 (Analysis of Gases Dissolved in Electrical Insulating Oil by Gas Chromatography) now allows the use of headspace sampling as an alternative to methods A and B. This application note examines the results obtained using the headspace sampling technique and the resulting increase in sample throughput.

ASTM method D-3612 has had a 25-year history of use in the power industry. The original method employed a glassware distillation using mercury as a manometer to extract the dissolved gases, followed by a gas chromatographic (GC) analysis. Circa 1990, a second procedure was added, method B, whereby a gas chromatograph was modified with a sparging vessel to extract the dissolved gases in-situ, followed by GC analysis. In 2001, method C was added to D-3612. Method C employs the use of headspace sampling, whereby the oil is brought in contact with a gas phase in a closed vessel. The oil sample is thermostated, shaken to an equilibrated state, and over-pressurized with argon gas. The headspace gas is then directed to the chromatographic analyzer.

Experimental

In a marketing collaboration with the PerkinElmer Corporation (Shelton, Conneticut), a Clarus 500 Gas Chromatograph and TurboMatrix Headspace sampler were utilized and integrated into the Arnel Model 4087 Transformer Oil Gas Analyzer (TOGA) system. The system includes a packed injector, capillary columns, thermal conductivity, flame ionization detectors, and a high-capacity methanizer. Instrument operating conditions were established as specified in method C of the ASTM method.

A known volume of oil is placed in a headspace vial of known capacity. (Note: The vial capacity is determined by selecting and weighing 20 vials at ambient laboratory temperature, filling each of them completely with water, and reweighing; then calculating the capacity of each, knowing the density of water at the ambient laboratory temperature. The capacity determined for each of the 20 vials is used to determine the mean volume and standard deviation. The analytical performance of method C was established with a less than 0.7% RSD over the 20 vials.) The vial is then maintained at a constant temperature under mechanical agitation until thermodynamic equilibrium between the oil sample and the headspace is reached. The time required to reach equilibrium is minimized by the TurboMatrix Headspace Sampler's shaker feature. The headspace is then over-pressurized with argon and the content of a sampling loop is filled by the depressurization of the headspace against the ambient atmospheric pressure. The gases contained in the loop are then introduced into the system for analysis.

Results

Figure 1 illustrates a typical chromatogram obtained using the Model 4087 system. Baseline separation of the components of interest are achieved in approximately 10 min.

Conclusions

Using method C, significant time savings can be realized compared with methods A and B (Figure 2). Analyzing 100 samples will yield a savings of 16 h versus method B and 183 h versus method A.

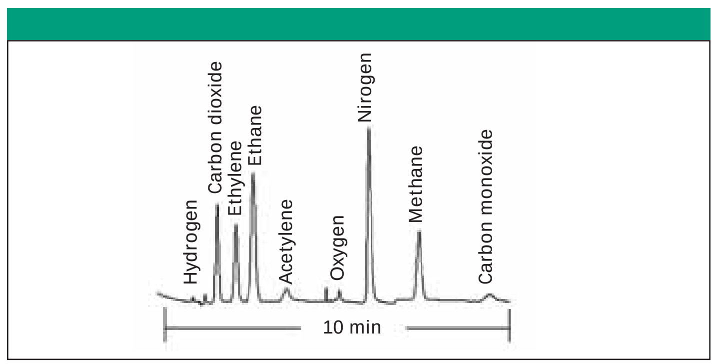

Figure 1: Typical chromatogram from the Model 4087 TOGA system.

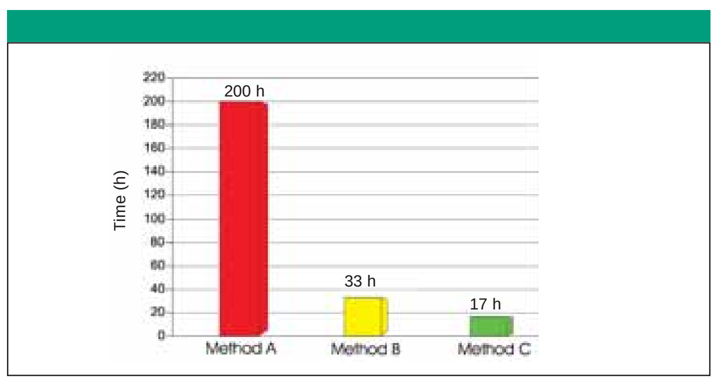

Figure 2: Comparison of time to analyze 100 samples.

Arnel Incorporated
3145 Bordentown Avenue, Parlin, NJ 08859
tel. (732) 721-4300, fax (732) 721-4551
www.arnelinc.com

© Reprinted from LCGC APPLICATION NOTEBOOK September 2005 AN ADVANSTAR PUBLICATION Printed in U.S.A.

P4000A102005