Semi-quantitative Determination of Volatile Oligomers of Halogenated Compressor Oil in a Manufacturing Process using a Person Portable GC-MS

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During the manufacturing process of industrial gases, volatile oligomers of compressor fluids can enter the product stream. These impurities are typically within specification, but failure of a diaphragm or seal can spike the concentration in a fill batch and the industrial gas no longer meets product specifications. A screening method has been developed to extract, identify and quantitate chlorotrifluoroethylene-based impurities from the compressor fluid using a CUSTODION™ solid phase micro-extraction (SPME) syringe and a TRIDION™-9 person-portable gas chromatograph–toroidal ion trap mass spectrometer (GC-TMS). The compounds were separated and detected rapidly in less than 5 min. analysis time.

In this screening method a CUSTODION SPME syringe offers a unique sampling capability where the syringe needle can be exposed to the gas containers via an appropriate interface. The SPME fiber concentrates trace amounts of the volatile components of compressor fluid. Using a TRIDION-9 GC-MS to analyze samples is fast and decision making can take place during the fill process or at least before the product container is sent to a customer. The SPME-GC-MS method can provide contaminant trace analysis at ppb level, which is typically below the lower limit of acceptance for impurities in industrial gases. This analytical method can save time and money by eliminating rework, and can assure the customer that the product meets the specification.

Experimental Conditions

Bromopentafluorobenzene (BPFB) and dibromotetraflurobenzene (DBTFB) were spiked into a Tedlar™ bag at known vapor concentrations, and were used as reference standards. Liquid compressor oil samples were weighed into vials that simulated gas containers. The volatile oligo-mers were allowed to equilibrate between the liquid and vapor phases (air, at ambient pressure) for 24 h. Both the standards and sample ana-lytes were extracted from the gas phase at ambient temperature for 2 min. using a CUSTODION SPME syringe with a 65 µm polydimethylsi-loxane/divinylbenzene (PDMS/DVB) fiber.

Following each sample extraction, the SPME syringe was inserted into the TRIDION-9 GC-TMS injection port where the target analytes were de-sorbed into a split-splitless injector (280 °C) coupled with a low thermal mass metal-clad capillary GC column (MXT-5, 5 m × 0.1 mm, 0.4 µm df). After an initial 10 s hold at 40 °C, the GC temperature was increased at 2 °C/s to 280 °C for a total run time of 2 min. and 20 s. The capillary GC is coupled to a TMS detector having a mass range of 45-500 m/z.

Total ion chromatogram area of the samples and standards were used for quantitation. Semi-quantitative results are often applied for in-field measurements. The procedures for in-field calibration are de-

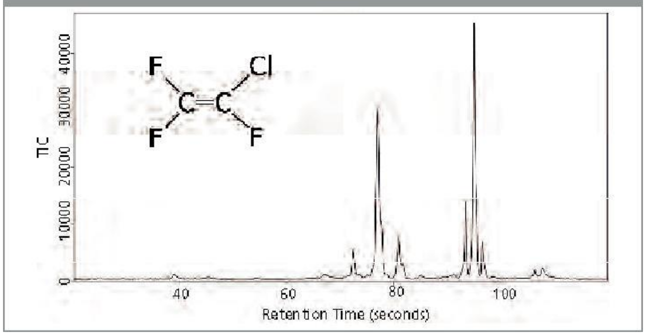

Figure 1: Total ion chromatogram of the volatile chlorotrifluoroethylene oligomers (sample 2).

signed to be simple with minimal preparation and generate a level of data quality that provides actionable results.

Results

Figure 1 shows the GC-TMS analysis of the volatile chlorotrifluoroethyl-ene oligomers samples.

Conclusions

The CUSTODION SPME syringe and TRIDION-9 GC-TMS are uniquely suited for rapid quality assurance analysis of product gases for com-pressor oil oligomers and other organic impurities in the gas phase. This method can be performed at the filling location allowing decisions regarding product purity and filling process integrity be made in real-time. The ~5 min total analysis time makes it possible to collect ad-ditional samples within the timeframe of the batch fill.

Acknowledgements

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