Composition Analysis of Contaminated and Counterfeit Refrigerants Using the Agilent 490 Micro GC

Application Note

Micro Gas Chromatography, Refrigerant Analysis, Product Quality

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Abstract

The importance of refrigerant composition analysis became clear when contaminated and counterfeit refrigerant 1,1,1,2-tetrafluorethane (R134a) appeared on the market. Chloromethane (R40), present in this counterfeit R134a, is found to be incompatible with aluminum surfaces in the refrigerant system. A chemical reaction takes place resulting in a pyrophoric compound upon contact with air. This application note highlights fast, on-site characterization of the refrigerant mixture using the Agilent 490 Micro GC.

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Introduction

A refrigerant is a single component or mixture of halogenated hydrocarbons. These chloro-, fluoro-, and chlorofluorohydrocarbons are used in a refrigeration system for a wide range of temperature control applications. Some examples are air conditioning in cars, houses, and large buildings, temperature control during chemical and pharmaceutical processes, and refrigeration during production, storage, and retailing of food.

Since the phase-out of refrigerant R12 (dichlorodofluoromethane) for environmental reasons, the use of 1,1,1,2-tetrafluoroethane (R134a) has grown. R134a is also a component in many refrigerant blends on the market. Counterfeit R134a, contaminated with chloromethane (R40) was found to create dangerous situations.

Chloromethane reacts with the aluminium compressor surfaces, used in the refrigeration plants, where trimethylaluminum is formed. This pyrophoric organometallic compound can ignite upon contact with air, resulting in a need for fast, accurate refrigerant composition determination. Moreover, for certification purposes, port authorities and regulatory bodies across the world require confirmation of the refrigerant's composition.

Cambridge Refrigeration Technology (CRT), based in the United Kingdom, provides expertise for the industry within the area of environmental testing, refrigerated systems, insulated structures, refrigerated transport, and perishable cargo storage. One of their tools in daily operations is the Agilent 490 Micro GC, providing fast compositional analysis of a refrigerant mixture. The instrument's dimensions and low operational gas consumptions facilitates transportability, and enables easy relocation when tests are finalized.

Instrument and sampling information

For this analysis, the 490 Micro GC (p/n G3581A) was used. The 490 Micro GC can accommodate one to four independently controlled channels, all packaged in a shoe-box sized, portable instrument. Each channel includes an electronic carrier gas control, micro-machined injector, narrow-bore capillary column and micro-thermal conductivity detector (μTCD). For this application, the instrument was equipped with a 20 meter Agilent J&W PoraPLOT U column channel [1]. Table 1 shows the method settings for this application.

Table 1. Simplicity - The Agilent 490 Micro GC Requires Just a Few Settings to Complete the Method

Agilent 490 Micro GC with J&W PoraPLOT U, 20 m (p/n SP1 G3581-0023)	
Injector temperature	80 °C
Column temperature	120 °C
Column pressure	165 kpa, helium
Injection time	5 ms
Sample line temperature	110 °C
Sampling time	20 seconds

From the refrigerant bulk gas supply, a sample was collected in a gas sampling bag. The tedlar bag was directly connected to the 490 Micro GC's sample inlet. The system's chip injector was flushed and loaded using the built-in sample pump. This sample pump, located behind the injector, eliminates sample carryover.

Results and Discussion

The 490 Micro GC, equipped with a J&W PoraPLOT U column channel, separated chloromethane (R40) from the other refrigerants in the sample in just over 3 minutes. A column length of 20 m was chosen since all the refrigerants of interest elute between propane and *i*-butane, as shown in the comparison with the hydrocarbon mix (Figure 1B). This column length provided more separation power, due to the increased number of theoretical plates. A known refrigerant mixture used as a calibration mixture, in Figure 1B, shows baseline separation of all compounds of interest. Sample analysis results for a R134a refrigerant, chromatogram depicted in Figure 1A, clearly shows that this refrigerant was contaminated with chloromethane.

Instead of taking a sample and bringing it to the lab, which can take up to a few days before the results are known, CRT performs direct, on-site compositional analysis of the refrigerant gas. As a result, shipment purity could be easily determined, and sample integrity was secured. Moreover, out-of-spec values were directly communicated, and corrective actions taken accordingly.

Compared to sensor-based refrigerant detectors available on the market, the 490 Micro GC delivers a full characterization of the sample. It provides many more details about the refrigerant composition and, with R134a, its potentially dangerous impurities.

Conclusion

Cambridge Refrigeration Technology used the Agilent 490 Micro GC for its speed of analysis. The system enabled reliable and fast refrigerant compositional analysis; chloromethane impurities in refrigerant mixtures were identified in just over 3 minutes using an Agilent J&W PoraPLOT U column channel. No specialized operator personnel were required due to the simplified instrument setup. With just a handful of method parameters, the system was ready to go.

Portability, featuring the system's shoe-box size dimensions and low consumption of operating gas, was another reason to use the 490 Micro GC. System relocation is important for CRT as refrigerated systems, located around the world, require periodic check-ups and refrigerant refills. On-site analysis eliminated sample storage and shipment, ensuring direct availability of sample results and securing the integrity of the sample. When values are found to be outside the purity specification document, direct corrective action can be taken.

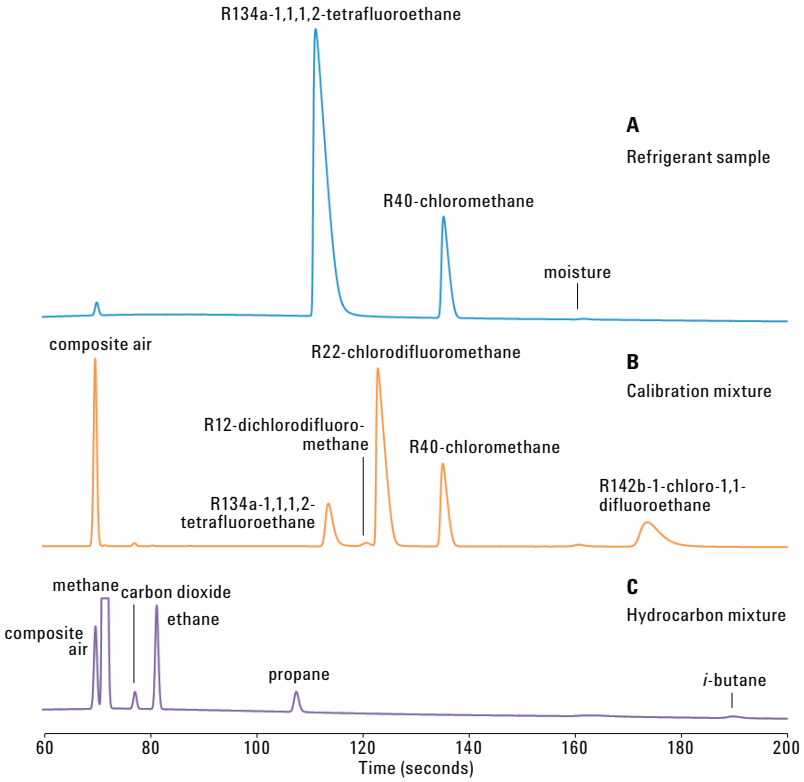

Figure 1. Speed-of-analysis - The Agilent 490 Micro GC delivers fast characterization of the refrigerant mixture.

Reference

1. G. A Sturrock, P. G. Simmonds, G. Nickless, D. Zwiep. "Analysis of chlorofluorocarbon replacement compounds by capillary gas chromatography" *J. of Chromatog.*, **648**, 423-431 (1993).

For More Information

These data represent typical results. For more information on our products and services, visit our Web site at www.agilent.com/chem.

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