

Sensitive, selective detection and identification of a wide variety of chemical species is a necessity in many applications including chemical warfare agents (CWAs), explosives, environmental pollutants, as well as other toxic industrial compounds and materials (TICs/TIMs). In many instances, field-deployable sensors are preferred because valuable time, resources and chemical information are conserved by performing analyses directly on-site, rather than retrieving samples to be studied at a later time in the laboratory. The ICx family of mass spectrometers have been developed to fulfill this need. Based on Cylindrical Ion Trap (CIT) technology, the products are compact and transportable. One widely applicable sample introduction method supported is a Low Thermal Mass Gas Chromatograph (LTM-GC). Performance characterization of the LTM-GC-equipped product using the diagnostic Grob mixture is presented. It has been designed to perform rapid separations without significantly compromising performance.

In order to assess the conditioning of the chromatographic system and separation capabilities over a wide range of temperature regimes, Grob mixture (Sigma Aldrich, St. Louis, MO) was used. This diagnostic mixture contains various classes of organic components including hydrocarbons, fatty acid methyl esters (FAMES), acids, bases and alcohols^{1,2} (Table 1). The resulting elution order and chromatographic peak shapes are indicative of column type and age/condition. Periodic analysis of the Grob mixture allows for convenient assessment of the columns deterioration over the period of use.

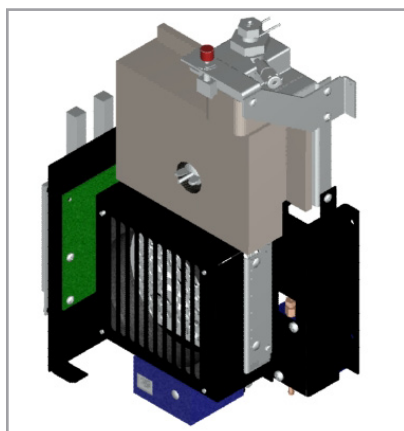


Figure 1: Low Thermal Mass Gas Chromatograph

ANALYTE CLASS	APPRAISAL FUNCTION
Hydrocarbon (n-dodecane)	Proper column installation, system integrity
FAMES (methyl undecanoate)	Column separation efficiency, number of theoretical plates
Acid (2-ethylhexanoic acid), Bases (N-cyclohexanamine)	Active sites or underivatized surfaces
Alcohol (1-octanol)	Active sites in column or injector

Table 1: Grob mixture components and diagnostic function

The total ion current plot obtained for 1 μ L injection of Grob mix standard is shown in Figure 2. The blue total ion chromatogram (bottom) shows the results of a separation of the Grob mixture components using a conventional temperature program, i.e. with a column temperature ramp of 6°C/min. It is clear that the column is performing well in that all the components are present and the individual peaks are sharp and symmetrical. The only peak that is not sharply defined is that of 2-ethylhexanoic acid which is to be expected due to the non-polar nature of the Rtx-1MS column. The hydrocarbon and FAMES peaks show the column is properly installed and the peaks resulting from the alcohol components indicate that there are no significant active sites within the injector or column.

Also shown in Figure 2 is a Grob analysis performed with an accelerated temperature profile. The data is shown as the red total ion chromatogram on the same timescale as the previous, slower separation (top left). In addition, an expanded view of the Grob peaks is provided (top right). The low-thermal mass column allowed for a temperature ramp of 80°C/min which provided baseline separation for all but two of the components. Even with such a rapid separation, the peaks appear symmetrical indicating good separation efficiency. As in the conventional separation, the distorted shape of the 2-ethylhexanoic acid peak can be attributed to the use of a non-polar stationary phase (Rtx-1MS).

These data represent typical results.

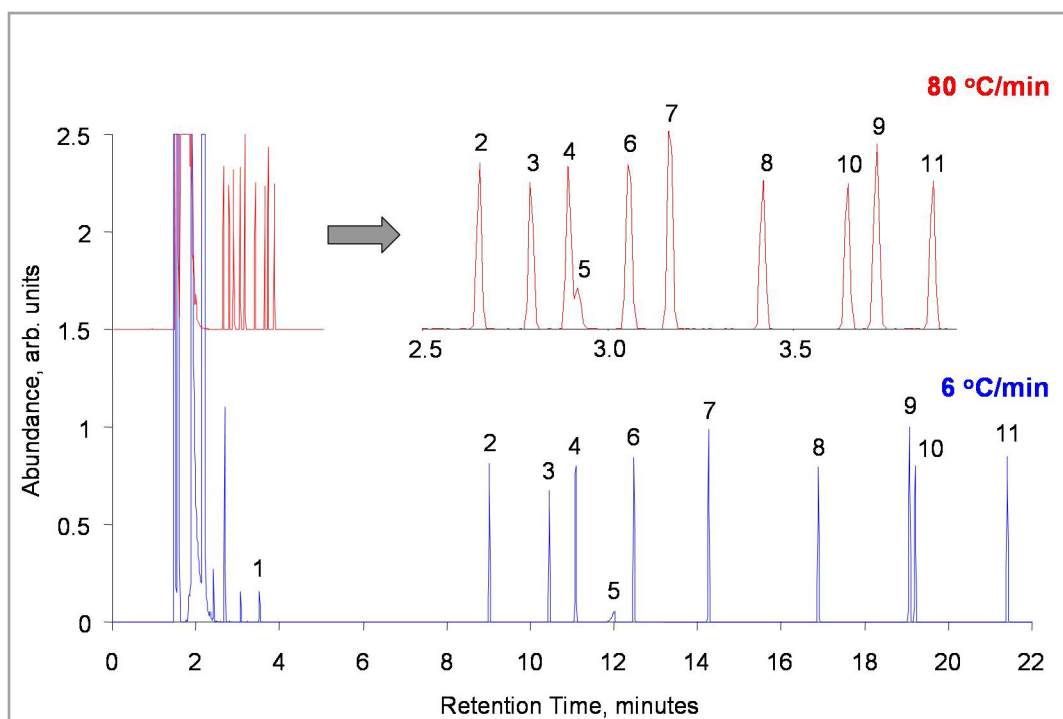


Figure 2: Total ion current traces for injections of Grob test mixture on the ICx LTM-GC/MS with GC temperature ramps of 6°C/min (blue) and 80°C/min (red)

GC column: Rtx-1MS, 30 m
0.25 mm i.d.
0.25 μ m df

Carrier Gas: He, 1 mL/min
15:1 split ratio

Grob Mixture Components:

- 1) 2,3-butanediol
- 2) n-decane
- 3) 1-octanol
- 4) 2,6-diphenylphenol
- 5) 2-ethylhexanoic acid
- 6) 2,6-dimethylbenzenamine
- 7) n-dodecane
- 8) methyl decanoate
- 9) N-cyclohexanamine
- 10) methyl undecanoate
- 11) methyl decanoate

References

1. Grob, Jr., K., G. Grob, and K. Grob, Comprehensive, Standardized Quality Test for Glass Capillary Columns, J. Chrom., 156, 1-20, 1978.
2. Grob, K., G. Grob, and K. Grob, Jr., Testing Capillary Gas Chromatographic Columns, J. Chrom. 219, 13-20, 1981.

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