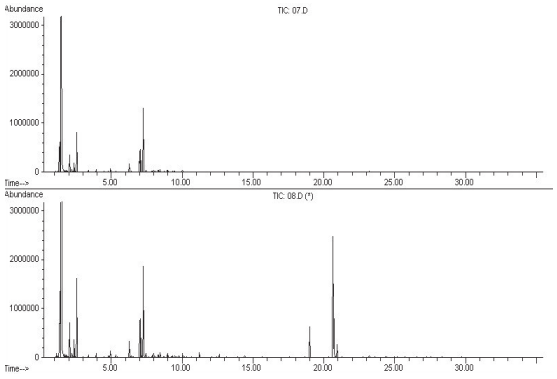
GAS CHROMATOGRAPHY / MASS SPECTROSCOPY

DESCRIPTION OF TECHNIQUE

Gas chromatography / mass spectrometry (GC/MS) is the marriage of two analytical methods into a versatile technique for the identification of complex volatile materials. Gas chromatography (GC) effectively separates the different constituents of the sample for subsequent analysis and identification by mass spectrometry (MS).

The chromatographic separation relies on the interaction of the sample with a mobile phase and a stationary phase within the GC instrument column. The sample is carried through the column by the mobile phase, typically an inert gas. However, the sample is slowed in its travel through the column as the sample molecules repeatedly adsorb and desorb from the stationary phase in the column. The affinity of a particular molecule for the stationary phase determines the retention time of that constituent in the column. The molecules for each component of the sample will travel through the column at nearly the same rate and exit (elute) from the column within a narrow time band that is specific to that component. Thus, compounds with different retention times in the column are physically separated for presentation to a detector and analyzer.



Total Ion Chromatogram for Two Vegetable Oils

The typical GC capillary column consists of a small-diameter tube with a thin film of a high-molecular-weight polymer coated on the inside. The polymer is the stationary phase for the chromatographic process. The mobile phase can be any inert gas, but is typically helium. The instrument also includes a heated injection port to vaporize all volatile constituents of the sample and an oven to keep the constituents in gas form as they pass through the column.

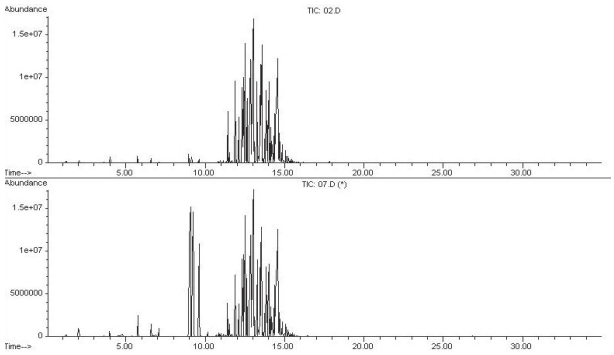
As a sample constituent elutes from the GC column, it enters the ionization chamber of the mass spectrometer where the molecules are ionized, typically by electron impact. When an electron impact with a sample molecule results in the loss of an electron from the molecule, a positive ion is formed. Some of the molecular ions are further fragmented into daughter ions and neutral fragments. The positive ions are then repelled out of the ionization chamber by a small positive charge within the chamber. Negative ions are also formed by the electron impact, but are not analyzed.

The positive ions are separated according to their mass by a mass analyzer. The mass analyzer most commonly used in GC/MS is the quadrapole filter, in which the ions pass by four hyperbolic magnetic poles created by a radio frequency field. The magnetic poles separate the ions by their mass/charge ratio, successively focusing ions with increasing mass onto a detector for counting. The analyzer scans step-wise through a set range of mass values to evaluate the relative abundance of ions at each mass value. The quadrapole filter can perform a complete mass scan within the duration of a single GC elution band.

ANALYTICAL INFORMATION

Material Identification - The first result from the compiled data is a total-ion chromatogram (TIC), which is a plot of the total mass eluting from the GC and detected by MS as a function of time. Each peak or band in the chromatogram represents a discrete chemical compound, or a mixture of compounds with identical retention times. The retention times in the chromatogram provide the first indication of the sample constituents.

More specific identification of the compound(s) for each band can then be made from the mass spectrum corresponding to the band. Compounds are identified from the mass spectrum by their unique ion fragmentation patterns. This compound identification analysis is performed by a computerized comparison of the mass spectra for the sample with spectra library for known compounds.



Chromotagram for Outgassing Compounds from Two Epoxies

Quantitation - The analysis results can be quantified using the data from the chromatogram. The area under each peak in the chromatogram is proportional to the concentration of the compounds represented by that peak. The concentration for each compound in the sample is calculated from a standard curve of known concentrations established for that compound. The analysis sensitivity can be as low as a few nanograms.

TYPICAL APPLICATIONS

- Identification of foreign material contamination
- Analysis of outgassing products for disk drive components
- Identification of polymer additives
- Analysis of polymer cure by-products

SAMPLE REQUIREMENTS

The samples for GC/MS can be gases, liquids, or solids. However, only those constituents that are gaseous and stable at the analysis temperature can be analyzed. Gases and liquids can be injected directly into the sample injector. Volatile compounds from solid materials are collected using a headspace sampler. The sample from a headspace attachment can be collected and injected at one time (static headspace), or the sample can be collected and analyzed continuously over a set time period (dynamic headspace).