Training Guidelines for the Fire Debris Analyst

Lesson Plan (Module) 12

Date: December 2004 **Instructor:** Qualified Instructor

Subject: Mass Spectrometry **Total Time:** 12 hours

Learning Objectives

- Demonstrate a basic understanding of how a mass spectrometer operates (instrumentation).
- Demonstrate a basic understanding of mass spectrometry theory.
- Describe selected ion monitoring and how extracted ions are selected.
- Demonstrate how to properly interpret mass spectral data.

Suggested Reading

1. Arson Analysis by Mass Chromatography, Smith, R.M., Analytical Chemistry, Vol. 54 (13), pages 1399A-1409A, 1982.
2. Mass Chromatograpic Analysis of Arson Accelerants, Smith, R.M., Journal of Forensic Sciences, Vol. 28 (2), pages 318-329, 1983.
3. Recent Advances Toward the Detection of Accelerants in Arson Cases, Holzer, C., Bertsch, W., American Laboratory, pages 15-19, Dec. 1988.
4. Gas Chromatography-Mass Spectroscopy of Simulated Arson Debris Using Gasoline as an Ignitable liquid, Mach, M.H., Journal of Forensic Sciences, Vol. 22 (2), pages 348-357, 1977.
5. Accelerant Identification in Fire Debris by Gas Chromatography/Mass Spectrometry Techniques, Kelly, R., Martz, R., Journal of Forensic Sciences, Vol. 29 (3), pages 714-727, 1984.
6. "Automated Sampling and Computer Assisted Identification of Hydrocarbon Accelerants," Tontarski, R., Strobel, R., Journal of Forensic Sciences , Vol. 27 (2), pages 710-714, 1982.
7. Arson Analysis by Mass Spectrometry, Chapter 5, Forensic Mass Spectrometry, J. Yinon (Ed.), CRC Press, Inc., Boca Raton, FL, pages 131-159, 1987.
8. GC/MS Data from Fire Debris Samples: Interpretation and Applications, Wallace, J.R., Journal of Forensic Sciences, Vol. 44 (5), pages 996-1012, 1999.
9. Detection of Petroleum-Based Accelerants in Fire Debris by Target Compound Gas Chromatograph/Mass Spectroscopy, Keto, R.O., Wineman, P.L., Analytical Chemistry, Vol. 63, pages 1964-1971, 1991.
10. An Accelerant Classification Scheme Based on Analysis by Gas Chromatography/Mass Spectrometry (GC-MS), Nowicki, F.F., Journal of Forensic Sciences, Vol. 35 (5), pages 1064-1086, 1990.

11. A GC-MS Database of Target Compound Chromatograms for the Identification of Arson Accelerants, Lennard, C.J., Rochaix, V.T., Margot, P., Science and Justice, Vol. 35 (1), pages 19-30, 1995.
12. ASTM E 1618.,Standard Guide for Identification of Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.
13. GC-MS Guide to Ignitable Liquids, Newman, Reta, Gilbert M. and Lothridge, K., CRC Press, 1998.

Introduction

This lesson covers the theoretical basis of mass spectrometry and its application to the analysis of ignitable liquids and other materials.

Outline

1. Mass Spectrometer
   - a. Comparison of GC detectors
     - i. strengths and weaknesses
     - ii. sensitivity
     - iii. resolving power
   - b. Components
     - i. vacuum systems
     - ii. GC/MS interfaces
     - iii. electron impact ionization
     - iv. chemical ionization
     - v. mass separation methods
       - quadrupole
       - ion trap
       - magnetic sector
       - time of flight
       - MS/MS
     - vi. detection/ion abundance determination
   - c. Operation and maintenance
     - i. tuning and calibration
     - ii. routine maintenance

2. Basic Interpretation of Mass Spectral Data
   - a. TIC
   - b. Molecular ions
   - c. Base peaks
   - d. Nitrogen rule
   - e. Isotopic ratios
   - f. Fragmentation
   - g. Evaluation of the quality of mass spectral data

h. Libraries

3. Selected Ion Monitoring

   a. Chemical structure review
      i. alkanes
      ii. alkenes
      iii. aromatics
      iv. naphthalenes
      v. polynuclear aromatics
      vi. indanes and indenes
      vii. styrenes
      viii. terpenes
   b. Extracted ions
      i. selection of ions to monitor
      ii. use of ratios
   c. Comparison to standards and references

4. Sample Matrix Effects

   a. "filtering" out interfering compounds
   b. microbial degradation in soil
   c. pyrolysis of polyethylene and other plastics
   d. wood thermal degradation

Teaching Aids

Handout
PowerPoint presentation
Exercises

Summary

Mass spectrometry, when interfaced with gas chromatography is a powerful and valuable analytical tool for identifying ignitable liquids, whether they are single component product or complex mixtures of hydrocarbons. The technique is sensitive and discriminating, capable of offering a level of data not available by other GC detection systems. Mass spectrometry offers the additional advantage of being able to "filter" out pyrolysis products or other interfering compounds.

Test Questions

1. A mass spectrometer is one of several available detectors for a gas chromatograph. **True** or False
2. Electron impact is the predominant form of ionization for fire debris analytes. **True** or False

3. A fundamental understanding of mass spectrometry theory is not needed for evaluation of mass spectral data. True or **False**

4. Alcohols and other very volatile compounds are not detected by GC-MS. True or **False**

5. The ratio of alkanes to aromatics can become important in evaluating mass spectral data. **True** or False

6. Extracted ions for aromatic compounds typically include
   a. 43, 57, 71, 85
   b. 55, 69, 83
   c. 91, 105, 119
   d. 128, 142, 156

7. Extracted ions for alkenes/cycloalkanes typically include
   a. 43, 57, 71, 85
   b. 55, 69, 83
   c. 91, 105, 119
   d. 128, 142, 156

8. An instrument library match of a mass spectrum is adequate for identifying a liquid. True or **False**

9. The presence of terpenes in a sample can significantly interfere with the interpretation of an extracted ion profile. True or **False**

10. The presence of toluene, ethylbenzene and xylenes in a sample is a sure indication of gasoline. True or **False**