Training Guidelines for the Fire Debris Analyst

Lesson Plan (Module) 11

Date: November 2006 **Instructor:** Qualified Instructor

Subject: Gas Chromatography **Total Time:** 10 hours

Learning Objectives

- Understand the theoretical aspects of chromatography.
- Understand the theoretical aspects of gas chromatography.
- Understand the advantages and disadvantages of gas chromatography.
- Demonstrate familiarity with gas chromatography terminology.
- Demonstrate familiarity with instrumentation.
- Demonstrate how to properly interpret gas chromatographic data.

Suggested Reading

1. Modern Practice of Gas Chromatography, Grob, R.L and Barry, E.F., John Wiley & Sons, 2004.
2. Principles of Instrumental Analysis, 4th ed., Skoog, D.A., et al., Saunders College Publishing, Chapter 18 (Mass Spectrometry), Chapter 25 (Introduction to Chromatographic Separations) and 26 (Gas Chromatography), 1998.
3. Gas Chromatography in Forensic Science, Tebbett, I., Ellis Horwood (pub.), Chapter 1 "Forensic gas chromatography", Stafford, D.; and Chapter 5 "Gas chromatography in arson and explosives analysis", Fultz, M.L. and DeHaan, J.D. 1992.
4. Forensic Science Handbook, Saferstein, R. (ed.), Vol. I, Prentice-Hall, Chapter 3 (Forensic Applications of Mass Spectrometry), 1982.
5. A Study to Determine the Limit of Detectability of Gasoline Vapor from Simulated Arson Residues, Loscalzo, P.J., DeForest, P.R., Chao, J.M., Journal of Forensic Sciences, Vol. 25, (1), pages 162-167, 1980.
6. Relative Hydrocarbon Detectability by Flame Ionization Detection for Various Isolation Methods, Cooper, D.G., Stackhouse, C., Arson Analysis Newsletter, Vol. 6, page 73, 1982.
7. ASTM E 1386-00, "Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction", ASTM International.
8. ASTM E 1387-01, "Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography", ASTM International.
9. ASTM E 355-96 (2001), "Standard Procedure for Gas Chromatography Terms and Relationships", ASTM International.
10. ASTM E 1510-95 (2005), "Standard Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs", ASTM International.

11. ASTM E 594-96 (2006), “Standard Practice for Testing Flame Ionization Detectors Used In Gas or Supercritical Chromatography”, ASTM International.

Introduction

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

Outline

1. Theoretical aspects
    a. History of chromatography
    b. Introduction to the various chromatographic methods
    c. Gas/liquid phase equilibrium
    d. Van Deemter curves
    e. Cross contamination
    f. Temperature vs. retention behavior

2. Chromatographic columns
    a. Liquid
    b. Packed
    c. Polar/non-polar
    d. Column efficiency
    e. Resolution

3. Carrier Gas
    a. Gas selection
    b. Flow rate
    c. Troubleshooting

4. Detectors
    a. Thermal
    b. Flame ionization
    c. Photoionization
    d. Mass Spectrometry
    e. Other

5. Quantitative evaluation
    a. Triangulation
    b. Correlation of area and quantity
    c. Peak characteristics

6. Qualitative evaluation
    a. Peak pattern comparison (with standards)
    b. Instrument Coupling

i. GC/MS
ii. GC/MS/MS
iii. GC/FTIR

Teaching Aids

1. Provide actual GC runs of various volatile components. Demonstrate actual events of contamination, overload, improper oven temperatures and other factors that have an affect on interpretation.
2. Practical exercises involving various gas chromatographic parameters.
3. Practical exercises involving interpretation of gas chromatographic data.
4. PowerPoint presentation
5. Attend a course from an instrument manufacturer.

Summary

After completion of this module, the student should be able to determine correct sample preparation per sample attributes, how to obtain the best signal, the required instrumentation parameters per the specifics of each sample and be able to interpret a chromatographic chart.

Test Questions

1. Which of the following gases provides the best chromatographic separation over the widest temperature range?
   a. air
   b. hydrogen
   b. helium
   c. nitrogen

2. The temperature of the injection port should be:
   a. hotter than the final oven temperature
   b. room temperature
   c. optimized for the desired results
   c. 250^0C

3. Tailing of the peak can be an indication of:
   a. weak sample
   b. active sites in the GC system
   b. empty carrier gas
   c. faulty injection port septum

4. Inadequate resolution of two or more components could yield a chromatographic profile exhibiting:
   a. fronting
   b. shouldering
   b. tailing
   c. ghost peaks

5. What is the function of makeup gas?
   a. to optimize the flame
   a. to burn residue
   b. clean the detector
   c. provide fuel for the flame

6. Retention time can be affected by:
   a. carrier gas leakage
   b. insufficient carrier gas pressure
   c. overloaded sample
   d. deviation from the oven temperature program
   e. all of the above

7. Which of the following is a non-polar stationary phase?
   a. phenymethylpolysiloxane
   b. cyanopropylpolysiloxane
   c. Tenax
   d. dimethylpolysiloxane

8. Single column gas chromatographic results are a means of positive identification for a single component. True or **False**

9. What physical properties affect the elution order of components on a non-polar column?
   a. vapor pressure
   b. molecular weight
   c. polarity
   d. unsaturation
   e. all of the above

10. A gas chromatograph calibration can be used as a check of:
    a. deviation in retention time
    b. reduced detection limits
    c. proper instrument functioning
    d. injection technique
    e. all of the above

11. A blank chromatogram can be the result of:
    a. a faulty septum

b. a blank sample
c. plugged column
d. flame is out in the FID
e. all of the above