

# **Idaho State Police Forensic Services**

# Fire Debris Analysis Training Plan



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# **Revision History**

| Revision # | Description of Changes   |
|------------|--|
| 1          | Original Qualtrax issue  |
| 2          | Changed 5.4.2.11, 7.4.5.6, 8.4.18.1.5, 8.4.18.1.7, 8.4.19.4.2, 10.4.54.2, 10.4.55.5,  Numbering in section 7.3 |
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Fire Evidence Training Manual

Revision 2

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# 1.0 Introduction

1.1 This training plan is intended as a guide to introduce a forensic analyst to the many aspects of fire evidence and its analysis. It is designed to provide the analyst with the background necessary to process fire evidence to recover, identify and classify ignitable liquids. In no particular order this training plan addresses issues such as how fire evidence must be packaged, appropriate safety measures, relevant principles of chemistry, the chemistry and physics of fire, products generated from substrate materials and petroleum refining. For the fire chemist to effectively communicate with fire investigators, fire scene processing and the Idaho Statutes pertaining to arson are also addressed. The plan includes the methods for the recovery of ignitable liquids from fire evidence, instrumental methods of analysis, classification of ignitable liquids, and the interpretation of detected ignitable liquids.

# 1.2 Approach to Training

- 1.2.1 To address training plan subject matter, the cited references, or equivalent, must be reviewed by the Trainee. The training references provided or a newer edition should be used. Both the education and work experience of the Trainee will be considered; however, at a minimum, a verbal review of material must be done to the satisfaction of the Trainer.
- 1.2.2 The trainee will demonstrate competency in training plan subject matter, answers to training plan questions will be provided in written form.
- 1.2.3 All training does not have to proceed in the order used in this training plan

# 1.3 Hands of the Analyst during training,

- 1.3.1 As part of the training process, the Trainee will perform un-supervised hands-on analysis on exercise samples, simulated case samples and/or "old" proficiency tests.
- 1.3.2 The trainee will observe the trainer performing casework and can assist the trainer when appropriate, as the hands of the analyst. This includes, but is not limited to, such tasks as reference material preparation as well as data analysis.
- 1.4 This training is divided into several different modules. Generally each module has a mixture of exercises and tests that must be completed to the satisfaction of the discipline leader. Answers to all test questions must be correct. Partial or incorrect answers will be reviewed with the trainer and corrections will be resubmitted. All tests will be kept by the technical leader.

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- 1.5 A competency test consisting of at least five sample types must be completed with 100% accuracy.
- 1.6 After the competency test has been completed the trainee will present the results in a mock court.
- 1.7 Upon successful completion of the mock court the trainee will perform the number of supervised cases as determined by the discipline leader.
- 1.8 Continual Awareness of Relevant Literature

  The new or experienced analyst is reminded that this training plan only
  addresses the core of training for fire evidence analysis. After the completion
  of training, the analyst is responsible for keeping their knowledge current
  through continual literature review. This must include relevant journals,
  newsletters, textbooks and national data bases.
- 1.9 Technical review. After a minimum of five cases have been completed the trainee may start technical review training. A minimum of five cases will be reviewed by the trainee and the reviews verified by the technical leader. The number of cases required for review may be increased depending on case content and any errors detected in the review process.



# 2.0 Roles and Responsibilities

### 2.1 Supervisor

The supervisor in coordination with the Technical Lead, Trainer, and the Trainee will set up a schedule outlining expectations for the trainee and will then monitor the progress of the trainee.

#### 2.2 Technical Lead

The Technical Lead (TL) will appoint a Trainer and monitor the trainee's progress. The TL will report to the supervisor any delays to the training schedule. The TL will be available to the trainer to answer any questions. The TL will review results from the exercises, arrange and grade competency tests, and mock courts.

#### 2.3 Trainer

The Trainer will provide the trainee with the materials necessary to complete training. Materials include but are not limited to samples, equipment and most importantly knowledge. They will keep the TL up to date on the progress of the trainee.

#### 2.4 Trainee

In order to get the most out of this training it is important for trainees to realize that the information provided through this manual is only a portion of what will be needed in order to not only become proficient but also to become a productive member of the team. It is incumbent on the trainee to utilize not only the information presented but also knowledge learned in school and/or from previous work experience. The use of outside sources of information is encouraged as are questions.

# 3.0 General Laboratory

# 3.1 Background and Theory

The trainee should start their training with a fundamental understanding of good laboratory practices learned from classes at school.

# 3.2 Objectives, Principles, and Knowledge

To gain and hone the skills necessary to be able to conduct analysis and testify to the results for the justice system

# 3.3 Health and Safety Hazards

Universal Precautions should be practiced at all times
PPE is provided, but it is up to the trainee to protect themselves. Parts of the training consists of using open flame, use caution.

# 3.4 Reading and Practical Exercises

Most sections will contain reading and some practical exercises.

- 3.4.1 Review Fire Debris Analytical methods
- 3.4.2 Demonstrate competence in using laboratory equipment.



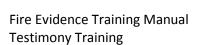
# 4.0 Testimony Training

# 4.1 Background and Theory

At its heart, Forensic Science applies scientific theory and analysis to items of evidence. The end goal of this analysis is to be able to present the results in a court of law. The forensic scientist must not only be competent in performing the analysis but must be able to explain and defend what they did and why they did it. It is assumed that their audience does not possess scientific knowledge, as such, the forensic scientist must be able to explain complex principals and procedures in layman's terms.

# 4.2 Objectives, Principles, and Knowledge Through observation of actual testimony, discussions with the trainer, preparations for and participating in mock court, the trainee will gain familiarity with presenting testimony in court.

- 4.3 Health and Safety Hazards
  NA
- 4.4 Reading and Practical Exercises
  - 4.4.1 Observation of court testimony, at least three times and in different disciplines if possible.
  - 4.4.2 Prepare for and attend their own mock courts. Attend other mock courts if possible.
  - 4.4.3 Pass the trainee's mock court using the competency test as the case record.



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# 5.0 Chemistry and Physics of Fire and Fire Investigation

# 5.1 Background and Theory

Fire is a complex heat generating chemical reaction involving a fuel and an oxidizer. By knowing these basic requirements and the behavior of fire an investigator may be able to identify the cause of a fire and explain its behavior.

- 5.2 Objectives, Principles, and Knowledge
  - 5.2.1 Familiarization with the Basic Elements of Fire Behavior.
  - 5.2.2 The trainee should demonstrate an understanding of the following:
    - 5.2.2.1 The concept of a fire triangle versus fire tetrahedron.
    - 5.2.2.2 The three modes of heat transfer.
    - 5.2.2.3 The phases of the burning process
  - 5.2.3 Define the phenomenon of fire; describe each of the four classifications of fire.
  - 5.2.4 Combustion Chemistry including the concept of pyrolysis and the resulting pyrolysis products produced at the fire scene.
  - 5.2.5 Properties of Ignitable Liquids
  - 5.2.6 Ignition Sources
  - 5.2.7 Effects of heat and fire
  - 5.2.8 Effects of evaporation and combustion of ignitable liquids
  - 5.2.9 Fire Suppression
  - 5.2.10 Fire investigation
    - 5.2.10.1 Scene preservation and contamination
    - 5.2.10.2 Identification of origin
    - 5.2.10.3 Identification of ignition sources
    - 5.2.10.4 Application of the scientific method to the fire investigative process
    - 5.2.10.5 Establishment of cause
    - 5.2.10.6 Selection of laboratory samples and comparison samples
    - 5.2.10.7 Recovery and packaging (See also Section 3)
    - 5.2.10.8 Investigator's expectations of forensic analysis
    - 5.2.10.9 Identification of product
    - 5.2.10.10 Unique source identification
- 5.3 Health and Safety Hazards

NA

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# 5.4 Reading and Practical Exercises

- 5.4.1 Background Reading
  - 5.4.1.1 DeHaan, J.D., Chapters 2, 3, 4, 5, 6, and 7 in: Kirk's Fire Investigation, 7th edition, Prentice Hall: New Jersey, 2012.
  - 5.4.1.2 DeHaan, J.D. and Bonarius, K. *Pyrolysis Products of Structure Fires*, J. For. Sci Soc, 28(5/6):299-309, 1988.
  - 5.4.1.3 Clodfeller, R.W. and Hueske, E.E. *A Comparison of Decomposition Products from Selected Burned Materials with Common Arson Accelerants*, J. For. Sci, 22(1): 116-118, 1977.
  - 5.4.1.4 Stauffer, E., *Concept of pyrolysis for fire debris analysts*, Sci & Justice, 43(1):29-40, 2003.
  - 5.4.1.5 Stauffer, E., Sources of Interference in Fire Debris Analysis, pp. 191-225. *in*: Fire Investigation, Daéid, N.N ed, CRC Press:Boca Raton, 2004.
  - 5.4.1.6 Daéid, N.N., An Introduction to Fires and Fire Investigation Chapter 1, pp. 1-12. *in*: Fire Investigation, Daéid, N.N ed, CRC Press:Boca Raton, 2004.
  - 5.4.1.7 Daéid, N.N., Fires from Causes Other Than Electrical malfunctions: Theory and Case Studies Chapter 2, pp. 13-59. *in*: Fire Investigation, Daéid, N.N ed, CRC Press:Boca Raton, 2004
  - 5.4.1.8 Idaho Code Title 18, Crimes and Punishments, Chapter 8, Arson: §18-801, §18-802, §18-803, §18-804 and §18-805.
- 5.4.2 Submit definitions of the following terms to the trainer.
  - 5.4.2.1 Accelerant
  - 5.4.2.2 Arson (and related legal definitions)
  - 5.4.2.3 Auto Ignition Temperature
  - 5.4.2.4 Combustible Liquid
  - 5.4.2.5 Conduction
  - 5.4.2.6 Convection
  - 5.4.2.7 Deflagration
  - 5.4.2.8 Detonation
  - 5.4.2.9 Direct Flame Impingement
  - 5.4.2.10 Fire
  - 5.4.2.11 Cause of Fire
  - 5.4.2.12 Fire Tetrahedron
  - 5.4.2.13 Flammable Limit
  - 5.4.2.14 Flammable Liquid
  - 5.4.2.15 Flash Point
  - 5.4.2.16 Flame Point
  - 5.4.2.17 Flaming Fire

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- 5.4.2.18 Glowing Fire
- 5.4.2.19 Ignition
- 5.4.2.20 Point of Origin
- 5.4.2.21 Pyrolysis
- 5.4.2.22 Radiation
- 5.4.2.23 Ignition Temperature
- 5.4.2.24 Spontaneous Ignition
- 5.4.2.25 Lower Explosive Limit
- 5.4.2.26 Vapor Density
- 5.4.3 Answer these key points and submit to the trainer:
  - 5.4.3.1 What factors are needed for a fire to start? What is needed for a fire to progress?
  - 5.4.3.2 Describe how fire occurs, progresses and behaves.
  - 5.4.3.3 What happens to an ignitable liquid when it is exposed to a fire?
  - 5.4.3.4 What are the fire investigator's goals at a fire scene? How does the fire investigator proceed during an investigation? What steps are taken to determine the cause and origin of a fire?
  - 5.4.3.5 How should samples be collected from a fire scene? How should they be stored? Why?
  - 5.4.3.6 What are the fire investigator's expectations of and needs of the laboratory? When can and can't those expectations or needs be met?
  - 5.4.3.7 What is NFPA 921?
  - 5.4.3.8 Define what is meant by overhaul of a fire scene. Discuss why selective overhaul by fire personnel is crucial for reconstruction purposes.
  - 5.4.3.9 Discuss potential sources of contamination by ignitable liquids at the fire scene and ways to prevent or minimize them.
  - 5.4.3.10 Discuss the factors affecting the potential to recover an ignitable liquid from a fire scene.
  - 5.4.3.11 Define the crime of arson based on Idaho Code §18-801.
  - 5.4.3.12 Describe the key elements for being charged with arson in the first (§18-802), second (§18-803) and third degree §18-804).
  - 5.4.3.13 Describe what events took place for a person to be charged under §18-805, aggravated arson.
- 5.4.4 A written examination must be successfully completed before proceeding to the next module.

# 6.0 Sources and Classifications of Ignitable Liquids and Review of Organic Chemistry

# 6.1 Background and Theory

Before analysis of an ignitable liquid can begin an understanding of what one is and where it comes from is essential.

- 6.1.1 A review of oil refinery processes
- 6.1.2 A review of basic organic chemistry.

### 6.2 Objectives, Principles, and Knowledge

- 6.2.1 Demonstrate a basic understanding of the process of refining petroleum products from crude oil.
- 6.2.2 Describe and evaluate domestic and foreign sources of crude oil used in petroleum product manufacturing.
- 6.2.3 Describe the distillation process of crude oil in terms of the categories of products recovered.
- 6.2.4 Describe the general physical and chemical properties of each distillation fraction.
- 6.2.5 Relate these fractions to commercially available petroleum products and their use.
- 6.2.6 Describe other physical separation techniques used in refinery processes.
- 6.2.7 Describe the chemical conversion processes of cracking, alkylation, reformation, and others, used to increase yield and improve specifications of fuel and specialty products.
- 6.2.8 Understand the history of petroleum products.
- 6.2.9 Understand the importance of chemical composition of petroleum products in the classification and identification of ignitable liquids.
- 6.2.10 Understand the relationship between carbon number(s) in homologous series and physical properties.
- 6.2.11 Describe the main chemical groups of hydrocarbons and oxygenates.
- 6.2.12 Understand the difference between petroleum products and petroleum distillates.
- 6.2.13 To understand the sources and types of ignitable liquids not derived from petroleum.
- 6.2.14 Refinery Processes
  - 6.2.14.1 Crude Oil Sources and Composition
  - 6.2.14.2 Refining Processes
  - 6.2.14.3 Relating Refinery Fractions to Commercially Available Products

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- 6.2.14.4 Distribution
- 6.2.14.5 Obtaining Specific Product Information
- 6.2.15 Petroleum Products
  - 6.2.15.1 Alkanes
  - 6.2.15.2 Aromatics
  - 6.2.15.3 Cycloalkanes
  - 6.2.15.4 Alkenes
  - 6.2.15.5 Alkynes
  - 6.2.15.6 Indanes/Indenes
  - 6.2.15.7 Oxygenates
  - 6.2.15.8 Light, Medium and Heavy Petroleum Products
  - 6.2.15.9 Miscellaneous Products
- 6.2.16 Non-Petroleum Products
  - 6.2.16.1 Sources of non-petroleum ignitable liquids
  - 6.2.16.2 Uses of non-petroleum ignitable liquids
  - 6.2.16.3 Considerations affecting analysis
- 6.2.17 Classification of Ignitable Liquids
  - 6.2.17.1 Gasoline
  - 6.2.17.2 Petroleum distillates
  - 6.2.17.3 Isopariffinic products
  - 6.2.17.4 Aromatic products
  - 6.2.17.5 Naphthenic paraffinic products
  - 6.2.17.6 Normal alkane products
  - 6.2.17.7 Oxygenated solvents
  - 6.2.17.8 Miscellaneous
- 6.3 Health and Safety Hazards

NA

- 6.4 Reading and Practical Exercises
  - 6.4.1 Gasoline and Other Motor Fuels. Encyclopedia of Chemistry and Technology. Vol. 10. 2nd Ed. 1982: 463-498.
  - 6.4.2 Petroleum (Refinery Process, Survey), Kirk/Othmer Encyclopedia of Chemical Technology. Vol. 17. Third Edition. John Wiley & Sons, Inc. 1982:183-256.
  - 6.4.3 Thornton, J.I., Fukiyama, B. The Implications of Refining Operations to the Characterization and Analysis of Arson Accelerants. Arson Analysis Newsletter. Part I. Physical Separation, pages 1-16, May 1979; Part II. Chemical Conversions, Treating Processes, and Subsidiary Processes, pages 1-16, August 1979.

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- 6.4.4 Petroleum (Refinery Process, Survey), Kirk/Othmer Encyclopedia of Chemical Technology. John Wiley & Sons, Inc. 1974.
- 6.4.5 Trimpe, M.A. Turpentine in Arson Analysis. Journal of Forensic Sciences. 1991;36: 1059-1073.
- 6.4.6 Vaerenbergh, G.V. An Unusual Arson Case: Polymer Grains, A Mineral Spirit, Solid Bricks Containing Kerosene, and a Glycoether Used as Fuels. Forensic Science Communications. 2002; 4.
- 6.4.7 Current version of ASTM E1618
- 6.4.8 Newman R, Gilbert M, Lothridge K. GC-MS Guide to Ignitable Liquids. New York: CRC Press; 1998.
- 6.4.9 Submit definitions of the following terms to the trainer.
  - 6.4.9.1 Petroleum Product Refining From Crude Oil
  - 6.4.9.2 Petroleum Tower Distillation
  - 6.4.9.3 Petroleum Catalytic Cracking
  - 6.4.9.4 Petroleum Catalytic Reforming
  - 6.4.9.5 "Gasoline" (Both as a created product and as a product classification)
  - 6.4.9.6 Distillate
  - 6.4.9.7 Isoparaffinic Products
  - 6.4.9.8 Aromatic Products
  - 6.4.9.9 Naphthenic-Paraffinic Products
  - 6.4.9.10 Normal Alkane Products
  - 6.4.9.11 De-aromatized Distillates
  - 6.4.9.12 Oxygenated Solvents
  - 6.4.9.13 "Miscellaneous" Products
  - 6.4.9.14 Gum Turpentine
  - 6.4.9.15 Oil (Distilled) Turpentine
  - 6.4.9.16 Classifications of Ignitable Liquids
- 6.4.10 Answer these key points and submit to the trainer:
  - 6.4.10.1 Discuss the cracking and reforming processes.
  - 6.4.10.2 What is the purpose of desalting?
  - 6.4.10.3 What is the purpose of alkylation?
  - 6.4.10.4 List 5 refinery processes used to physically separate crude oil. Briefly describe how the processes accomplish separation.
  - 6.4.10.5 List the 6 primary straight run products which result from fractional distillation of crude oil. For 3 of these, list 3 common commercially available products.
  - 6.4.10.6 Explain octane numbers and knocking.

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- 6.4.10.7 List 5 major compounds in gasoline.
- 6.4.10.8 Discuss petroleum production from crude oil to products.
- 6.4.10.9 Discuss the uses of petroleum products based on their characteristics and properties.
- 6.4.10.10 Discuss the relative boiling temperatures of n-hexane, 3-methylpentane and cyclohexane. What is the major consideration?
- 6.4.10.11 Draw the basic structure for alkanes, alkenes, alcohols, ketones, aromatics and aldehydes.
- 6.4.10.12 Discuss the origin of terpenes. What are some commonly encountered terpenes?

### 6.4.11 PRACTICAL EXERCISES

Obtain, analyze and classify at least a ten new liquid reference samples for the in-house library.

6.4.12 A written examination must be successfully completed before proceeding to the next module.



# 7.0 Fire Evidence Handling

# 7.1 Background and Theory

As with all forensic samples, data from the analysis of a sample is only as good as the collection, handling, and storage of the samples. If correct procedures are not followed then the data may be worthless.

- 7.2 Objectives, Principles, and Knowledge
  - 7.2.1 Demonstrate an understanding of evidence collection.
  - 7.2.2 Demonstrate knowledge of correct documentation and packaging of evidence.
  - 7.2.3 Demonstrate correct evidence preservation techniques.
  - 7.2.4 Demonstrate correct procedures to establish valid chain of custody.
  - 7.2.5 Demonstrate an understanding of the need for comparison samples in fire debris cases.
  - 7.2.6 Demonstrate the best sources for comparison samples in specific situations.
  - 7.2.7 Demonstrate an understanding of absorbent materials effective for collecting ignitable liquid residues from non-removable, porous matrices.
  - 7.2.8 Review all components of the ILIMS Fire analysis worksheet
  - 7.2.9 Preservation of evidence
    - 7.2.9.1 Types of packaging
    - 7.2.9.2 Lined vs. Unlined Cans
    - 7.2.9.3 Glass jars
    - 7.2.9.4 Nylon, polyethylene or other vapor-tight fire debris bag
    - 7.2.9.5 Paper or zip-lock type plastic bags
    - 7.2.9.6 Refrigeration
    - 7.2.9.7 Freezing
    - 7.2.9.8 Protection
      - 7.2.9.8.1 Sunlight
      - 7.2.9.8.2 Heat
      - 7.2.9.8.3 Breakage (glass containers)
    - 7.2.9.9 Time
      - 7.2.9.9.1 Shelf Life
      - 7.2.9.9.2 Visual Inspection
    - 7.2.9.10 Chain of Custody
      - 7.2.9.10.1 Intact
      - 7.2.9.10.2 Legible
      - 7.2.9.10.3 Complete
      - 7.2.9.10.4 Documented transfers
  - 7.2.10 Comparison Samples

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- 7.2.10.1 Pyrolysis products
- 7.2.10.2 Oxidative combustion
- 7.2.10.3 Reductive decomposition
- 7.2.10.4 Petroleum background
- 7.2.10.5 Burned versus unburned samples
- 7.2.11 Sampling materials
  - 7.2.11.1 Recovering ignitable liquid residues from non-collectible, porous surfaces
  - 7.2.11.2 Non-clumping, non-scented kitty litter
  - 7.2.11.3 Other absorbent materials
- 7.3 Health and Safety Hazards
  - 7.3.1 Properly handling of ignitable liquids and samples must be observed.
- 7.4 Reading and Practical Exercises
  - 7.4.1 DeHaan, J.D. Pyrolysis Products of Structure Fires. Journal of the Forensic Science Society. 1988; 28: 299-309.
  - 7.4.2 DeHaan, J.D., Skalsky, F.A. Evaluation of Kapak Plastic Pouches. Arson Analysis Newsletter. 1981; 5; 6-11.
  - 7.4.3 Additional articles at I:fire/training
  - 7.4.4 Submit definitions of the following terms to the trainer.
    - 7.4.4.1 Absorbent
    - 7.4.4.2 Chain of Custody
    - 7.4.4.3 Comparison Sample
    - 7.4.4.4 Desorption
    - 7.4.4.5 Sample Matrix
  - 7.4.5 Answer these key points and submit to the trainer:
    - 7.4.5.1 Methods for Storing and Transporting Fire Debris Samples Pros/Cons
    - 7.4.5.2 Why should comparison samples be collected?
    - 7.4.5.3 What collection methods should be employed when it is not practical or possible to remove the sample matrix from the scene?
    - 7.4.5.4 Where should samples be taken from if there is a pour pattern?
    - 7.4.5.5 Is carpet or concrete a better sample? Why?
    - 7.4.5.6 How should liquid samples be packaged to be shipped and how can they be shipped?

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#### 7.4.6 PRACTICAL EXERCISES

Goals of the exercise: To collect ignitable liquid samples from non-removable, porous matrices utilizing numerous absorbent materials. Procedure: Obtain the non-scented, non-clumping kitty litter, non-self rising flour, and sand. Prepare a test mixture of gasoline and diesel fuel in approximately a 1:1 ratio. Place several milliliters of the test mixture in four different areas on concrete, asphalt, and tile surfaces. Cover each sample area with one of the absorbents for approximately one hour. Prepare a comparison sample for each absorbent on each type of surface. Collect the test mixture samples and the comparison sample in appropriate evidence containers. Prepare a set of control samples by placing each of the absorbent materials in appropriate evidence containers. The samples will be evaluated as part of the passive adsorption -elution exercise in Section 8.

7.4.7 A written examination must be successfully completed before proceeding to the next module.



# 8.0 Methods for the Recovery of Ignitable Liquids and Ignition Testing

- 8.1 Background and Theory
  - 8.1.1 There are several methods of collecting volatile liquids from samples. The various pros and cons will be explored as well as procedures to minimize the chance of contamination.
  - 8.1.2 Ignition testing of liquids can reveal useful information about the nature of the sample.
- 8.2 Objectives, Principles, and Knowledge
  - 8.2.1 Preliminary Examinations
    - 8.2.1.1 Visual
      - 8.2.1.1.1 Evidence inventory
      - 8.2.1.1.2 Evaluate to determine which recovery procedure to use
      - 8.2.1.1.3 Check for the presence of incendiary devices or other evidence present
      - 8.2.1.1.4 May only be able to only do a cursory visual examination due to the risks of prolonged exposure to the air
    - 8.2.1.2 Olfactory
      - 8.2.1.2.1 Always work in well ventilated area.
      - 8.2.1.2.2 Waft contents to determine if there is a strong ignitable liquid odor.
      - 8.2.1.2.3 Odor of strong ignitable liquids may influence recovery method choice.
    - 8.2.1.3 Procedure
      - 8.2.1.3.1 Note the condition of the container and whether or not it has been properly sealed. Note any damage which may have compromised the integrity of the container.
      - 8.2.1.3.2 Open the exhibit container and carefully waft vapors toward the nose while checking for any obvious ignitable liquid odor.

        Smelling the item is not recommended if moldy or if it is a biohazard.
      - 8.2.1.3.3 Visually examine the item and note its contents (type of material or debris present) and condition (burned, partially burnt, burnt, etc.).

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|            | devices. If found, such materials may be subjected to additional examinations.   |
|------------|--|
| 8.2.1.3    |  |
| 8.2.1.3    | , and the second |
| 8.2.1.3    | .7 If necessary, transfer the contents of the exhibit to a container suitable for the type of sampling method which will be used.  |
| 8.2.2 Head | Ispace   |
| 8.2.2.1    | Room temperature and heated  |
| 8.2.2.2    | Equipment needed   |
| 8.2.2.3    | Advantages   |
| 8.2.2.4    | Disadvantages  |
| 8.2.2.5    | Reference ASTM E 1388  |
| 8.2.2.6    | Define the term vapor pressure and discuss any effect temperature,   |
|            | volume of liquid, and space above the liquid, has on vapor   |
|            | pressure?  |
| 8.2.2.7    | Discuss how and why the headspace sampling temperature and   |
|            | sampling volume should be optimized.   |
|            |  |
| 0 2 2 Dagg | ive Adsorption-Elution (PAE)   |
|            | Equipment needed   |
|            | Adsorption considerations  |
| 8.2.3.2    |  |
| 8.2.3.2    |  |
| 8.2.3.2    |  |
| 8.2.3.2    |  |
|            | .5 Re-extraction of the sample vs. saving original adsorbent   |
|            | Desorption considerations  |
| 8.2.3.3    |  |
| 8.2.3.3    |  |
|            | Advantages   |
|            | Disadvantages  |
|            | Reference ASTM E 1412  |
| 0.2.3.0    | Reference Hotel L 1112   |

If requested, examine for any evidence of incendiary materials or

# 8.2.4 Solvent Extraction

8.2.1.3.4

8.2.4.1 Equipment needed

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| 8.2.4.2 | Advantages |
|---------|------------|
|---------|------------|

- 8.2.4.3 Disadvantages
- 8.2.4.4 Reference ASTM E 1386

#### 8.2.5 Solvent Wash

- 8.2.5.1 Used with non-porous material
- 8.2.5.2 Best when visible liquid droplets can be seen
- 8.2.5.3 Same blanking procedure used in solvent extractions
- 8.2.5.4 Advantages
- 8.2.5.5 Disadvantages

#### 8.2.6 Solvent Dilution

Liquid samples are dissolved in an appropriate amount of solvent.

### 8.2.7 Ignition Testing

- 8.2.7.1 Equipment needed
- 8.2.7.2 Good practice to determine if a liquid is ignitable
- 8.2.7.3 Advantages
- 8.2.7.4 Safety Considerations
- 8.2.7.5 Refer to ignition testing exercise.

# 8.3 Health and Safety Hazards

- 8.3.1 Personal protective equipment should be worn at all times
- 8.3.2 Gloves must be changed in between each sample.
- 8.3.3 The analyst must have a thorough understanding of necessary safety measures to protect against the hazards associated with the use of carbon disulfide and pentane.

### 8.4 Reading and Practical Exercises

- 8.4.1 Andrasko, J. The Collection and Detection of Ignitable Liquid Vapors Using Porous Polymers and Curie Point Pyrolysis Wire Coated with Activated Charcoal. Journal of Forensic Sciences. 1983; 28: 330-334.
- 8.4.2 ASTM E 1386, Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction, Current Version
- 8.4.3 Buckleton, J.S., Bettany, B.L., Walsh, K.A.J. A Problem of Hydrocarbon Profile Modification by

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- 8.4.4 Dietz, W.R. Improved Charcoal Packaging for Ignitable Liquid Recovery by Passive Diffusion. Journal of Forensic Sciences. 1991; 36:111-121.
- 8.4.5 Ettling, C.V. Determination of Hydrocarbons in Fire Remains. Journal of Forensic Sciences. 1963; 8: 261-267.
- 8.4.6 Higgins, K.M. High Speed Extraction of Accelerants from Arson Debris. Journal of Forensic Sciences. 1984; 29: 874-880.
- 8.4.7 Juhala, J. A Method for Adsorption of Flammable Vapors by Direct Insertion of Activated Charcoal into the Debris Samples. Arson Analysis Newsletter, 1982; 6: 32-40.
- 8.4.8 Kubler, D. The Isolation of Accelerants by Headspace Sampling and by Steam Distillation. Arson Analysis Newsletter. 1981; 5: 64-79.
- 8.4.9 Lentini, J.J., Armstrong, A.T. Comparison of the Eluting Efficiency of Carbon Disulfide with Diethyl Ether: The Case for Laboratory Safety. Journal of Forensic Sciences. 1997; 42: 307-311.
- 8.4.10 Newman, R.T., Dietz, W.R., Lothridge, M.S.M. The Use of Activated Charcoal Strips for Fire Debris Extractions by Passive Diffusion. Part 1: The Effects of Time, Temperature, Strip Size, and Sample Concentration. Journal of Forensic Sciences. 1996; 41: 361-370.
- 8.4.11 Phelps, J.L Chasteen, C.E., Render, M.M. Extraction and Analysis of Low Molecular Weight Alcohols and Acetone from Fire Debris Using Passive Headspace Concentration. Journal of Forensic Sciences.1994; 39:194-206.
- 8.4.12 Stauffer, E., Dolan, J., Newman, R., Fire Debris Analysis. Academic Press; 2008.
- 8.4.13 Waters, L., Palmer, L. Multiple Analysis of Fire Debris Samples Using Passive Headspace Concentration. Journal of Forensic Sciences. 1993; 38: 165-183.
- 8.4.14 Material safety data sheets (MSDS) for carbon disulfide and pentane.
- 8.4.15 ISPFS Fire Debris Analytical Method
- 8.4.16 Submit definitions of the following terms to the trainer.
  - 8.4.16.1 Adsorption
  - 8.4.16.2 Displacement
  - 8.4.16.3 Elution
  - 8.4.16.4 Headspace Sampling
  - 8.4.16.5 Ignition Testing
  - 8.4.16.6 Passive Adsorption-Elution
  - 8.4.16.7 Solvent Extraction
  - 8.4.16.8 Solvent Wash

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- 8.4.17 Answer these key points and submit to the trainer:
  - 8.4.17.1 Discuss the usage of charcoal adsorption-elution, headspace, solvent extraction, and solvent wash. What type of samples work best with each of the four extraction types? What are the advantages and disadvantages of each of the four extraction types?
  - 8.4.17.2 How would heating temperature affect the chromatographic data in headspace and charcoal adsorption-elution techniques?
  - 8.4.17.3 How does sample concentration affect the chromatographic data in headspace and charcoal adsorption-elution techniques?
  - 8.4.17.4 What other adsorbents can be used to trap ignitable liquid residues in adsorption-elution techniques?
  - 8.4.17.5 Is charcoal a good adsorbent for alcohol? For hydrocarbons?
  - 8.4.17.6 What are the two basic types of desorption? Which is used for active charcoal and why?
  - 8.4.17.7 What solvents can be used for ignitable liquid solvent extractions?
  - 8.4.17.8 Discuss the factors that can lead to distorted recovery (discuss both skewing toward the light ends as well as toward the heavy ends) and how these factors can be minimized.
  - 8.4.17.9 What additional steps must be added to the standard passive headspace concentration extraction for petroleum products if detection of alcohols and/or low molecular weight oxygenated solvents is desired?
  - 8.4.17.10 Can kerosene and fuel oil #2/diesel fuel-type products be differentiated when passive headspace concentrations is the method of extraction? Explain.
  - 8.4.17.11 Under what conditions is solvent extraction preferred over adsorption-elution extractions?
  - 8.4.17.12 How can you determine if a liquid sample is aqueous or non-aqueous?
  - 8.4.17.13 When and why would ignition testing be utilized?
  - 8.4.17.14 What parameters affect the PAE recovery process?

# 8.4.18 PRACTICAL EXERCISES

- 8.4.18.1 Heated Headspace/Passive Adsorption-Elution
  - 8.4.18.1.1 Goals of the Exercise: The student will evaluate a series of standard ignitable liquids on simple background matrices using the heated headspace injection and passive adsorption/elution methods. Differences in detection vs. concentration and ignitable liquid type will be reviewed.

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- 8.4.18.1.2 Procedure: You will receive a series of ignitable liquid standards in paint cans. Each of the two cans in a set will have the same ignitable liquid standard at about the same concentration level. Coleman fuel, automotive gasoline, charcoal lighter fluid, kerosene, and diesel fuel at various concentrations will be used. One can is marked as to its contents and "HHS", and the other "PAE".
- 8.4.18.1.3 Before evaluating any samples, the standard reference ignitable liquid component mixture is evaluated using the fire debris program.
- 8.4.18.1.4 Using the "PAE" marked cans and while wearing gloves (changing them between samples to prevent cross contamination), suspend a charcoal strip on an ornament hook. The other end of the hook is placed into the seal groove of the can and the lid is securely fastened using the mallet. (Note that there are many ways to place charcoal in a sample container-some use alligator clamps holding the strip inside the can and magnets outside, others suspend the charcoal strip on pre-tested thread, and some use loose charcoal in a glass dish placed directly onto the sample) Use any method you are comfortable with.
- 8.4.18.1.5 Cans are heated in an oven up to 90 degrees C for a minimum of 2 hours. During the last 30 minutes, the syringe used for heated headspace analysis is placed in the oven as well.
- To process a Heated Headspace or "HHS" sample: First a hole is 8.4.18.1.6 placed in the top of the can and the hole is sealed with aluminum tape. The GC/MSD program for hand injection/fire debris evaluation is loaded on the instrument, allowing for enough time for the instrument to stabilize at the initial temperature while the "HHS" sample is heating. (Alternatively the autosampler program can be used to create a sample table, but the method must be the one that requires hand injection) The autoinjector tower is removed from the instrument. When ready to inject, remove the can from the oven.. While wearing gloves, the hot syringe is removed from the oven, the needle placed through the tape into the small hole, and a 0.5 cc sample of hot vapor removed from the sample (the hole can be plugged using a rubber stopper or tape after the sample is removed). The sample is quickly injected in the GC/MSD and the "Start" button depressed on the face of the GC/MSD control panel to start the

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run. The syringe is then "rinsed" with air and then placed back in the oven for use with the next sample. A "room air" blank is evaluated before injecting samples, and in some laboratories between each sample to show that the gas syringe is clean between uses. For these samples, speed is of the essence. The longer the time before injection, the more the sample can condense in the can or in the syringe. The syringe should be checked between uses to insure septa material has not plugged the needle. The runs should be evaluated splitless, or as close to splitless as possible.

8.4.18.1.7

To process a "Passive Adsorption Elution or "PAE" sample: The fire debris program using the autoinjector system is used for these evaluations. Remove the can from the oven and allow to cool to room temperature. Prepare two vials for the autoinjector using a 0.5 ml insert. The second vial is rinsed with CS2 and the rinse placed in the first vial to form a blank. The first vial is capped. While wearing gloves the can is opened in the hood and the charcoal strip removed. The charcoal strip is removed from the hook and inserted in the empty rinsed injection vial and CS2 added to cover. The vial is then sealed and allowed to stand for about 15 minutes (which is the normal time it takes for the system to ramp down and stabilize at the starting temperature of 35 degrees). The autoinjector system is then used to inject the sample for evaluation, first by injecting the solvent blank, and then the sample. (In case work, a "Preparation Blank" is created using a clean charcoal strip processed with either a stock blank can/packaging or packaging provided by the submitting agency. The strip is processed in the same manner as the samples.

8.4.18.1.8 This sample is placed in a clean lined injection vial and the CS2 to be used in the extraction steps for samples is added. This Preparation Blank is then evaluated to show that the common charcoal stock and solvent to be used are free of contamination. The normal order for injection would be the Calibration mix, followed by the Preparation Blank, then followed by the sample blank and sample until each of the blank and sample pairs have been evaluated.) Heated Headspace samples should be evaluated in succession, as they each require hand injection, and the PAE evaluated using the autoinjection system overnight.

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- 8.4.19 Data Evaluation: For each of the HHS and PAE pairs, evaluate the chromatograms and mass spectral data produced by each general class and concentration level. Using the data answer the following questions:
  - 8.4.19.1.1 As the concentration decreases, does one of the methods produce better results?
  - 8.4.19.1.2 As the samples begin to contain larger molecules, does one of the methods produce better results?
  - 8.4.19.1.3 What are the limitations of each method as samples contain larger molecules? Is there a practical limit to what can be "seen" in fire debris analysis?
  - 8.4.19.1.4 Based on the data, which method would you choose to use and when? What are the advantages and disadvantages of each way of proceeding?
  - 8.4.19.2 Solvent Extraction/Solvent Washes
    - 8.4.19.2.1 Goals of the Exercise: The student will evaluate samples requiring solvent extraction and/or solvent washes to concentrate ignitable liquid residues for analysis.
    - 8.4.19.2.2 Procedure: You will receive a set of three samples for evaluation. One will be marked "SE" for Solvent Extraction, one marked "PAE", and the third marked "SW" for Solvent Wash.
    - 8.4.19.2.3 Before evaluating any samples, the standard reference ignitable liquid component mixture is evaluated using the fire debris program.
    - 8.4.19.2.4 Open the "PAE" can and determine the size, type, and characteristics of the sample. Observe any odor present, and record your findings. This sample is evaluated using the charcoal strip technique used in the first practical exercise in this module. The data obtained will be used for later comparison with the "SE" sample.
    - 8.4.19.2.5 Open the "SE" can and determine the size, type, and characteristics of the sample. Observe any odor present, and record your findings.
    - 8.4.19.2.6 Obtain a clean beaker of sufficient size to contain the sample, and a second beaker for evaporation. In a fume hood, thoroughly rinse the second beaker with an amount of reagent grade pentane you estimate is sufficient to cover the debris sample you will be evaluating. Pour this pentane into the evaporation

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- beaker. Carefully evaporate the pentane down to about 1 ml and place this "blank" sample into an injection vial.
- 8.4.19.2.7 Place the sample into the first previously evaluated beaker and add enough pentane to cover and soak the sample. Allow this to stand for long enough to soak the sample and extract ignitable liquid traces. Carefully pour off the solvent into the evaporation beaker, trying to restrict the amount of particulate material being transferred. Evaporate the pentane down to about 1-2 ml for evaluation. In actual casework, the appearance and viscosity of the extract may suggest halting evaporation at a larger volume. Filtration using glass wool in a clean disposable pipette may be necessary to remove particulate material from a concentrated extract.
- 8.4.19.2.8 The sample is then placed into an autoinjection vial and the blank and sample are evaluated using the fire debris program.
- 8.4.19.2.9 Note that while pentane is usually used, both carbon disulfide and methylene chloride have been used in this procedure.

  Carbon disulfide is avoided for considerations of health and environmental factors, and methylene chloride has not proved as successful for extraction.
- 8.4.19.2.10 For the "SW" can: open the can and determine the size, type, and characteristics of the sample. Observe any odor present, and record your findings.
- 8.4.19.2.11 Solvent washes are used when a sample (usually glass fragments from a suspected incendiary "Molotov Cocktail" type device, or other non-porous surface) is recovered with visible drops of liquid present.
- 8.4.19.2.12 A blank is created with the pentane to be used in the rinse, and the injection vials to be used. Solvent washes may require the use of small beakers or large test tubes (blanked using a procedure similar to that for the solvent extraction) to concentrate the wash for evaluation.
- 8.4.19.2.13 Pieces to be rinsed are removed with forceps and washed with small amounts of pentane into the holding beaker or test tube to remove visible drops, until the entire sample has been appropriately rinsed. The sample is concentrated if necessary and evaluated using the fire debris program.
- 8.4.19.3 Evaluation: Using the data obtained, answer the following questions-8.4.19.3.1 How well did the solvent extraction isolate the ignitable liquid residue?

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- 8.4.19.3.2 How much were the SE results affected by background factors?
- 8.4.19.3.3 Is solvent extraction preferable to Passive Adsorption-Elution? When might you use SE?
- 8.4.19.3.4 How well did the solvent wash isolate the ignitable liquid residue?

# 8.4.19.4 Ignition Testing

- 8.4.19.4.1 Goals of the Exercise: The student will safely carry out ignition testing on a series of ignitable liquid standards and unknown materials.
- 8.4.19.4.2 Procedure: The procedure must be carried out in a hood containing no other flame sources. A few drops of each ignitable liquid standard to be tested are placed, one at a time, on a piece of glass wool on a clean watch glass. A lit match or other flame source is moved slowly toward the liquid and the point at which ignition (if any) occurs is noted. Note also the color of the flame and the type of smoke generated, if they are visible. After the liquid is consumed, note the type and color of any residue present.. If a light oxygenated solvent is suspected, the glass wool can be omitted and the flame test done on a few drops of the liquid.
- 8.4.19.4.3 For some liquids, such as diesel type fuels, will not ignite even when the match is placed physically in the liquid, but will "wick up" the match and sustain a flame. An alternative method for flame testing involves using a small amount of glass wool on a watch glass and placing the liquid into that for the testing procedure.
- 8.4.19.4.4 For the unknowns, perform either of the testing procedures as listed above, and attempt to classify the unknowns by comparing them to the results for the ignitable liquid standards tested.
- 8.4.19.5 Evaluating Absorbent Materials Used to Collect Ignitable Liquid Residues
  - 8.4.19.5.1 Goals of the exercise: To determine which absorbent material used in Section 3, Practical Exercise A is most effective in the recovery of ignitable liquid residues.
  - 8.4.19.5.2 Procedure: Using PAE, extract the questioned samples, comparison samples and control samples previously collected in Section 3, Practical Exercise A.
  - 8.4.19.5.3 Evaluation: Using data obtained, answer the following questions:

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| 8.4.19.5.4 | Which absorbent materials had the most background           |
|------------|---|
|            | interference?   |
| 8.4.19.5.5 | Which matrices had the most interference?                   |
| 8.4.19.5.6 | Which absorbent material recovered the gasoline-diesel mix  |
|            | most effectively?   |
| 8.4.19.5.7 | Were gasoline and diesel equally recovered? Why or why not? |

8.4.20 A written examination must be successfully completed before proceeding to the next module.



# 9.0 Gas Chromatography/ Mass Spectrometry

# 9.1 Background and Theory

The GC/MS is the instrument used to analyze samples for the presence of ignitable liquids. Before an analyst can work case samples they must demonstrate the understanding of both the theory behind and the operation of both the Gas Chromatograph and the Mass Spectrometer.

- 9.1.1 Describe selected ion monitoring and how extracted ions are selected.
- 9.1.2 Demonstrate how to properly interpret mass spectral data.
- 9.1.3 To develop an understanding of in-house methods of obtaining and cataloging ignitable liquid comparison samples.
- 9.1.4 To compile and classify a wide variety of commercially available ignitable liquid products.

### 9.2 Objectives, Principles, and Knowledge

- 9.2.1 Theoretical aspects of GC
  - 9.2.1.1 History of chromatography
  - 9.2.1.2 Introduction to the various chromatographic methods
  - 9.2.1.3 Gas/liquid phase equilibrium
  - 9.2.1.4 Van Deemter curves
  - 9.2.1.5 Cross contamination
  - 9.2.1.6 Temperature vs. retention behavior
- 9.2.2 Chromatographic columns
  - 9.2.2.1 Polar/non-polar
  - 9.2.2.2 Column efficiency
  - 9.2.2.3 Resolution
- 9.2.3 Carrier Gas
  - 9.2.3.1 Gas selection
  - 9.2.3.2 Flow rate
  - 9.2.3.3 Troubleshooting
- 9.2.4 GC data interpretation

Peak pattern comparison (with standards)

- 9.2.5 Mass Spectrometer
  - 9.2.5.1 Components
  - 9.2.5.2 Vacuum systems
  - 9.2.5.3 GC/MS interfaces
  - 9.2.5.4 Electron impact ionization

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# 9.2.5.5 Detection/ion abundance determination.

| 9.2.6 Basi | c Interpretation of Mass Spectral Data                             |
|------------|--|
| 9.2.6.1    | TIC  |
| 9.2.6.2    | Molecular ions   |
| 9.2.6.3    | Base peaks   |
| 9.2.6.4    | Nitrogen rule  |
| 9.2.6.5    | Isotopic ratios  |
| 9.2.6.6    | Fragmentation  |
| 9.2.6.7    | Libraries  |
| 9.2.6.8    | Demonstrate the ability to execute macro programs used to generate |
|            | extracted ion profiles and rescaling of total ion chromatograms.   |
| 9.2.6.9    | Extracted Ion Chromatograms  |
| 9.2.6.10   | Selected Ion Monitoring  |
| 9.2.6.11   | Comparison to standards and references                             |

### 9.2.7 Chemical structure review

- 9.2.7.1 alkanes
- 9.2.7.2 alkenes
- 9.2.7.3 aromatics
- 9.2.7.4 naphthalenes
- 9.2.7.5 polynuclear aromatics
- 9.2.7.6 indanes and indenes
- 9.2.7.7 styrenes
- 9.2.7.8 terpenes

# 9.2.8 Sample Matrix Effects

- 9.2.8.1 "filtering" out interfering compounds
- 9.2.8.2 Microbial degradation in soil
- 9.2.8.3 Pyrolysis of polyethylene and other plastics
- 9.2.8.4 Wood thermal degradation

# 9.3 Health and Safety Hazards

NA

### 9.4 Reading and Practical Exercises

9.4.1 Analytical Gas Chromatography, Jennings

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- 9.4.2 Basic Gas Chromatography (Techniques in Analytical Chemistry), McNair & Miller.
- 9.4.3 Gas Chromatography in Forensic Science (Ellis Horwood Series in Forensic Science), I. Tebbett (Editor).
- 9.4.4 High Resolution Gas Chromatography, Hyver & Sandra (HP)
- 9.4.5 Modern Practice of Gas Chromatography, edited by Robert L. Grob 3rd Ed. John Wiley & Sons, Inc. 1995.
- 9.4.6 Armstrong, A.T., Wittkower, R.S., Identification of Accelerants in Fire Residues by Capillary Column Gas Chromatography, Journal of Forensic Sciences Vol. 23 (4), pages 662-671, 1978
- 9.4.7 ASTM E 1618, Standard Guide for Identification of Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry, Current Version.
- 9.4.8 Bertsch, W., Sellers, C.S., Babin, K., Analysis of Suspect Arson Samples by GC and GC-MS, LC-GC, Vol. 6 (11), pages 1000-1014, 1988.
- 9.4.9 Holzer, C., Bertsch, W., Recent Advances Toward the Detection of Accelerants in Arson Cases, American Laboratory, pages 15-19, Dec. 1988.
- 9.4.10 Kelly, R., Martz, R., Accelerant Identification in Fire Debris by Gas Chromatography/Mass Spectrometry Techniques, Journal of Forensic Sciences, Vol. 29 (3), pages 714-727, 1984.
- 9.4.11 Loscalzo, P.J., DeForest, P.R., Chao, J.M., A Study to Determine the Limit of Detectability of Gasoline Vapor from Simulated Arson Residues, Journal of Forensic Sciences, Vol. 25, (1), pages 162-167, 1980.
- 9.4.12 Manufacturer's manuals
- 9.4.13 McLafferty and Turececk, Interpretation of Mass Spectra.
- 9.4.14 Midkiff, C.R., Jr., Washington, W.D., Gas Chromatographic Determination of Traces of Ignitable Liquids in Physica1 Evidence, Journal of the Association of Official Analytical Chemists, Vol. 55 (4), pages 840-845, 1972.
- 9.4.15 Smith, R.M., Mass Chromatograpic Analysis of Arson Accelerants, Journal of Forensic Sciences, Vol. 28 (2), pages 318-329, 1983.
- 9.4.16 Smith, R.M., Arson Analysis by Mass Chromatography, Analytical Chemistry, Vol. 54 (13), pages 1399A-1409A, 1982.
- 9.4.17 Smith, R.M. "Arson Analysis by Mass Spectrometry", Forensic Mass Spectrometry, edited by Yinon, Chapter 5.
- 9.4.18 Tontarski, R., Strobel, R., Automated Sampling and Computer Assisted Identification of Hydrocarbon Accelerants, Journal of Forensic Sciences ,Vol. 27 (2), pages 710-714, 1982.
- 9.4.19 Watson, Introduction to Mass Spectrometry
- 9.4.20 National Center for Forensic Science's Ignitable Liquid Reference Collection http://www.ncfs.org/ilrc1.html

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- 9.4.21 Newman, R., Gilbert, M., Lothridge, K., GC-MS Guide to Ignitable Liquids, CRC Press, 1998.
- 9.4.22 Answer these key points and submit to the trainer:
  - 9.4.22.1 What is the TIC?
  - 9.4.22.2 What is ion profiling?
  - 9.4.22.3 Understand how the arson macro sorts and presents data.
  - 9.4.22.4 Describe the difference between ion profiling and selected ion monitoring.
- 9.4.23 Practical Exercises
  - 9.4.23.1 Complete the training as outline in the GC/MSD portion of the Controlled Substances training manual.
  - 9.4.23.2 Analyze a set of unknown samples using the preparation methods as described in Section 5.
  - 9.4.23.3 Analyze reference samples using dilution, heated-headspace, and PAE.
- 9.4.24 A written examination must be successfully completed before proceeding to the next module.



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# 10.0 Classification, Data Interpretation, Matrix Interferences of Ignitable Liquids

# 10.1 Background and Theory

The difficulty in analyzing samples for the presence of ignitable liquids is not in the preparation of the instrument run but rather in the interpretation of the data. There are many factors that can affect the results, low concentration, matrix effects, sample degradation etc. All of these must be accounted for.

# 10.2 Objectives, Principles, and Knowledge

- 10.2.1 To familiarize the student with the data generated by the recovery methods and the difficulties in interpreting the meaning of the data generated from complex fire environments.
- 10.2.2 To work with situations involving determinations of a possible common source for two ignitable liquids, as well as determination of whether a liquid can be classed as "ignitable".
- 10.2.3 Using the ASTM 1618 classification system including carbon ranges.
- 10.2.4 Data Analysis: GC/MS
  - 10.2.4.1 Compound identification
  - 10.2.4.2 Visual comparison (TIC)
  - 10.2.4.3 Extracted ion chromatography (EIC)
  - 10.2.4.4 Target compound chromatography (TCC)
- 10.2.5 Identification of Altered Ignitable Liquids
  - 10.2.5.1 Evaporation
  - 10.2.5.2 Microbial degradation
  - 10.2.5.3 Vapor transfer
  - 10.2.5.4 Sampling technique effects
- 10.2.6 Interference from Substrate Materials
  - 10.2.6.1 Carpet and carpet padding
  - 10.2.6.2 Wood and plant products
  - 10.2.6.3 Paper products
  - 10.2.6.4 Shoes and clothing
  - 10.2.6.5 Condensates
  - 10.2.6.6 Vehicle fires
  - 10.2.6.7 Others
- 10.2.7 Comparison of Ignitable Liquids
  - 10.2.7.1 Gasoline
  - 10.2.7.2 Aromatics in petroleum distillates

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10.2.7.4 Mixtures

# 10.3 Health and Safety Hazards

NA

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- 10.4.51 Newman, R., Gilbert, M. and Lothridge, K., GC-MS Guide to Ignitable Liquids, CRC Press: New York, 1998
- 10.4.52 Answer these key points and submit to the trainer:
  - 10.4.52.1 What are the criteria for the classification of ignitable liquids?
  - 10.4.52.2 What are the effects that evaporation will have on the appearance of ignitable liquid data?
  - 10.4.52.3 What are the effects of microbial degradation?
  - 10.4.52.4 How might different sampling methods affect the appearance of ignitable liquid data?
  - 10.4.52.5 What impact might different substrates have on the appearance of fire debris data?
  - 10.4.52.6 Can you properly identify a group of ignitable liquids in accordance with ASTM E1618?
  - 10.4.52.7 When are comparisons of ignitable liquids possible? What are the strengths and limitations of those comparisons?

## 10.4.53 Practical Exercises

These practical exercises will be completed to the satisfaction of the instructor.

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# 10.4.53.1 Data Interpretation Exercise

- 10.4.53.1.1 Goals of the Exercise: The student will evaluate and identify a series of unknown ignitable liquid data packets containing single liquids and mixtures.
- 10.4.53.1.2 Procedure: The instructor will provide the student with a data packet for each unknown containing the total ion chromatogram (TIC), extracted ion chromatographic profiles (EIC), and possible component mass spectra data. The student will attempt to determine the classification of the ignitable liquid or liquids present in the unknown using the methodology as listed in the current edition of ASTM E1618. Before proceeding, review ASTM E1618 concerning data analysis and the ignitable liquid classification scheme.

## 10.4.53.2 Interpretation:

- 10.4.53.2.1 Examine the TIC for signs of a familiar pattern when compared to known standards previously evaluated, and for direct comparison by retention time to known standard materials and test component mixtures, and answer the following questions:
- 10.4.53.2.2 What is the predominant n-alkane range (if any)?
- 10.4.53.2.3 Does the pattern appear to contain a homologous series of nalkanes?
- 10.4.53.2.4 Does the pattern appear to be that of gasoline/evaporated gasoline?
- 10.4.53.2.5 If there is a pattern present that contains multiple groups, i.e. gasoline, do the group concentration ratios make sense both within the group (e.g. C2 alkyl benzenes, C3 alkyl benzenes, etc.) and between the groups?
- 10.4.53.2.6 Does there appear to be more than one identifiable pattern present?

# 10.4.53.3 Using the EIC profiles:

- 10.4.53.3.1 What does the total n-alkane profile look like? Any obvious series present?
- 10.4.53.3.2 Examine the total aromatic profile: Does a pattern appear consistent with gasoline or the normal aromatics present from a distillate?
- 10.4.53.3.3 Is there a large alkene profile? This in pair with an n-alkane profile could indicate burned plastics or asphalt.
- 10.4.53.3.4 Does the naphthalene profile show peaks consistent with gasoline?

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- 10.4.53.3.5 Examine the indane profile as gasoline is rich in indane compounds.
- 10.4.53.3.6 A very large styrene/methylstyrene set of peaks in the same range around the C2 alkyl benzenes could mean a strong background from burned polystyrene.
- 10.4.53.3.7 The naphthenic/paraffinic profile is useful for spotting these (rare) solvents.
- 10.4.53.3.8 Using the information from both the TIC and EICs, attempt to identify the unknown.

# 10.4.54 Burned Background Matrix Study

- 10.4.54.1 Goals of the Exercise: The student will process, extract, analyze and evaluate the data from a series of common "background" items.

  Analyze at least ten new samples of materials not currently on the list.
- 10.4.54.2 Procedure: Using clean new paint cans, two sets of samples will be prepared, one without ignition, and the other to be safely ignited in a hood or outside, allowed to burn/melt, and then sealed in a can with a charcoal strip for extraction and analysis: newspaper, magazine pages, "ziplock" type plastic bags, polyethylene sheet plastic wrap, "egg crate" foam padding, carpet padding, carpet samples (nylon, polypropylene and blended materials), roof shingles, linoleum flooring, foam mattress type padding, old leather shoes, athletic type shoes, pine wood, and clothing (or anything else you can find to try out). Also run five samples of different matrixes spiked with the SAM mix and burnt.
- 10.4.54.3 Interpretation: Compare the data from the unburned material to the data from the burned/melted sample. Referring especially to the articles "Volatiles from Carpet: A Source of Frequent Misinterpretation in Arson Analysis"; "The Petroleum Laced Background"; and "Pyrolysis Products of Structure Fires", determine what types of compounds may be contributed by these background materials and how they could complicate making a determination in a fire debris case. What information would you like to have about a case before writing a report?

## 10.4.55 Microbial Degradation Study

10.4.55.1 Goals of the Exercise: The student will determine how gasoline and hydrocarbon mixtures degrade over time when exposed to common soil bacteria.

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- 10.4.55.2 Procedure: Review "Degradation of Gasoline, Barbecue Starter Fluid, and Diesel Fuel by Microbial Action in Soil", "Microbial Degradation of Gasoline in Soil", and "Microbial Degradation of Petroleum Hydrocarbons: Implications for Arson Residue Analysis". Using automotive gasoline, charcoal lighter fluid, and diesel fuel from the standards collection, and clean unlined metal paint cans with lids, prepare the following using about 250 grams of soil and a syringe or pipet capable of delivering 200 microliters of the liquid: Five cans are prepared for each of the three liquids to be evaluated, labeled "Blank", "2 Days", "4 Days", "7 Days", and "14 Days". For each can set, 200 microliters of one of the ignitable liquids is placed into the soil and the soil homogenized, except in the blank, where no ignitable liquid is added. Each can is then covered with its lid and stored at room temperature.
- 10.4.55.3 A set of extracted liquid standards is prepared by placing a piece of tissue in a clean new can and adding 200 microliters of the ignitable test liquid to it.
- 10.4.55.4 At the time specified on the can label, they are extracted with a charcoal strip and then evaluated by GC/MSD as time permits. At the end of the study, there will be data for the soil without any added ignitable liquid, data for the liquid used without the soil, and four samples that show how the presence of the soil affects the ignitable liquid.
- 10.4.55.5 Interpretation: Directly evaluate the samples sequentially to see what changes have occurred over time. Be aware that different species of bacteria will consume components selectively, and that results may not be reproducible from one soil set to another.
- 10.4.56 Ignitable Liquid Comparison Exercise
  - 10.4.56.1 Goals: To understand the limitations of ignitable liquid comparisons.
  - 10.4.56.2 Procedure: Review listed references and any new references regarding the comparison of ignitable liquids. Prepare a written report discussing the limitations of comparing ignitable liquids and under what circumstances this may be possible.
- 10.4.57 A written examination must be successfully completed before proceeding to the next module.

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# 11.0 Incendiary Devices, Unusual Evidence, and Special Situations

# 11.1 Background and Theory

On rare occasions unusual evidence including incendiary devices, not to be confused with explosives, may be submitted to the laboratory. The analyst must be able to recognize this evidence and be able to process it accordingly.

## 11.2 Objectives, Principles, and Knowledge

- 11.2.1 To familiarize the student with commonly-seen incendiary devices, such as Molotov cocktails and issues related to their analysis.
- 11.2.2 To familiarize the student with incendiary mixtures that do not involve ignitable liquids and issues related to the preservation of their residues and their analysis.
- 11.2.3 To make the student aware of situations in which other types of forensic analysis may be required in addition to ignitable liquid analysis and how to work with other scientists to prioritize analyses and best preserve the evidentiary value of submitted exhibits
- 11.2.4 Incendiary Devices
- 11.2.5 Types of Incendiary Devices
  - 11.2.5.1 Components of Incendiary Devices
    - 11.2.5.1.1 Container
    - 11.2.5.1.2 Wick
    - 11.2.5.1.3 Sealant
    - 11.2.5.1.4 Fuel
    - 11.2.5.1.5 Timer (if any)
    - 11.2.5.1.6 Igniter
  - 11.2.5.2 Non-Petroleum Incendiary Mixtures
    - 11.2.5.2.1 Pool chlorine and brake fluid
    - 11.2.5.2.2 Safety flares (fusees)
    - 11.2.5.2.3 Linseed oil
    - 11.2.5.2.4 Thermite
    - 11.2.5.2.5 Napalm
    - 11.2.5.2.6 Others
- 11.2.6 Unusual Evidence
  - 11.2.6.1 Non-gasoline evaporated residues
  - 11.2.6.2 Non-gasoline microbially degraded residues
  - 11.2.6.3 Inhalants
  - 11.2.6.4 Sprays

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- 11.2.6.5 Others
- 11.2.7 Some Other Potential Analyses
  - 11.2.7.1 Explosives
  - 11.2.7.2 Other chemical analyses
    - 11.2.7.2.1 Vegetable oils
    - 11.2.7.2.2 Pepper spray et al.
    - 11.2.7.2.3 Strong acids
    - 11.2.7.2.4 General chemical analysis
  - 11.2.7.3 Latent prints
  - 11.2.7.4 DNA
  - 11.2.7.5 Trace
    - 11.2.7.5.1 Shoe wear impressions
    - 11.2.7.5.2 Physical matches
    - 11.2.7.5.3 Trace evidence
  - 11.2.7.6 Documents
  - 11.2.7.7 Firearms
  - 11.2.7.8 Computers
- 11.2.8 Planning the Analytical Sequence
  - 11.2.8.1 Evaluation and Consultation
  - 11.2.8.2 Prioritization
  - 11.2.8.3 Packaging
- 11.2.9 Special Situations
- 11.3 Health and Safety Hazards
  - 11.3.1 Incendiary devices are inherently dangerous.
- 11.4 Reading and Practical Exercises
  - 11.4.1 Bertsch W, Holtzer G, Sellers C. Chemical Analysis for the Arson Investigator and Attorney. Heidelberg; Hüthig Verlag GmbH; 1993.
  - 11.4.2 Coulson, S.A., Morgan-Smith, R. K. The Transfer of Petrol on to Clothing and Shoes While Pouring Petrol Around a Room. Forensic Science International 2000;112;135-141.
  - 11.4.3 Dean WL. Examination of Fire Debris for Flare (Fusee) Residues by Energy Dispersive X-Ray Spectrometry. Arson Analysis Newsletter. 1984; 8: 23-46.
  - 11.4.4 DeForest, PR, Gaensslen, RE, Lee, H. Forensic Science An Introduction to Criminalistics. McGraw-Hill, 1983.
  - 11.4.5 DeHaan J. Kirk's Fire Investigation. Upper Saddle River, New Jersey: Prentice Hall; current edition.

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- 11.4.8 Fisher BAJ. Techniques of Crime Scene Investigation. Boca Raton: CRC Press; current edition.
- 11.4.9 Midkiff CR. Arson and explosive investigation. In: Saferstein, R, ed. Forensic Science Handbook, Vol. I. Upper Saddle River, New Jersey: Prentice-Hall; current edition.
- 11.4.10 Powell, W. The Anarchist Cookbook. Any edition.
- 11.4.11 Saxon, K. The Poor Man's James Bond. Desert Publications, any edition.
- 11.4.12 Shelef R, et al. Development of Latent Fingerprints from Incendiary Bottles. Journal of Forensic Identification. 1996; 46(5):556-69.
- 11.4.13 Submit definitions of the following terms to the trainer.
  - 11.4.13.1 Incendiary device
  - 11.4.13.2 Molotov cocktail
  - 11.4.13.3 Incendiary mixture
  - 11.4.13.4 Thermite
  - 11.4.13.5 Pyrophoric
  - 11.4.13.6 Latent print
  - 11.4.13.7 Cellular DNA
  - 11.4.13.8 Footwear impression
- 11.4.14 Answer these key points and submit to the trainer:
  - 11.4.14.1 What are the concerns with the analysis of a Molotov cocktail? What other types of analysis might be needed and how can they be accommodated?
  - 11.4.14.2 What are the concerns with the analysis of incendiary devices in general? What other types of analysis might be needed? What are the needs and concerns of those types of analyses?
  - 11.4.14.3 Discuss how to prioritize analyses based on the needs of the investigation.
  - 11.4.14.4 Discuss how to prioritize analyses based on preservation of evidence.
  - 11.4.14.5 How might ignitable liquid analysis destroy certain types of evidence? How might it affect other analyses?
  - 11.4.14.6 How might other types of analysis ruin evidence for ignitable liquid analysis?

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11.4.14.7 How will the way an item is packaged and stored affect the different types of analysis?

#### 11.4.15 Practical Exercise

These practical exercises will be completed to the satisfaction of the instructor.

#### 11.4.15.1 Mock Consultations

- 11.4.15.1.1 Goals: The student will evaluate several types of potential fire debris evidence and determine what types of additional analyses could potentially be required. The student will consult with scientists in other functional areas and develop one or more strategies to accommodate as many analytical needs as possible based on the case scenario and needs of the submitting agency.
- 11.4.15.1.2 Procedure: Consider the following examples of evidence. For each example, try to think of what other types of analysis might be needed by the investigator. Try to think of the order of priority these analyses might have. Consult with scientists in the appropriate functional areas to determine their needs and concerns with analysis of the item. Develop one or more schemes of analysis for each item, depending on the possible needs of the investigator.
  - Suspect athletic shoes with bloodstains.
  - Molotov cocktail remains bottle neck with a twisted piece of t-shirt in it.
  - Gasoline can fragments with a disrupted pipe bomb taped to it.

## 11.4.15.2 Case Studies

- 11.4.15.2.1 Goals: The student will hear about unusual cases that required the scientist to adjust their usual procedures or use different chemical techniques.
- 11.4.15.2.2 Procedure: Ask two or three fire debris analysts other than the instructor to discuss a few of their more unusual cases. Find out how the scientist handled the analysis and if other functional areas were involved. Discuss these cases with the instructor.

#### 11.4.16 Written Examination

A written examination must be successfully completed before proceeding to the next module.

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