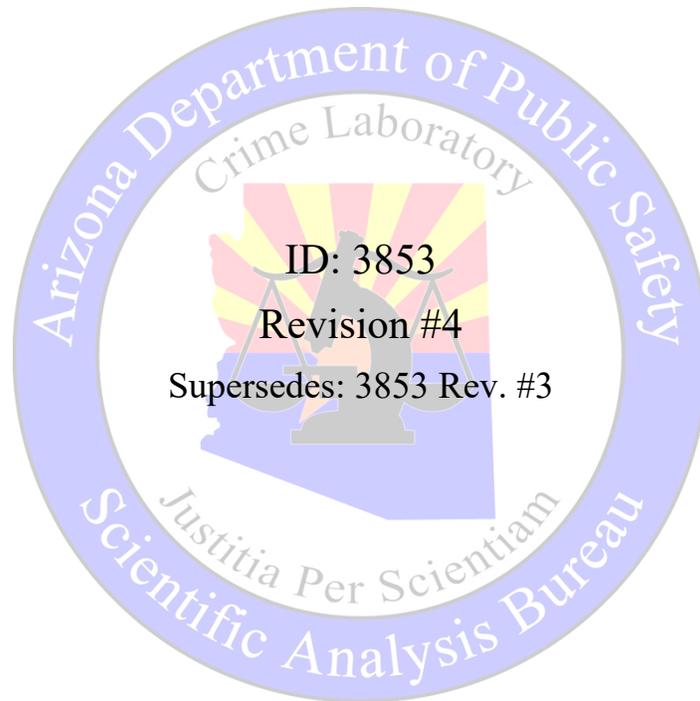


Arizona Department of Public Safety

Scientific Analysis Bureau

Ignitable Liquids Analytical Protocol



Issuing Authority:
SAB Superintendent

Effective Date: 09/09/2019



Ignitable Liquids Analytical Protocol

ID: 3853

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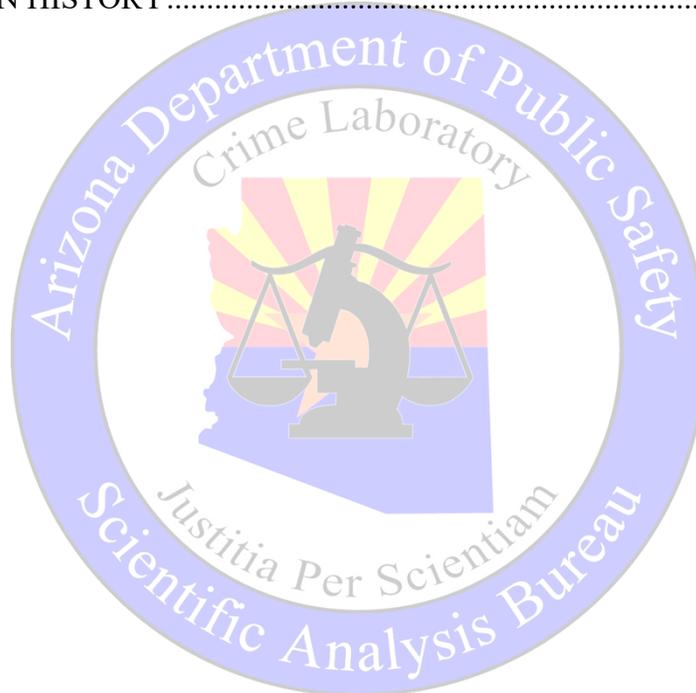
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1 GENERAL

The following DPS analytical protocol uses ASTM standards E 1386, E 1412, and E 1618 as references. Each laboratory conducting ignitable liquid analysis will have copies of these standards.

2 INSTRUMENTATION

For instrumentation specifications, parameters, and data analysis information see lab printouts for GC/MS.

3 CALIBRATION AND STANDARDS**3.1 Calibration**

Each day the GC/MS is used, the instrument will be tuned. A standard ignitable liquid mixture of 50% evaporated gasoline and diesel fuel and a sensitivity check mixture (defined in ASTM E 1618) will be run monthly when casework is being performed on the instrument, to ensure the reproducibility of chromatographic patterns and mass spectral data. The GC/MS TIC and EIP should approximate the resolution of the major component groups as shown in Figures 2-3 (see Appendix A). These calibrations will be filed in a location near the instruments.

3.2 Standards

- Each laboratory will generate a reference library run on its individual instrumentation.
- Neat and appropriate evaporated samples obtained from consumer products representing the major classes of ignitable liquids.
- Analytical samples available from chromatography or chemical suppliers (i.e. n-alkane mixes, pure compounds, isoparaffins, refinery samples etc.).
- Fire debris free of ignitable liquids, which contain typical pyrolysis products found in burnt carpet/padding, flooring materials, plastic/rubber products, and wood products such as particleboard and plywood.
- No classification may be identified in casework without the appropriate standard(s). Whenever possible, the standard(s) should be run on the specific instrument and under the same parameters as used for the analysis. However, many of the reference standards are commercial products, and variations in the formulations can occur over time during manufacture, even for the same product. Therefore, locating and running the standard(s) with the exact formulation on the laboratory instrument may not be possible. In such instances, a suitable known standard from a published reference source may be used to identify/classify the sample.
- A standard of the same ignitable liquids classification should be included in the case notes for any positive results.

4 EXTRACTION TECHNIQUES**4.1 Passive Adsorption**

Passive adsorption is the method of choice for arson samples that are packaged in airtight containers (i.e., paint cans, arson bags and mason jars).

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4.1.1 Procedure

- The container is briefly opened and a small amount of charcoal adsorbent is placed into the container.
- The container is resealed and placed into an approximately 80-90° C oven for a minimum of two hours. Depending on the sample matrix or density, a longer heating time may be required. After heating, the container is removed from the oven and allowed to cool.
- The adsorbent material is transferred to a test tube or auto sampler vial and eluted with carbon disulfide, pentane or an appropriate solvent.
- Approximately 1.0 ul of the sample extract is analyzed by gas chromatography and/or gas chromatography-mass spectrometry.

4.2 Solvent Wash

Due to size and/or packaging, certain items may not be suited for sampling using the passive adsorption technique. In these instances the solvent extraction technique may be used.

4.2.1 Procedure

- Place as much of the sample as is practical into a clean beaker.
- Pour pentane or carbon disulfide over the sample.
- Decant the solvent into a separate clean beaker.
- Evaporate the eluent to a small volume (approx. 1.0 ml) to concentrate any ignitable liquid residues that may be present.
- Approximately 1.0 ul of the eluent is analyzed by gas chromatography and/or gas chromatography-mass spectrometry.

4.3 Direct Sample

- Neat liquid samples may be diluted with carbon disulfide, pentane or an appropriate solvent.
- A dilution of approximately 1:20 is recommended but it is realized that the dilution factor will be instrument dependent.
- Approximately 1.0 ul of the dilute sample may be analyzed by gas chromatography and/or gas chromatography-mass spectrometry.

5 ANALYSIS TECHNIQUES**5.1 Blanks**

Each ignitable liquid analysis should include one of the following:

- A blank solvent elution of a sample of the charcoal adsorbent used for the passive adsorption extraction to ensure that the solvent and charcoal adsorbent were free of contamination.
- A solvent blank, to show that the solvent used for sample extraction or dilution was free of contamination. (Note: Blanks for solvent extraction samples should consist of an

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equal volume of extraction solvent, evaporated to the same volume as the analyte.)

Blanks must be run between positive samples to ensure that there is no carry-over contamination in the instrumental system.

5.2 GC/MS

- Utilizing one of the above described extraction techniques, a sample is obtained for analysis with a gas chromatograph/mass spectrometer.
- The resulting total ion chromatogram and/or mass spectral data and extracted ion profiles are evaluated by comparison to the laboratory's reference library and by use of ASTM standard E 1618 classification criteria (Appendix B).

5.3 Flame Test

If a liquid sample is submitted for analysis, a flame test may be run. An example of a flame test procedure is: place a few drops of the liquid into a non-flammable container such as a metal weigh boat and then expose the liquid to a flame. The results of this test are then documented in the analyst's notes.

5.4 Preserving Samples

A portion of the charcoal adsorbent, original liquid or solvent extract will be preserved in the original evidence container in the event that defense re-analysis is required. This sample should be packaged in an airtight container, such as a crimp cap or screw top vial.

6 REPORTING RESULTS**6.1 General**

- When GC/MS analysis indicates the presence of an ignitable liquid, the report should reflect the class name of the material and list examples from that class. Note: class names and examples are taken from ASTM standards (see attached classification table, Appendix B.)
- When no significant characteristics of an ignitable liquid are detected, a report reflecting that no ignitable liquids were detected should be written.
- When an analysis indicates characteristics of an ignitable liquid but contains significant contaminants which cannot be resolved by GC/MS, the report should indicate inconclusive results.

6.2 Suggested Report Verbiage**6.2.1 Positive**

- Contained residue consistent with the _____ class of ignitable liquids. Some examples of this class are _____.
- The liquid is consistent with the _____ class of ignitable liquids. Some examples of this class are _____.
- Item #1 contains _____ (gasoline, mpd, etc.). Some examples of this class of ignitable liquids are _____.



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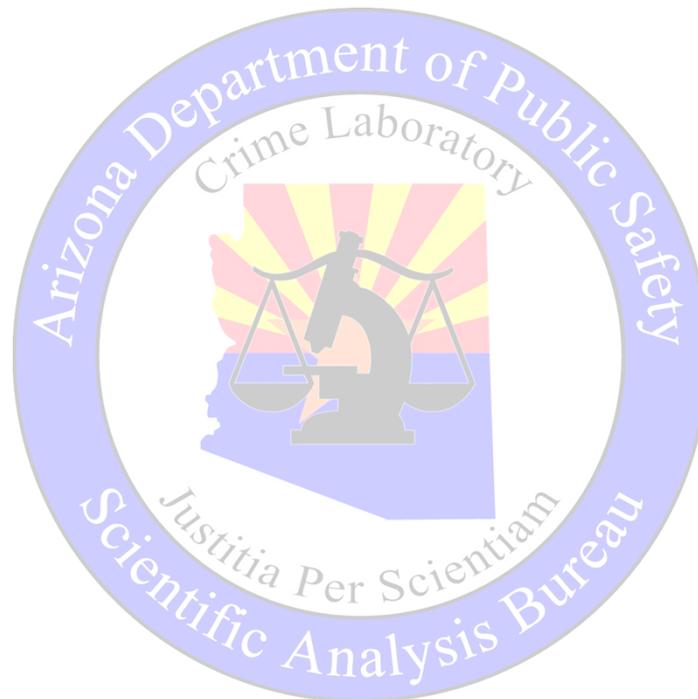
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6.2.2 Negative

- No ignitable liquids were detected.
- No ignitable liquid residues were identified.

6.2.3 Inconclusive

- The analysis of Item XXX could neither establish nor exclude the presence of ignitable liquid residues. The results were inconclusive due to (limited abundance of peaks, extraneous peaks, etc.)





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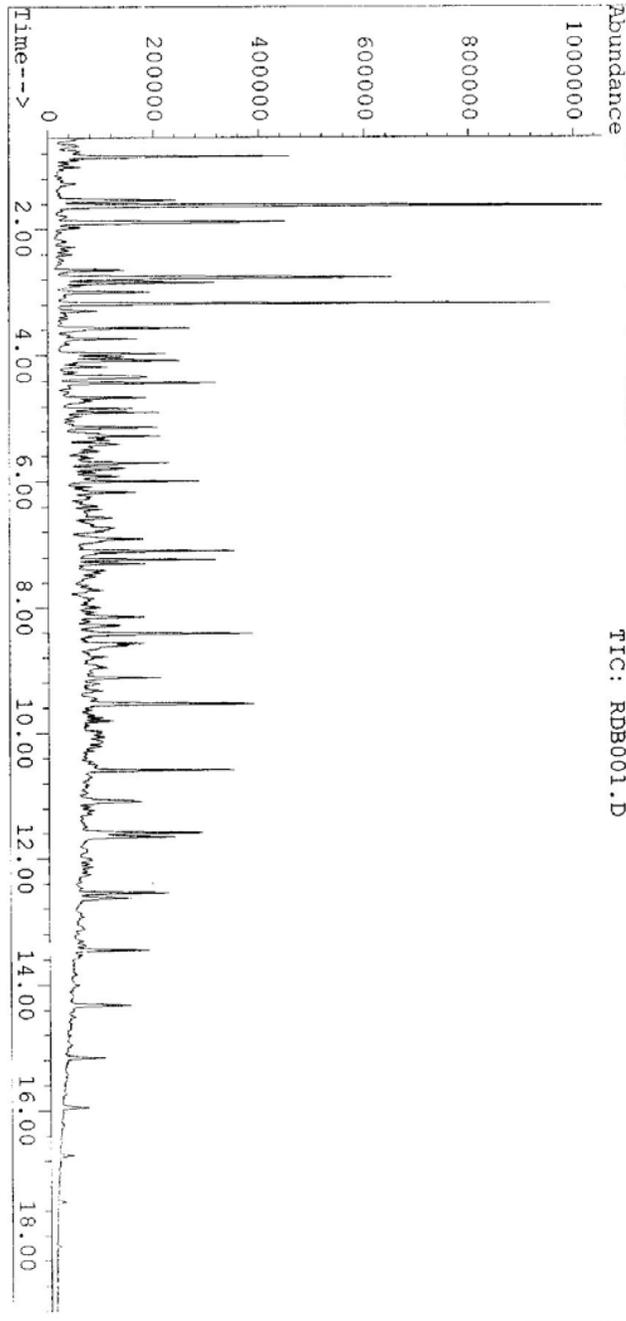
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Appendix A CALIBRATION DATA

FIGURE 2

STD IGN LIQ MIX



ARION REPORT PAGE 1

FILE NAME : C:\HPCHEM\1\DATA\IGLQOTTO\RDB001.D
 SAMPLE ID : STD IGN LIQ MIX
 INFO : 50% EVAP GASOLINE/ DIESEL FUEL
 INSTRUMENT : HEWLETT PACKARD 5890 GC - 5972 MSD

VIAL NUMBER : 1
 ACQUISITION METHOD: IGLQOTTO
 DATE OF ANALYSIS: 19 May 99

8:31

ARIZONA DPS CRIME LABORATORY
 FIRE DEBRIS ANALYSIS SECTION
 system S/N 3321A63055 MSD S/N 3402A01442
 (520) 773-3687



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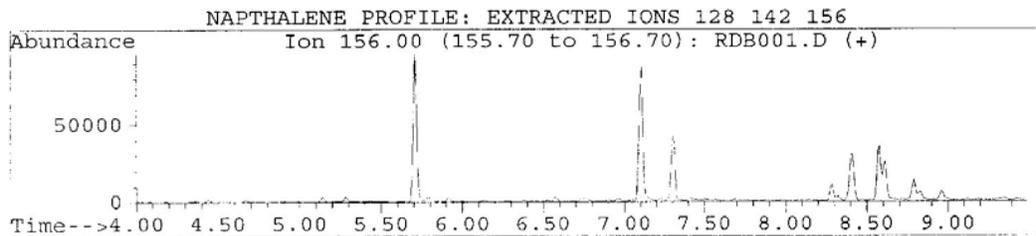
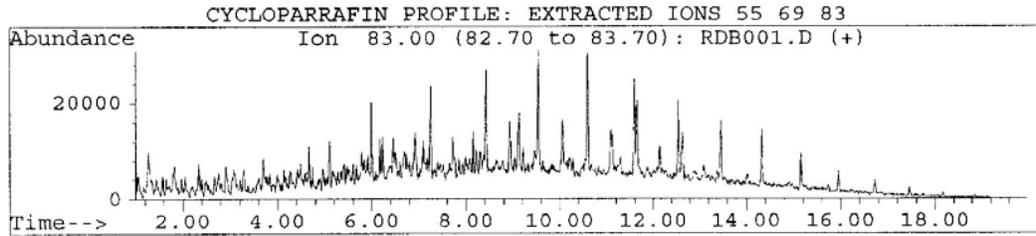
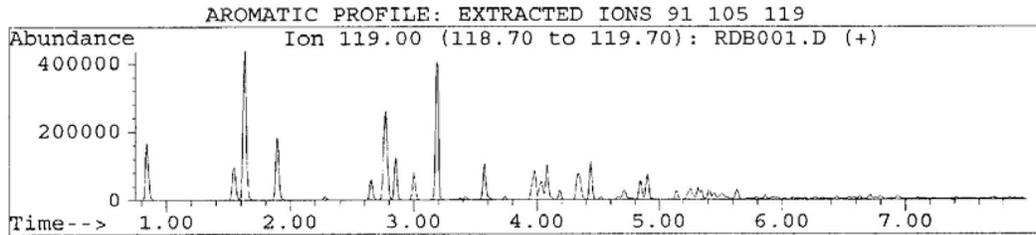
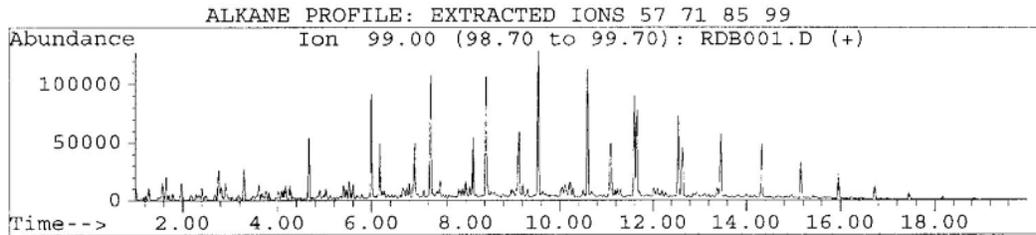
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FIGURE 3

ARIZONA DPS CRIME LABORATORY
FIRE DEBRIS ANALYSIS SECTION
system S/N 3321A63055 MSD S/N 3402A01442
***** EXTRACTED ION PROFILING REPORT *****

SAMPLE : STD IGN LIQ MIX
INFO : 50% EVAP GASOLINE/ DIESEL FUEL



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Appendix B IGNITABLE LIQUID CLASSIFICATION SYSTEM

Class Name	Peak Spread	Examples
Light Petroleum Distillate	C4 - C9	Petroleum ethers, pocket lighter fuels, some rubber cement solvents, Skelly solvents, V M & P Naphtha, some camping fuels
Gasoline	C4 – C12	All brands & grades of automotive gasoline, including gasohol
Medium petroleum Distillate	C8 – C13	Mineral spirits, some paint thinners, some charcoal starters, dry cleaning solvents, some torch fuels, some solvents for insecticides and polishes, some lamp oils
Heavy Petroleum Distillate	C9 – C20+	No. 1 & No. 2 fuel oil, kerosene, diesel fuel, some jet fuels, some charcoal starters, some solvents for insecticides and polishes
Miscellaneous	Variable	Single compounds, turpentines, enamel reducers, specialty mixtures that cannot be further classified into one of the categories below.
Oxygenated Solvents	Variable	Alcohols, esters, ketones
Isoparaffinic Products	Variable	Some charcoal starters, some copier toners, some aviation gasolines, some lamp oils, some specialty/industrial solvents
Normal Alkanes	Variable	Specialty products formulated from normal alkanes, some lamp oils, copier toner, carbonless paper
Aromatic Products	Variable	Specialty cleaning solvents, fuel additives, some paint and varnish removers, some automotive parts cleaners, xylenes, toluene based products
Naphthenic Paraffinic Products	Variable	Some charcoal starters, some paint thinners, some insecticide vehicles, some lamp oils, industrial solvents



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Appendix C REVISION HISTORY

Changes Made	Date	Issuing Authority
ILP2007.01 - <ul style="list-style-type: none"> • Added flame test • Removed dearomatized distillate class 	2007	SAB Superintendent
ILP2014.01 - <ul style="list-style-type: none"> • Modified requirements for standards to allow the use of published references/standards if matching standard could not be located and run on DPS laboratory equipment. • Changed suggested verbiage for an “inconclusive” to include the reason for the inconclusive finding. 	2014	SAB Superintendent
1 st Revision in Qualtrax	October 2015	SAB Superintendent
ILP Rev 2- <ul style="list-style-type: none"> • Changed ignitable liquid mixture to run monthly when casework is being performed. 	2016	SAB Superintendent
ILAP Rev 3 Significant Changes Removed auto open macro and changed inconclusive verbiage, fixed grammatical errors/punctuation. No additional changes	Aug 2018	SAB Superintendent
ILAP Rev 4 Removed GC-FID procedure and references, (withdrawn by ASTM)	March 2019	SAB Superintendent