

Clinical Forensic Applications note

cat.# 59574

Fire Debris Analysis

Capillary gas chromatography is the method of choice for analyzing suspected arson samples from fire debris. Because of the complex composition of most accelerants used in arson cases, it is crucial that positive identification be made of the material used to start the fire. The American Society for Testing Materials (ASTM) has developed standard test procedures for analyzing these samples. The information presented in this article references ASTM E1387, "Standard Test Method for Flammable or Combustible Liquid Residues in Extracts from Samples of Fire Debris by Gas Chromatography".

As with all analytical procedures, sample collection, preservation, chain of custody, and sample preparation play crucial roles in the process. Samples may be extracted using several different techniques¹ (beyond the scope of this article) for introduction into the gas chromatograph.

Appropriate capillary column selection is the first decision the analyst must make. The ASTM standard allows the use of any capillary column and conditions, provided that a Resolution Test Mix is completely resolved into the individual components. This resolution test mix consists of equal weights of the even numbered n-alkanes from C6 to C20, plus several aromatic compounds. The aromatics specified are: p-xylene, toluene, 2-ethyltoluene, 3-ethyltoluene, and 1,2,4-trimethylbenzene.

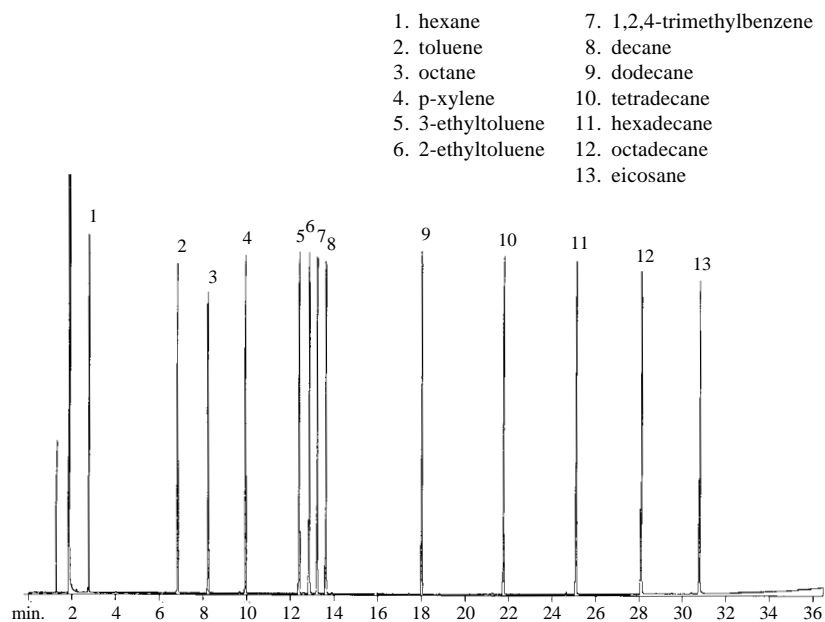
Several different stationary phases and column configurations can provide the resolution needed. Typically, laborato-

ries can use 30-meter columns coated with either Rtx[®]-1 (100% dimethyl polysiloxane) or Rtx[®]-5 (5% diphenyl 95% dimethyl polysiloxane). Film thicknesses can vary from 1.0 to 1.5 μ m. Choice of column ID should depend upon sample capacity and the detection system employed. The standard allows for the use of either FID or MS detectors. If MS detection is employed, use a 0.25mm ID column to minimize carrier gas flow. If FID detection is employed, use a 0.53mm ID to maxi-

mize column capacity. Use of 0.53mm ID columns can minimize expensive duplicate analyses or dilutions if the concentration of accelerants are very high. **Figure 1** shows the complete resolution of all 13 components in the column resolution check mix on a 30m, 0.53mm ID, 1.5 μ m Rtx[®]-1 column with an FID detector. The linear velocity and temperature program chosen allow the entire analysis to be completed in approximately 16 minutes.

Figure 1

Resolution Check Mix demonstrates proper column performance.

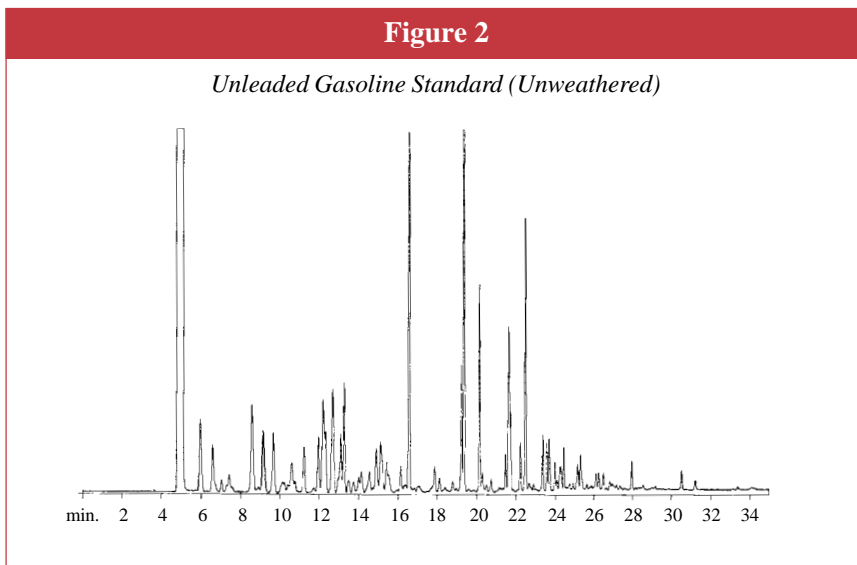


30m, 0.53mm ID, 1.5 μ m Rtx[®]-1 (cat.# 10170).

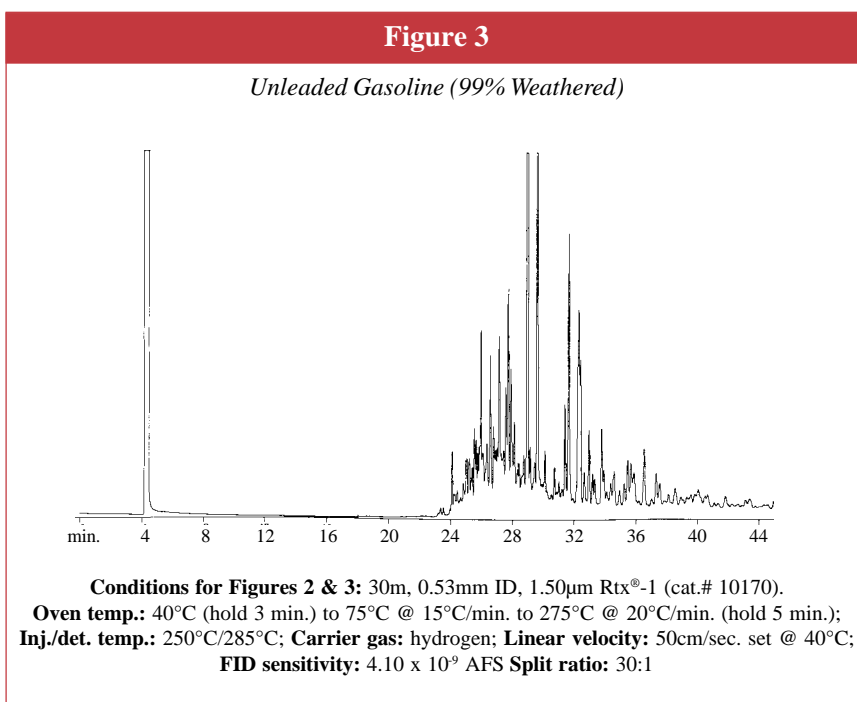
1.0 μ l split injection of E1387-90 Column Resolution Check Mix (cat.# 31224).

Oven temp.: 40°C (hold 3 min.) to 75°C @ 15°C/min. to 275°C @ 20°C/min. (hold 5 min.);
Inj./det. temp.: 250°C/285°C; Carrier gas: hydrogen; Linear velocity: 50cm/sec. set @ 40°C;
FID sensitivity: 4.10 x 10⁹ AFS Split ratio: 30:1

After establishing the correct conditions to obtain complete resolution of the test mix components, the analyst must then calibrate the instrument. In fire debris analysis, this involves purchase and preparation of common accelerants used to ignite fires with subsequent injection into the GC. Quantitation of unknown samples is not performed. The analyst must provide positive identification to the field investigators of any accelerants found in the samples collected. To do this, the analyst must be able to recognize typical chromatographic patterns of each accelerant. **Figure 2** shows the chromatographic pattern obtained from an injection of an unleaded gasoline standard.



To further complicate this analysis, many factors will change the chromatographic pattern obtained from fire debris. The first is weathering of the material from the heat of the fire along with dilution of water used to extinguish the blaze. This weathering can dramatically change the chromatographic pattern of the material. Typically, lower boiling materials are lost by the heat, leaving the higher boiling compounds remaining. This type of weathering can be simulated in the laboratory by evaporating the material under controlled conditions. The advantage of performing the evaporation in the laboratory is that the exact amount of weight loss compared to the original starting material can be measured and controlled. **Figure 3** shows the analysis of a 99% weathered unleaded gasoline. The gasoline has been weathered to a 99% weight loss and an exact concentration calibration standard was prepared with the remaining material. By analyzing known weathered products, the analyst can more readily recognize the type of original starting material.



Conditions for Figures 2 & 3: 30m, 0.53mm ID, 1.50µm Rtx®-1 (cat.# 10170).
Oven temp.: 40°C (hold 3 min.) to 75°C @ 15°C/min. to 275°C @ 20°C/min. (hold 5 min.);
Inj./det. temp.: 250°C/285°C; **Carrier gas:** hydrogen; **Linear velocity:** 50cm/sec. set @ 40°C;
FID sensitivity: 4.10 x 10⁹ AFS **Split ratio:** 30:1

There are five basic classes of complex petroleum distillates normally identifiable in arson samples. A sixth class of accelerants (Class 0) includes single compounds sometimes used and identified. **Table I** shows the complete list of classes, typical chromatographic range of each material (based upon hydrocarbon elution), and examples of each type.

Table I - Typical Hydrocarbons

Class #	Range	Examples
1 Light Petroleum	C4-C8	Pocket lighter fuel, petroleum ethers, some rubber cement solvents
2 Gasoline	C4-C12	Gasoline (ALL), some camping fuels
3 Medium Petroleum Distillates (MPD)	C8-C12	Mineral spirits, paint thinners, some torch fuels, some charcoal fuels, some charcoal starters
4 Kerosene	C9-C16	Kerosene, No. 1 Fuel Oil, Jet-A Fuel Oil, Jet-A Fuel, some charcoal starters, some torch fuels
5 Heavy Petroleum Distillates (HPD)	C10-C23	No. 2 Fuel Oil, Diesel Fuel #2
0 Unclassified	Variable	Alcohols, acetone, toluene, some lamp oils, camping fuels, lacquer thinners

There are additional variables which can make identification of the petroleum residue more difficult. Included would be co-extracted volatiles and pyrolyzates from the fire debris. The extent of these co-extracted interferences would, in part, be dependent upon the sample preparation method used. The ASTM method does provide minimum requirements for class identification for each type of accelerant, but in the end the experience of the analyst is crucial.

Adequate chromatographic resolution can be obtained on several different capillary columns. Typically, the best resolution can be obtained on an Rtx[®]-1 (100% dimethyl polysiloxane) stationary phase. Column configuration should be based upon the detection system being used and sample capacity. Calibration with weathered petrochemical standards also plays an important part in identification of accelerants extracted from fire debris samples. ❖

References

- 1) ASTM Standard Practices for Fire Debris Extraction:
 - ASTM E1412 Passive Headspace Concentration
 - ASTM E1413 Dynamic Headspace Concentration
 - ASTM E1385 Steam Distillation Concentration
 - ASTM E1386 Solvent Extraction Concentration
 - ASTM E1388 Sampling of Headspace Vapors
 - ASTM E1389 Cleanup by Acid Stripping

Rtx[®]-1 Column Listing

30m, 0.53mm ID, 1.5µm cat.# 10170
 30m, 0.32mm ID, 1.0µm cat.# 10154
 30m, 0.25mm ID, 1.0µm cat.# 10153

Weathered Petrochemical Analytical Reference Materials

May be used for:

- Underground Storage Tank Monitoring
- ASTM fire debris analysis

Laboratories monitoring underground storage tanks often find it difficult to determine the type of petrochemical detected during an analysis. The main reason for this is the petroleum product has weathered from exposure to air, water, sunlight, and microbial action. All of these factors can lead to misidentification of the original product.

A similar situation occurs for forensic analysts investigating potential arson cases. When arson is suspected, samples are taken from the site and analyzed using ASTM E1387 methods. In these methods, quantitation is not performed but identification of the accelerant (if any) is crucial. Again, weathering of the petrochemical can drastically change the chromatographic profile when compared to non-weathered material.

Restek is now offering as stock products, weathered petrochemical products to meet many of these difficult situations. All of these standards are prepared from commercially acquired materials. The material is then weathered in the laboratory based upon a specific weight loss from the original weight of starting material. While we cannot duplicate all environmental or arson factors, these standards may be useful in identification of the type of petrochemical detected.

ASTM E1387 Fire Debris Analysis

Adequate column resolution is addressed in this protocol. Any capillary column can be used provided resolution of all analytes can be achieved. To demonstrate performance, a column resolution check mix must be analyzed prior to any sample analysis. Listed below is the required column performance mixture.

E1387 Column Resolution Check Mix

Contains the compounds listed at 2000µg/ml each in methylene chloride. Packaged 1ml per ampul.

decane	octadecane
dodecane	octane
eicosane	tetradecane
2-ethyltoluene	toluene
3-ethyltoluene	1,2,4-trimethylbenzene
hexadecane	p-xylene
hexane	

cat.#: 31224 ea.
 31224-500 ea. w/data pk.
 31224-510 5-pk.
 31224-520 5-pk. w/data pk.
 31324 10-pk. w/data pk.

Unleaded Gasoline ASTM Class 2 Accelerant	Single cat.#	Single w/data pack cat.#	5 pack cat.#	5 pack w/data pack cat.#	10 pack cat.#
Unleaded Gasoline Standard (unweathered)	30096	30096-500	30096-510	30096-520	30196
Unleaded Gasoline Standard (25% weathered)	30097	30097-500	30097-510	30097-520	30197
Unleaded Gasoline Standard (50% weathered)	30098	30098-500	30098-510	30098-520	30198
Unleaded Gasoline Standard (75% weathered)	30099	30099-500	30099-510	30099-520	30199
Unleaded Gasoline Standard (99% weathered)	30436	30436-500	30436-510	30436-520	30536
Weathered Gasoline Kit (cat.# 30100 or 30100-500 w/data pack) Contains one ampul (1ml) of unweathered standard (30096), 25% (30097), 50% (30098), and 75% (30099) weathered standards.					
Weathered Gasoline Kit #2 (cat.# 30437 or 30437-500 w/data pack) Contains one ampul (1ml) of unweathered standard (30096), 25% (30097), 50% (30098), 75% (30099), and 99% (30436) weathered standards.					

Mineral Spirits ASTM Class 3 Accelerant	Single cat.#	Single w/data pack cat.#	5 pack cat.#	5 pack w/data pack cat.#	10 pack cat.#
Mineral Spirits Standard (unweathered)	31225	31225-500	31225-510	31225-520	31325
Mineral Spirits Standard (25% weathered)	31226	31226-500	31226-510	31226-520	31326
Mineral Spirits Standard (50% weathered)	31227	31227-500	31227-510	31227-520	31327
Mineral Spirits Standard (75% weathered)	31228	31228-500	31228-510	31228-520	31328
Weathered Mineral Spirits Kit (cat.# 31237 or 31237-500 w/data pack) Contains one ampul (1ml) of unweathered standard (31225), 25% (31226), 50% (31227), and 75% (31228) weathered standards.					

Kerosene ASTM Class 4 Accelerant	Single cat.#	Single w/data pack cat.#	5 pack cat.#	5 pack w/data pack cat.#	10 pack cat.#
Kerosene Standard (unweathered)	31229	31229-500	31229-510	31229-520	31329
Kerosene Standard (25% weathered)	31230	31230-500	31230-510	31230-520	31330
Kerosene Standard (50% weathered)	31231	31231-500	31231-510	31231-520	31331
Kerosene Standard (75% weathered)	31232	31232-500	31232-510	31232-520	31332
Weathered Kerosene Kit (cat.# 31238 or 31238-500 w/data pack) Contains one ampul (1ml) of unweathered standard (31229), 25% (31230), 50% (31231), and 75% (31232) weathered standards.					

Diesel Fuel #2 ASTM Class 5 Accelerant	Single cat.#	Single w/data pack cat.#	5 pack cat.#	5 pack w/data pack cat.#	10 pack cat.#
Diesel Fuel #2 Standard (unweathered)	31233	31233-500	31233-510	31233-520	31333
Diesel Fuel #2 Standard (25% weathered)	31234	31234-500	31234-510	31234-520	31334
Diesel Fuel #2 Standard (50% weathered)	31235	31235-500	31235-510	31235-520	31335
Diesel Fuel #2 Standard (75% weathered)	31236	31236-500	31236-510	31236-520	31336
Weathered Diesel Fuel #2 Kit (cat.# 31239 or 31239-500 w/data pack) Contains one ampul (1ml) of unweathered standard (31233), 25% (31234), 50% (31235), and 75% (31236) weathered standards.					



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