Fire & Smoke Damage Testing

POCKET GUIDE

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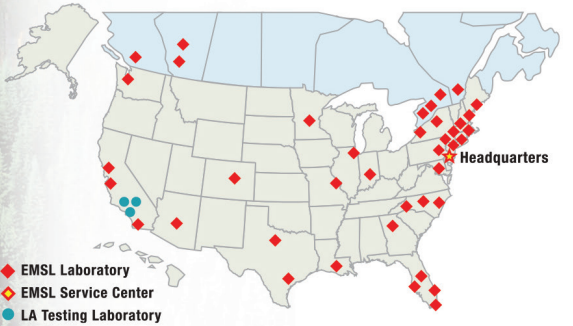

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Fire & Smoke Damage Testing

(Char, Ash, Black Carbon/Soot)

The purpose of this guide is to provide information regarding the typical sampling and analytical procedures involved in fire debris investigations. The components of fire debris are complex and their nature depends on the type of materials involved in the fire, the combustion conditions, and the presence of fuels/accelerators. In general, the common by-products of fires can be classified in three categories: char, ash, and soot/black carbon.

Char is defined in ASTM D6602-13 as particulate larger than 1 µm made by incomplete combustion which may not deagglomerate or disperse by ordinary techniques, may contain material which is not black, and may contain some of the original material's cell structure, minerals, ash, cinders, and so forth. Carbon is typically the predominant component in char.

Ash is pyrolized material obtained from advanced combustion of char. Because the carbon matrix of char is almost completely combusted, ash has a minerals-based matrix, with a high amount of elements such as calcium, potassium, magnesium, aluminum, silicon, phosphorus or sulfur. The particles are very brittle and may or may not contain some of the original material's cell structure.

Soot is defined in ASTM D6602-13 as submicron black powder generally produced as an unwanted by-product of combustion or pyrolysis. It consists of various quantities of carbonaceous and inorganic solids in conjunction with adsorbed and occluded organic tars and resins. The EPA defines black carbon as the sooty black material emitted from gas and diesel engines, coal-fired power plants, and other sources that burn fossil fuel.

Carbon black is a term usually associated with soot, although they are different materials and have distinctively different origins. Carbon black is an engineered,

industrially produced material, primarily composed of elemental carbon, obtained from the partial controlled combustion or thermal decomposition of hydrocarbons (most hydrocarbons are found in crude oil and other fuels).

Soot/black carbon and carbon black terms should not be used interchangeably.

The most important characteristics of soot/black carbon and carbon black particles are their size range and the aciniform morphology. *Aciniform carbon* is colloidal carbon having a morphology consisting of spheroidal primary particles (nodules) fused together in aggregates of colloidal dimension in a shape having grape-like clusters or open branch-like structures.

The Main Differences Between Soot/Black Carbon and Carbon Black are:

- For carbon black, the particle size and aggregate size vary depending on the carbon black grade; while particle and aggregate sizes vary greatly within a given grade of carbon black, the primary particle size is essentially uniform within an individual aggregate.
- Soot/black carbon may have several carbon morphologies depending on the source; the particle size range is wide and non-uniform.
- Carbon black grades are quite low in volatile organic content (<8 %) whereas soot/black carbon is normally high in volatile organic content (>20 %).
- Soot/black carbon contains minerals, residual tars and resins where carbon black may contain trace amounts of hydrocarbons used as precursors.

Air Sampling

Air sampling for combustion by-products

Option 1: Gravimetric analysis based on NIOSH 5000- Carbon Black

Sampler: tared 5-µm PVC membrane
Flow rate: 1-2 Liter/min
Min Volume: 30 Liters at 3.5 mg/m^3
Max Volume: 570 Liters
Results expressed in mg/m^3 as carbon black

Pre-Weighted/Tared 37 mm 5 µm PVC Cassette

Advantages

- Quick and simple procedures.
- Analysis is simple.

Disadvantages:

- Analysis is non-specific. The presence of any other particulate material in the air being sampled will be a positive interference since this is a gravimetric method. Information on any other particulate materials present should be assessed, by using electron microscopy for example.
- Sampling requires an air sampling pump and air sampling cassettes.
- The sample represents the particles present in the air at the time of collection.

Sampling Procedure

- Calibrate each personal sampling pump with a representative sampler in line. Sample at 1 to 2 L/min for a total sample volume of 30 to 570 L. Do not exceed a filter loading of approximately 2 mg total dust. Take two to four replicate samples for each batch of field samples for quality assurance on the sampling procedures.

- Remove the cassette from the sampling tubing.
- Complete the applicable EMSL Chain of Custody (COC), detailing client name and information, project name or number, sample #, description of area, and volume of air collected.
- Ship the samples along with COC to the laboratory.

Option 2: PCM or TEM Cassettes (based on NIOSH 7400 sampling method)

Sampler: 0.45 to 1.2 µm mixed cellulose ester membrane (MCE), 25 to 37 mm diameter
Flow rate: 0.5-16 Liter/min
Min Volume: 400 liters
Max Volume: 1000-1200 liters
Results expressed in one of the options below:

- Particles/liter
- Particles/cm^3
- Particles/m^3
- % Particles (by point count procedures)

0.8 µm Mixed Cellulose Ester Membrane (MCE), 25 mm Diameter

Advantages

- Able to determine the amount of target analytes present in the air at the time of collection, derived by particle count.

Disadvantages:

- The sample represents the particles present in the air at the time of collection; therefore collection is representative if the combustion event is current.

- Sampling requires pump and cassettes.
- Analysis provides full char and ash analysis; the soot/black carbon analysis is presumptive only.

Sampling Procedure

- Calibrate each personal sampling pump with a representative sampler in line. To reduce contamination and to hold the cassette tightly together, seal the crease between the cassette base and the cowl with a shrink band or light colored adhesive tape.
- Submit at least one field blank (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling.
- Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.
- Sample at 0.5 L/min or greater.
- Remove the cassette from the sampling tubing after sampling.
- Complete the applicable EMSL Chain of Custody (COC), detailing client name and information, project name or number, sample #, description of area, and volume of air collected.
- Ship the samples along with COC to the laboratory.

Option 3: Air-O-Cell

Sampler: Air-O-Cell cassette
Flow rate: 15 Liter/min
Min Volume: 15 liters (dusty environment)
Max Volume: 150 liters
(no visible dust, "clean" environment)
Typical volume: 75 liters
Results expressed in one of the options below:

- Particles/liter
- Particles/m^3
- Particles/cm^3

AIR-O-Cell Cassette

Advantages

- Quick and simple procedures.
- Analysis is simple.
- Able to determine the amount of target analytes present in the air at the time of collection, derived by particle count.

Disadvantages

- The sample represents the particles present in the air at the time of collection; therefore, collection is representative if the combustion event is current.
- Sampling requires an air sampling pump and air sampling cassettes.
- Analysis provides full char analysis; ash analysis may be hindered due to the presence of adhesive on the trace; the soot/black carbon analysis is presumptive only.

Sampling Procedure

- Prior to sampling, calibrate the pump at 15 liters/min with a rotameter.
- Remove and retain the tape seal covering inlet and outlet on the cassette.
- Attach the outlet (round hole) to a standard ½" PVC tubing (for use with high volume pumps only).
- Start the sampling pump and sample for an appropriate amount of time.
- Remove Air-O-Cell from tubing and reseal.
- Complete the applicable EMSL Chain of Custody (COC), detailing client name and information, project name or number, sample #, description of area, and volume of air collected.
- To reduce shipping damage, it is recommended that the Air-O-Cell be placed in a corrugated box with padding to ensure safe arrival at the laboratory.

Surface Sampling

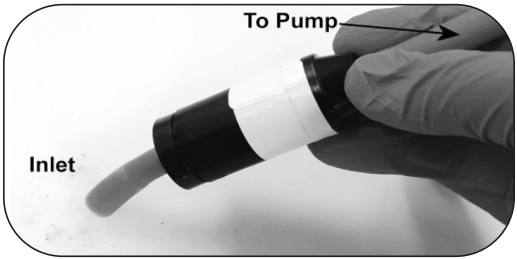
Surface sampling for combustion by-products

Option 1: Micro Vacuuming

Micro-vacuum (micro-vac) sample collection involves the use of air filter cassettes. Commonly, MCE filter cassettes are attached to high volume pumps in the same manner as air sampling. A Closed Face cassette setup is used in order to attach a short length of tubing to the sampling inlet. The particulate is then collected by crisscrossing the sampling area with the collection tube.

Carpet Sample Kit

Micro-Vac Cassette Options



Micro-Vacuuming Tubing Set-Up

Advantages

- Able to determine the amount of target analytes present on surfaces during or after the event.
- Very efficient sampling method for collecting particles from porous, uneven or difficult to access places with medium and heavy loading (rugs, fabrics and collection vents, narrow grills or cracks).
- The samples represent bulk amount of particle material, often of many different sizes.
- A variety of optical and electron microscopy methods can be used in the identification analysis.
- Confirmatory identification of aciniform soot by Transmission Electron Microscopy (TEM), as mandated in ASTM D6602-13, can be applied using the drop-mount technique.

- Chemical analysis of organic compounds associated with the fire deposits through bulk spectroscopy and/or chromatography (such as PAH's) can be applied.
- Corrosivity analysis via pH measurement or anions scan by Ion Chromatography can be applied.

Disadvantages

- Sampling requires an air sampling pump and air sampling cassettes.
- Poor efficiency for collecting particles from relatively smooth non-porous surfaces with low loading.
- It does not preserve the relative positions of the particles on the original surface and the population per unit area; however, this is a limitation only when the agglomerate size and the distribution over the collection surface is of interest.
- Can induce damage to brittle particles such as char and ash; if proper sampling and sample preparation procedures are applied, the damage can be greatly minimized.

Recommended surfaces for sampling

Main living areas, interior of door frame, corner of floors; door tracks; attic areas; soft surfaces, wood, upholstery, clothing.

Option 2: Tape Lifting (Forensic Adhesive Lifts, Transparent Office Tape)

Advantages

- Quick and simple sampling procedures.
- Able to determine the amount of target analytes present on surfaces during or after the event.
- Efficient sampling method for collecting particles from relatively smooth non-porous surfaces with typical monolayer loading (desks, furniture, glass and hard floors).
- Preserves the relative positions of the particles on the original surface and the population per unit area.
- A variety of optical microscopy methods can be used in the identification analysis, with minimal preparation.
- Scanning Electron Microscopy/Energy Dispersive X-rays (SEM/EDX) methodology can be applied for comprehensive characterization of char and ash and presumptive identification of soot clusters.

Disadvantages

- Poor efficiency for collecting on porous, uneven or heavily loaded surfaces, showing preferential sampling from the top layer particles.
- Application of overpressure during sampling can obscure or damage the brittle particles of char and ash.

Forensic Tape Lift (Instant Tape Lifters)

Tape Lift (Transparent/Clear Tape)

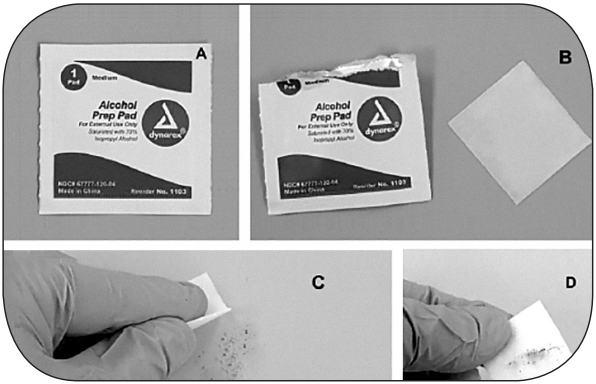
- Limited sampling area.
- Particles that are part of large agglomerations many not be correctly identified by applicable methods due to overlapping.
- Confirmatory identification of aciniform soot by Transmission Electron Microscopy (TEM), as mandated in ASTM D6602-13, cannot be applied.
- Chemical analysis of organic compounds associated with the fire debris through spectroscopy and/or chromatography (such as PAH's) cannot be applied.
- Corrosivity analysis via pH measurement or anions scan by Ion Chromatography cannot be applied.

Note: Packing tape should be avoided since these products have a thick layer of adhesive that can trap particles hence hindering analysis. Non-transparent and industrial tapes, such as duct tape cannot not be used.

Recommended surfaces for sampling
Main living areas, interior of door frame, corner of floors, door tracks, attic areas.

Option 3: Wet Wiping/Alcohol Prep Wipes

Unlike Ghost Wipes, paper towels and commercial products such as Wet-Wipes™, alcohol pads have a small surface area so particles are easily extracted for analysis. Particles tend to become trapped in larger wipes and paper products often interfering with accurate analysis. Avoid using water-based moistening agents as many common particles are water soluble.



Alcohol Prep Wipes

Advantages

- Quick and simple sampling procedures.
- Efficient sampling method for collecting particles from relatively smooth non-porous surfaces with low or heavy loading.

- A variety of optical and electron microscopy methods can be used in the identification analysis.
- Confirmatory identification of aciniform soot by Transmission Electron Microscopy (TEM), as mandated in ASTM D6602-13, can be applied using the drop-mount technique.
- Particle dispersion techniques for breaking up the agglomerates enables more accurate identification of individual grains, necessary when environmental interferences are suspected.
- Chemical analysis of organic compounds associated with the fire deposits through bulk spectroscopy and/or chromatography (such as PAH's) can be applied.
- Corrosivity analysis via pH measurement or anions scan by Ion Chromatography can be applied.

Disadvantages

- Poor efficiency for collecting on porous and uneven surfaces.
- It does not preserve the relative positions of the particles on the original surface and the population per unit area; however, this is a limitation only when the agglomerate size and the distribution over the collection surface is of interest.
- Can induce damage to brittle particles such as char and ash.

- There can be variance in what particles are successfully transferred from the wipe and therefore isolated for analysis.

Recommended surfaces for sampling
TVs, computer displays, plastic surfaces, furniture, windows, refrigerators.

Note: avoid painted surfaces due to transfer of the paint on the wipe.

Option 4: Bulk/ Grab Sample

Abundant deposits are generally treated as bulk when sampling. Sampling consists of transferring the material/fire debris to a zip-lock style bag or rigid container. Sampling may be performed by glove-bag, trowel, scraping the material into a pile or any other technique available.

Advantages

- Quick and simple sampling procedures.
- Efficient sampling method for collecting particles from various surfaces with heavy loading.
- A variety of optical and electron microscopy methods can be used in the identification analysis.
- Confirmatory identification of aciniform soot by Transmission Electron Microscopy (TEM), as mandated in ASTM D6602-13, can be applied using the drop-mount technique.

- Particle dispersion techniques for breaking up the agglomerates enables more accurate identification of individual grains, necessary when environmental interferences are suspected.
- Chemical analysis of organic compounds associated with the fire deposits through bulk spectroscopy and/or chromatography (such as PAH's) can be applied.
- Corrosivity analysis via pH measurement or anions scan by Ion Chromatography can be applied.

Disadvantages

- Poor efficiency on surfaces with light loading.
- It does not preserve the relative positions of the particles on the original surface and the population per unit area; however, this is a limitation only when the agglomerate size and the distribution over the collection surface is of interest.
- Can induce damage to brittle particles such as char and ash.

Analytical Options

Based on the identity and characteristics of the particulate components present in fire debris, the main analytical techniques used for identification are:

- **Light Microscopy**
  Stereomicroscopy
  Reflected Light Microscopy
  Polarized Light Microscopy

- **Transmission Electron Microscopy with Energy Dispersive X-Ray**
  (TEM/EDX)

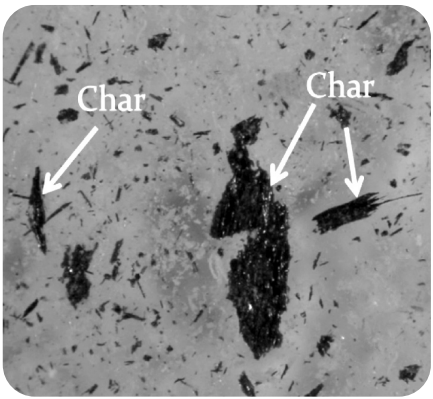
- **Scanning Electron Microscopy with Energy Dispersive X-Ray**
  (SEM/EDX)

Each of these techniques provides the means to observe and characterize specific fingerprinting traits of the target analytes in fire residues.

Stereomicroscopy

- It provides relatively low magnification observation and it gives the possibility of three dimensional observation of the components in the sample due to focusing on the same point from two different angles.
- It is used for initial examination of surface and bulk samples to observe the overall aspect of the components and the presence of large particulate.
- During the analysis by Stereomicroscope, the sample is analyzed to observe the characteristics of the particles such as color, size range, morphology, and evidence of cellular morphology of char and ash.

Image of Stereo Microscope

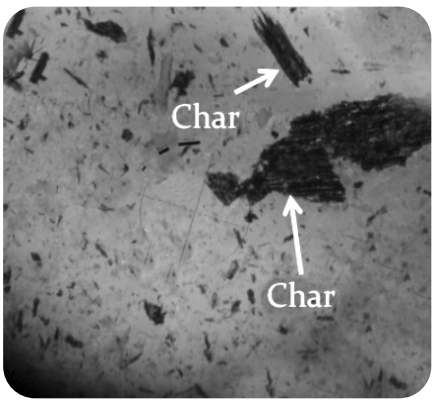


Stereo Image of Wood Char

Reflected Light Microscopy (RLM)

- Primarily used to examine opaque particles: metals, coal/coke, wood, slag, rock, plastics, alloys, composites, char.
- It is also used for initial examination of surface and bulk samples to observe the overall aspect of the components and the presence of large particulate.
- Similar to Stereomicroscope, the analysis by RLM is also used to observe the color, size range, morphology and evidence of cellular morphology of char and ash.

Image of Reflected Light Microscope

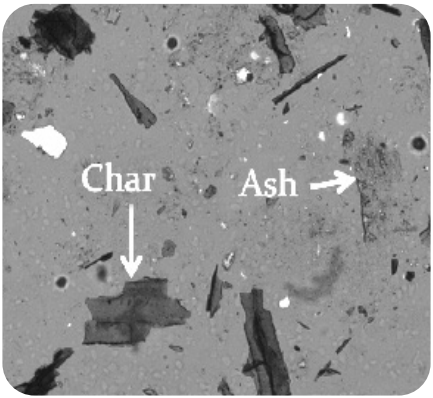


RLM image of Wood Char

Polarized Light Microscopy (PLM)

- Light microscopy technique involving observation with polarized light.
- Polarized light is a contrast-enhancing technique that improves the quality of the image obtained with birefringent materials.
- Identification is based on morphology, sign of elongation, birefringence, pleochroism, angle of extinction, and refractive index.
- The PLM technique is used for full identification of char and ash and screening/presumptive analysis of soot clusters.

Image of the Polarized Light Microscope (PLM)

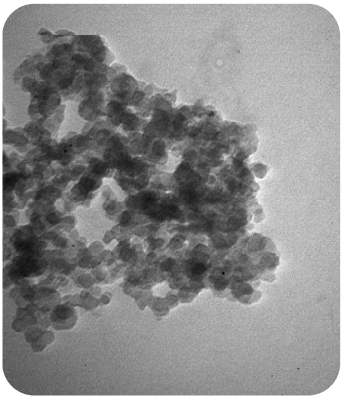


PLM image of Wood Char and Ash

Transmission Electron Microscopy with Energy Dispersive X-Ray (TEM/EDX)

- High magnification electron microscopy technique using a beam of high energy electrons transmitted through the specimen to create an image; the additional X-rays produced in the interaction are used to create elemental spectrum.
- This test method is a mandatory evaluation of soot/black carbon present in the sample because the high magnification allows determining if the morphology is consistent with grape-like or branchlike structures, the size range of individual particles and the elemental composition (with the EDX attachment).

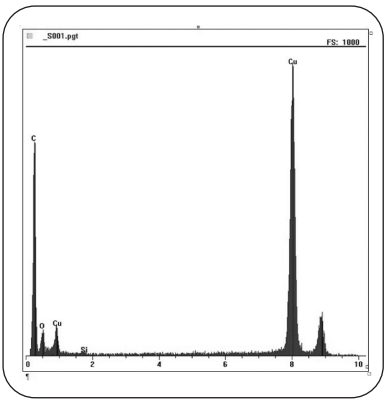
Image of TEM/EDX Instrument



TEM Image of Soot/Black Carbon; The Size of Individual Particles is ~50 nm

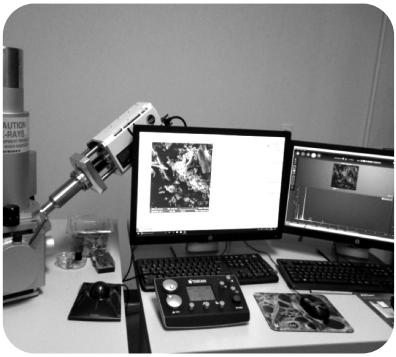
Scanning Electron Microscopy with Energy Dispersive X-Ray (SEM/EDX)

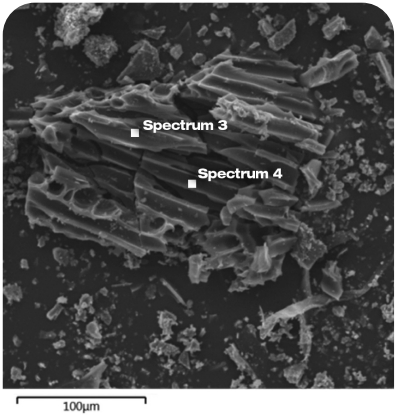
- Electron microscopy technique using a beam of high energy electrons to bombard the specimen and extract secondary electrons from the specimen to create an image; the additional X-rays produced in the interaction are used to created elemental spectrum.
- This method is used to obtain morphological details and elemental composition of char and ash; it can also be used for screening/ presumptive analysis of soot clusters.



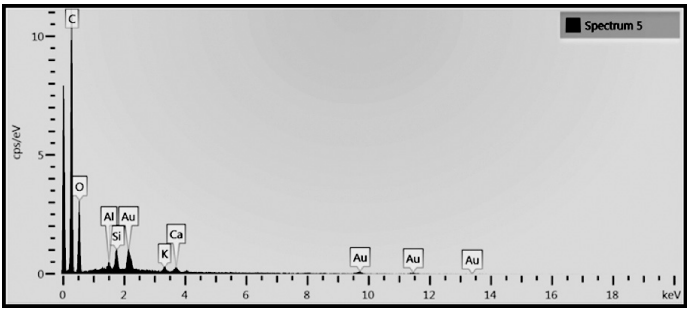
TEM/EDX Spectrum of Soot/Black Carbon; Copper Signal is Due to the TEM Copper Grid used for Mounting

Image of SEM/EDX Instrument

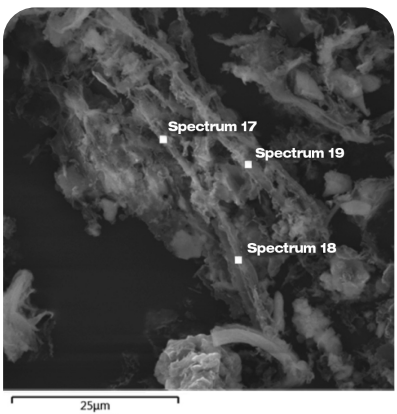




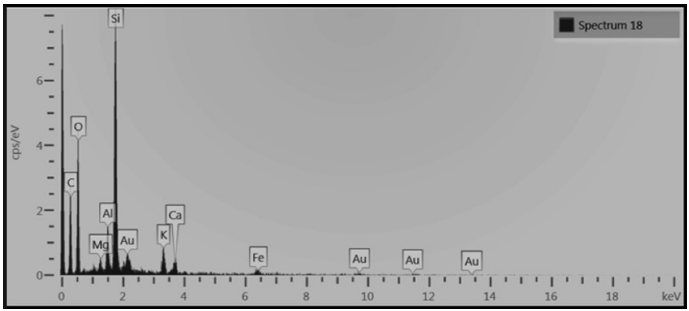
SEM Image of Wood Char



SEM/EDX Spectrum of Wood Char



SEM Image of Wood Ash



SEM/EDX Spectrum of Wood Ash

Methods for Deriving Concentrations

Air sample by NIOSH 5000

Gravimetric methods, similar to NIOSH 0500/0600 methods
Concentration, C (mg/m3) in the air volume sampled, V (L):

$$C = \frac{(W2 - W1) - (B2 - B1)}{V} .x10^3$$

Where

W1 = Tare weight of filter before sampling (mg)
W2 = Post-sampling weight of sample-containing filter (mg)
B1 = Tare weight of blank filter (mg)
B2 = Post-sampling weight of blank filter (mg)

Air sample with MCE or Air-o-Cell cassettes

Particle Count Method

- Results expressed as "number of particles/volume of air".
- Method does not take into consideration the particle size.

Surface or bulk/grab sample

A. Visual Area Estimation Method (defined in EPA 600/R-93/116)

- It estimates the relative projected area of a certain type of particulate from a mixture of particulate by comparison to data derived from analysis of calibration materials having similar texture and particulate content.

B. Point Count Method

- Results expressed as "%" area.
- The fraction of points within a rectangular grid superimposed on a two-dimensional planar microscopic field provides an estimate for the volume fraction of identifiable constituents.
- Method does take into consideration the particle size.

Recommendation

An effective interpretation of the results is based on the comparison with unaffected areas. If possible, obtain a control sample from an area not affected by fire to be used as background.

Analytical Packages ***(surfaces and bulk sampling)***

Level Provided by EMSL Analytical, Inc.	Analytes of Interest included in the Analysis	Analytical Techniques Applied for Analysis
Screening	• Char • Ash • Soot/Black Carbon (Presumptive/Screening)	• Light Microscopy (Stereo, RLM, PLM) • Presence/Absence; No Concentrations Reported
Level 1	• Char • Ash • Soot/Black Carbon (Presumptive/Screening)	• Light Microscopy (Stereo, RLM, PLM) • Concentrations of Analytes are Reported

Analytical Packages *(surfaces and bulk sampling)*

Level Provided by EMSL Analytical, Inc.	Analytes of Interest included in the Analysis	Analytical Techniques Applied for Analysis
Level 2	• Char • Ash • Soot/Black Carbon (confirmatory)	• Light Microscopy (Stereo, RLM, PLM) • TEM/EDX (for Soot/Black Carbon) • Concentrations of Analytes are Reported
Level 3	• Char • Ash • Soot/Black Carbon • Common Components Of Environmental Dust: MMVF's, Biological Components (Pollen, Mold), Cellulose, Synthetic Fibers, Quartz, Calcite	• Light Microscopy (Stereo, RLM, PLM) • TEM/EDX (for Soot/Black Carbon) • SEM/EDX (for Overall Elemental Composition of The Sample) • Concentrations of Analytes Are Reported
Level 4	• Char • Ash • Soot/Black Carbon • Common Components of Environmental Dust: MMVF's, Biological Components (Pollen, Mold), Cellulose, Synthetic Fibers, Quartz, Calcite pH	• Light Microscopy (Stereo, RLM, PLM) • TEM/EDX (for Soot/Black Carbon) • SEM/EDX (for Elemental Composition of Soot, Char, and Ash) • Concentrations of Analytes are Reported • Value of pH and Comparison with Blanks is Reported

Additional Information

Recommendation
An effective interpretation of the results is based on the comparison with unaffected areas. If possible, obtain a control sample from an area not affected by fire to be used as background.

Soot Source Identification

- Analysis for presence of residual products consumed in fires by Gas Chromatography-Mass Spectroscopy/GC-MS (such as aliphatic hydrocarbons from fuels, phthalates from plastics, paraffin from candles, Fatty Acids from edible oils, Levoglucosan from carbohydrates/cellulose).
- Residual sulfur associated with Methyl Mercaptan, the olfactive indicator from natural gas.
- Protein fires: analysis for Tryptophan and /or Albumin by Liquid Chromatography - Tandem Mass Spectrometry (LC-MS/MS).

Corrosion propensity of fire residues when resting on metal surfaces

- pH measurement.
- Anions scan associated with non-volatile acids, determined by Ion Chromatography/IC (chloride, fluoride, bromide, nitrate, nitrite, phosphate, and sulfate).

Metallic Elements in fire residues

- Analysis by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) for target elements, including heavy metals.

Aldehydes and Volatile Organic Compounds (VOCs)

- Compounds associated with odors and toxic organics.
- TO-15 analysis of air collected in specially-prepared canisters and analyzed by GC/MS.
- Aldehyde profile by HPLC (based on NIOSH 2016 method).

IESO/RIA Standard 6001

- Char is used as the primary indicator.
- Soot is used as the secondary indicator.
- It is a screening method.
- The method is not designed to provide identification of individual char particles nor to determine the origin of soot particles.

References:

ASTM D6602-13-Standard Practice for Sampling and Testing of Possible Carbon Black Fugitive Emissions or Other Environmental Particulate, or Both

EPA 600/R-93/116- Method for the Determination of Asbestos In Bulk Building Materials

IESO/RIA Standard 6001-2011-Evaluation of HVAC Interior Surfaces to Determine the Impact from Fire-related Particulate

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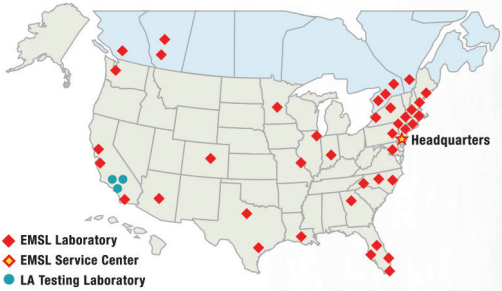
866.798.1089

United States and Canada Locations



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