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Laboratory Methods for Wildfire Impact Assessment

Good Day and welcome to IAQ Radio+ episode 768 blog. This week we welcomed Dr. Eugenia Mirica to discuss Laboratory Methods for Wildfire Impact Assessment and some current events around structural fires and wildfires including her candid thoughts on the new AIHA Wildfire Guidance and ASTM D6602.

Eugenia Mirica, PhD is the Laboratory Director of the Materials Science Laboratory at EMSL Analytical, Inc in Cinnaminson, NJ. Eugenia received her Ph.D. in Materials Science from Stevens Institute of Technology in 2002. She joined EMSL at the end of 2002 and she has been with the company ever since. Her expertise involves complex analyses employing a large variety of analytical techniques, utilized for the identification and the comprehensive characterization of various types of materials.

Nuggets mined from today's episode:

Eugenia's Immigration and Career Journey: Eugenia emigrated from Romania to the US in pursuit of higher education. She has been employed at EMSL for 23 years where she started as an analyst worked her way up to her current role as lab director. She is hands-on and loves doing analysis at EMSL where she gets to utilize a wide and varied range of analytical equipment.

Wildfire Impact Assessment Laboratory Methods Dr. Mirica discussed laboratory methods for wildfire impact assessment. She explained that the business of analyzing wildfire impact has grown significantly since 2017, with EMSL seeking lab accreditation in multiple locations including Canada, Cinnaminson, Pasadena, Florida, and Denver. Eugenia has presented on target analytes, sampling methods, analytical instrumentation, and data reporting, referencing standards such as AIHA Technical Guide for Wildfire Impact Assessment. The discussion highlighted the importance of analyzing char, which are the main components of wildfire residue, and preserving its original cellular morphology for accurate analysis.

Wildfire Residue Analysis Techniques:

Eugenia explained the identification and analysis of wildfire residues, focusing on char, ash, and soot. She described the characteristics and differentiation methods for these analytes, emphasizing the need for specialized training and equipment. The discussion also covered the ASTM standard D6602 for differentiating carbon black from soot, and the importance of proper sampling locations for analyzing wildfire residues in residential settings.

Char- is obtained by carbonization (chemical process of transformation of an organic substance) by means of pyrolysis in a residue with carbon as the main components. ASTM D6602 definition of char- particle layer, I µm- may preserve original cellular morphology of the material that was combusted.

In microscopy it's important to know the morphology. Char is easiest to determine with the right optical microscope. Ash is the result of higher heat over longer duration. Ash may not present with any original

morphology. Left with mostly minerals ash is not easy to determine; training and the right equipment are needed. Sometimes it's hard to distinguish between ash and something from the environment.

Instrumentation (the fun part)

Light microscopy is available at all labs. Scanning Electron Microscopy/Energy Dispersive Xray (SEM/EDX). Transmission Electron Microscopy/ Energy Dispersion Xray (TEM-EDX). Light microscopy- stereo microscope 60x magnification possible 3D used for screening. Determines presence of large samples. Simultaneous transmitted light Microscopy (PLM) and Darkfield Reflective Light Microscopy (DRLM) 500x magnification. DLRM- high degree of contrast making it easy to distinguish ash, metal dust/rust. OLM-morphology signs of elongation, birefringence, pleochroism, angle of extinction and refractive index.

Authors of AIHA wildfire document pioneered the PLM/DLRM method. It's a good method, by switching between modes analyst can see more things and helps distinguishment. Used for every single project.

SEM/EDX microscope uses electrons to collect image. EDX is used on all fire projects analyzed by EMSL. Provides a more sophisticated analysis compared with local references. Compares morphology & other chemicals calcium, metals, minerals, etc. + elemental analysis.

Tip for dealing with a lab: Tell the lab what you want to determine and ask what options do you have?

Fire Scene Particle Sampling Methods

Sampling for wildfires should begin on the exterior of the building (exterior windowsills, ledges, covered porches, HVAC intake, etc.).

Interior sampling: look for infiltration patterns- living areas, door tracks, electronics. Furniture, corners of rooms, attics, etc.)

Eugenia discussed sampling methods for analyzing particles from fire and wildfire scenes, focusing on the efficiency and representativeness of different sampling media. She explained the advantages and disadvantages of tape lifts, wipes, and micro-vacuuming, noting that tape lifts are currently the most used method due to their efficiency and availability. Eugenia emphasized the importance of selecting the appropriate sampling method based on the surface type and loading and highlighted that micro-vacuuming is particularly effective for collecting particles from porous, uneven surfaces with medium to heavy loading.

In order to obtain the best picture of a project, Eugenia recommends that multiple sampling methods be used. AIHA wildfire document prefers tape lifts and opines that tape lifts must be used.

Tape Sampling Pros

Efficient for smooth nonporous with monolayer loading preserved. By preserving monolayer loading preserves the original positioning of particles and the particle population of the area. Can be analyzed by a variety of optical methods with minimal preparation.

Tape Lifts: Premade 2”x3” tape lifts are available. Office tapes can be used either transparent or opaque. Don’t fold sample over itself. Don’t use packing tape.

Tape Sampling Cons

Poor efficiency of collection on porous surfaces. Pressure can damage brittle particles. Limited sampling area. Overlapping challenges. Cannot

use them to confirm soot. Chemical analysis is impossible. pH measurement cannot be made. SEM/EDX tape overloading- multiple layers on top, Hard to mount with oil.

Wet Wiping Pros

Works for any amount of loading. ASTM D6602-13 can be applied during drop mount technique particle dispersion techniques for breaking up agglomerates. May enable more accurate ID of individual grains, needed when environment interference is suspected, chemical analysis through bulk spectroscopy or chromatography such as PAHs can be applied, analysis via pH measurement or anion scans can be applied. Sonication can be used.

Wet Wiping Cons

Wipe sampling: dry or moist media may be used. Dry wipes do not hold particles. Alcohol wipes are available in varying concentrations of alcohol.

Poor efficiency for collecting on porous, uneven surfaces, doesn't preserve relational positioning of particles on original surface per unit area. 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.

Micro-vacuuming Pros

Efficient collection on porous, uneven surfaces with medium to heavy loading. Sample represents bulk amount of particle material, often many different sizes. Variety of optical & electron microscopy methods can be used for ID. Chemical analysis of organic compounds associated with fire deposits through bulk spectroscopy and/or chromatography (such as PAHs can be applied). TEM confirmation of actiniform soot indicated in ASTM D6602-13 can be applied using drop mount technique. Corrosivity analysis via pH measurement or anion scan by ion chromatography such as PAHs can be applied.

Micro-vacuuming Cons

Poor efficiency for collecting particles from relatively smooth surfaces with low loading. Does not preserve relative positioning of particles on original surface & population per unit area. Can damage brittle particles char/ash. Suspect contamination on clothing: determine multiple methods micro-vacuum extracts particles from fibers. Micro-vacuuming-need pump and tubing. Any filter cassette can be used. For sampling a soiled area 10cm x 10cm is recommended, for clean surfaces 1’ x 1’ is recommended. Is the sample representative of the area in question? Sample results are reported by visual concentration and are not surface related.

Sampling & analysis considerations.

- What are the target analytes?
- sampling methods
- analytical instrumentation
- analytical results
- interpretation of data

Microscopy Techniques in Laboratory Analysis

Eugenia presented a detailed overview of various microscopy techniques used in laboratory analysis, explaining their applications and limitations. She highlighted the importance of choosing the appropriate method based on the sample and the desired level of detail. She also discussed the use of scanning electron microscopy with energy dispersive X-ray (SEM/EDX) analysis for more advanced analysis, including the identification of elemental composition in samples like char, ash, and soot. The presentation emphasized the need for laboratories to offer a range of analytical tools to meet different project requirements.

Advantages of TEM for Soot Analysis

Eugenia presented a detailed comparison of different analytical instruments used to identify soot particles, highlighting the superiority of Transmission Electron Microscopy (TEM) over other methods like PLM (Polarized Light Microscopy) and SEM in providing definitive soot identification. She expressed concerns about inaccurate descriptions of soot behavior in the AIHA Second Edition, which she was not invited to review, and emphasized the importance of evidence-based scientific writing. The discussion concluded with a demonstration of how PLM can

misleadingly show similar appearances for samples with different compositions, underscoring the need for accurate identification methods like TEM.

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

Carbon black has numerous industrial uses and is manufactured with varying specified parameters depending on use. Carbon black has consistent characteristics while soot does not. ASTM D6602-13 Carbon black's morphology is uniformly sized particles (nano range of 10-100 µm) resembling clusters of grapes.

Eugenia discussed the use of ASTM D6602-13 for analyzing wildfire and structure fire residues, emphasizing that while it should not be used as a standalone method, it contains valuable elements that can be applied in such analyses. She highlighted the standard's definitions, visual estimation methods, and its ability to differentiate between carbon black and soot. Eugenia also criticized the AIHA Technical Guide's approach to data interpretation, describing it as complex and not user-friendly, suggesting that alternative methods might be more suitable for practical applications.

Soot from wildfires/structural fires is easily distinguished from other sources (e.g. candles, gas appliances, cooking, etc.)

Wildfire Residue Sampling Challenges

Eugenia discussed the complexities of sampling and analyzing wildfire residues, emphasizing the need for site-specific approaches and collaboration with laboratories. She highlighted the challenges in interpreting data and the importance of considering factors such as pH, heavy metals, and organic compounds. The discussion also touched on the use of composite sampling, with Eugenia noting its limitations and the need for careful application.

Z-Man's Questions: ***Are there similarities between wildfire particles (e.g. ash) and mold spores/fungal fragments?*** The only similarity could be that, with naked eyes, some wildfire components (fine char, soot) and mold could have a dark aspect.

What is your opinion on the use of Composite Wipe Sampling following wildfires? Best results are obtained with best samples. Analytically, in the lab, we can composite wipes, depending on the size of the wipes that were used and the particle loading. As far as composite sampling, it is the site investigator that decides if such approach is applicable for the project. It works best if similar types of surfaces are sampled, surfaces exposed to similar conditions, with similar particle loading. It is not an appropriate approach if the surfaces are vastly different.

Does obtaining agreement solely from similarly minded colleagues, while dismissing dissenting views from equally or more experienced experts, justify presenting this selective finding as "THE SCIENCE"? *This* would be a controversial discussion. What is science? There are many definitions for science. I prefer this one: science is a method for understanding the world by using observation and experimentation to test ideas and gather reliable knowledge. Science is expected to fundamentally be an objective inquiry; it requires accepting all the available evidence,

especially the ones that challenge existing ideas. Moreover, science is never a place for personal preference or personal bias.

In situations where there is an abundance of visual and olfactory evidence that wildfire residues are present, Can the presence or absence of wildfire be confirmed visually, without the use of tape-lift sampling and microscopic analysis? If the particles on surface are large enough, yes, presence of fire residues can be confirmed visually. The actual confirmation to determine if the residue is from wildfire, pit fire or wood fireplace does require a more advanced, forensic -type approach that requires microscopy and instrumentation able to obtain elemental composition, or other type of chemical fingerprint.

How does the process of sonication work? Sonication uses high frequency sound waves to create high-energy changes in the liquid. When applied to a liquid such as isopropanol where a wipe or the sponge is submerged, it dislodges the particles collected within the fibers or the pores and transfers them in suspension, offering the ability to collect them via filtration.

What in the revised AIHA Wildfire Document gives you heartburn and your recommendations for fixing it? A few things:
- some scientific inaccuracies regarding the solubility of particles when using wet wipes for sampling.
- the marketing of “assemblage analysis”, which is provided by this terminology only at the labs owned by some of the writers of the document. This has been a conflict of interest from the inception of this document.
- the very confusing chapter 5, results interpretation, which shows a statistical approach of looking at the data and using the results for making site restoration and cleaning decisions based on this approach; the annex which shows an actual example of how to use this approach is even more confusing.

- The removal of ASTM D6602 from the refence section, although it was present in the first edition. Since most of the information from the first edition is carried in the second, the removal seems political and far from objective, only to align the document with the earlier efforts of the same group of authors to discredit the usefulness of any part of the ASTM method when analyzing fire debris. It is wrong, but it was done.
- AIHA document opines that: methods must have appropriate limit of quantification for meaningful interpretation to allow for comparison with health-based criteria. Eugenia disagrees that relevant health-based criteria exist.

What can be done? I am not sure how AIHA could be approached about this. The Synergist accepts rebuttals for the articles they publish. But the revision of the Wildfire technical guide 2nd edition was not announced publicly. I was a reviewer for the first edition, since EMSL is one of the large labs that has been offering this analysis for a long time. I actively participated then, and we did have antagonistic discussions about the analytical approach. The group for the second edition only accepted people who aligned with only their analytical views, they did not want dissent any longer. It seems there is no public commenting forum available for AIHA documents, as there is for IICRC standards. Hence, a document that still has inaccuracies and personal views was produced.
Soot nanoparticles size range agglomerates morphology-

TEM/EDX is the most advanced technology allowing the analyst to see particles in nanometer ranges. These particles are it seems then run against the labs known standards for comparison. Sulphur mercaptans indicate particles originating from a petroleum source.

- PLM reports will indicate presumptive
- SEM reports will indicate presumptive
- TEM/EDX very clearly confirms, for sure.

To demonstrate the importance of instrumentation Eugenia showed a side-by-side comparison of 2 technologies, the lesser technology mistakenly identified the suspect particles as “soot” when superior technology was able to confirm the suspect parties were iron oxide.

Data interpretation- there are no recognized numerical values or generally accepted consensus regarding the concentrations associated with contamination or damage.

- Presence/absence is a radical approach. Visual estimation. VAE method LOQ=1% limit of quantification. For counting 1 particle in the entire sample.
- Cannot work with mean/average values. Battle-lines compare data Zone-to-Zone, contaminated or not contaminated, or contaminated with background.
- Labs don’t interpret data because the data is site related.
  TIP- When you don’t understand the data, request the lab to provide the analysis in a more user-friendly version.

ASTM D4972-13 Standard Test Method for pH of Soils

-Formaldehyde, acrolein, creosols, guaiacols, syringols, levoglucosan
-PAHs, dioxin, furans, nitrosamines.
-Heavy metals-debris as- As, Pb. Cd, Hg, Cr, Zn
-LIB- lithium batteries- Li, Co, Ni, Mn, P fluoride

Sampling and analysis considerations:

- What burned?
- Distance from fire?
- Level of contamination?

The lab needs to know what their client needs to know or want to test for?

- Correlate sampling methodologies/media with the complete scope of testing by discussing the project with your lab of choice:
- select the most appropriate analytical package for the scope
- Interpret results based on the event and background info.

Current technical resources:

- ASTM 6602-13 carbon black standard.
- AIHA Tech Guide for Wildfire Impact Assessments for the OEHS Professional 2018 1st edition/2025 is the revision.
- IESO/RIA 6001- is narrow and applies only to HVAC systems. Char is primary indicator and soot is the secondary indicator.

Z-Man signing off