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oil were also collected for comparison.

Results: Several sites were characterized. Coconut shell charcoal tube sample results for most of the sites were essentially non-detects. However one sampled site (synthetic charcoal) had significant findings for the headspace sample and lower results for the personal air samples. All of the bulk samples gave similar results for C_3-C_{20} hydrocarbons. Correlation of the field portable GC/MS results and the lab analyses showed good qualitative agreement. Quantitation was performed on 12 C_5-C_7 hydrocarbon solvent analytes. These analytes combined to produce bulk concentrations between 7–12%, headspace between 10,000-15,700 ppm, and personal between 0-1,500 ppm.

Lessons Learned: Exposure assessment for Peak and IDLH situations using a bag to collect instantaneous samples and either immediate analysis or stabilization to a sorbent tube can be a useful technique. Using field portable GC/MS to characterize a site can help determine what type of samples to collect showing that in this case light C_3-C_7 hydrocarbons should be characterized. Synthetic and coconut shell charcoal tubes do not retain propane and butane as desired. Even though the transfer from the bag to the tube passed 0.5 liter, significant breakthrough was observed on charcoal tubes when analytes were present in high concentrations.

CS-404-04
An Evaluation of a Quantitative X-ray Diffraction Method for Analysis of Asbestos-Containing Materials

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Situation/Problem: The use of appropriate test methods to identify and quantify asbestos in bulk samples is critical to ensure the best decisions may be made to protect human health. Although polarized light microscopy (PLM) is widely accepted in North America and Europe as the standard analysis method for bulk asbestos, powder X-ray diffraction (XRD) is also a useful tool for asbestos analysis. In the United States, XRD is included as a complement to PLM in the Environmental Protection Agency's 1993 Method for the Determination of Asbestos in Bulk Building Materials. XRD is limited, however, by potential interferences from components commonly found in bulk building materials and may not be able to achieve a detection limit low enough to identify asbestos at or near 1% in all bulk samples.

Resolution: A quantitative XRD method was developed for the determination of asbestos in bulk materials. This standard additions method differs from the internal standard method outlined in the 1993 EPA test method. Powder mixtures of known composition were used for preliminary evaluation of the method. Both the method and the sample preparation techniques used to remove matrix interferences were further refined in routine use of XRD for the characterization of "real-world" materials used as proficiency testing materials.

Results: Analysis of formulated mixtures of known composition indicates that the XRD method has good weight composition accuracy and precision, with a detection limit approaching 1% or even lower. Matrix interferences challenge application of the method to complex real-world materials in which there is peak overlap. However, judicious application of gravimetric sample reduction techniques greatly enhances the method's ability to quantify asbestos at low levels in many real-world materials.

Lessons Learned: The successful application of quantitative powder XRD to bulk asbestos analysis is sample-dependent. Method limitations include potential matrix interferences and the inability to differentiate between fibrous and nonfibrous analogs. When both fibrous and nonfibrous mineral forms occur in one sample, the presence of the nonfibrous component hampers quantitation of the fibrous component by XRD. XRD may be most effective as a complement and confirmation to microscopy techniques, such as visual estimation, that are subjectively dependent on analyst training and experience.

SR-404-05
Comparison of the Standard and Dark-Medium Objective Lens in Counting Asbestos Fibers by Phase-Contrast Microscopy

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Objective: A Japanese round-robin (RR) study revealed that the analysts who used a dark-medium (DM) objective lens reported higher fiber counts of Proficiency Analytical Testing (PAT) chrysotile samples than those with a standard objective lens but no causes of such differences were investigated. The purpose of this study was to determine the major sources of the differences of fiber counts between two objective lens types by performing two sets of RR studies.

Methods: For the first RR study, 15 proficiency test sample filters (five each of chrysotile and Amosite generated by water-suspended method and five chrysotile generated by aerosolization method) were purchased from the American Industrial Hygiene Association (AIHA) and slides were prepared with relocatable cover slips. Nine labs volunteered to participate in the exercise. A single microscope with both standard and DM objectives was circulated to each laboratory along with the prepared slides. A second RR study was then performed with six chrysotile field sample slides. Six out of nine labs who participated in the first RR study participated. Additionally, an 8-form diatom test slide was examined by eight analysts to compare resolutions between two objectives.

Results: For the PAT chrysotile reference slides, use of the DM objective resulted in consistently higher fiber counts (1.45 times for all data) than the standard objective (p-values: < 0.05), regardless of the filter generation (water-suspension or aerosol) method. For the PAT Amosite and chrysotile field sample slides, the fiber counts between the two objectives were not significantly different. There was no differences of resolution from the 8-form diatom test slide examination.

Conclusions: The findings support the contrast caused by the different phase plate absorption between the two objectives as the main factor affecting high number of PAT chrysotile fiber counts using the DM objective. The chrysotile fibers in the PAT samples are thinner than the airborne contaminant fibers collected from the field. If the thin PAT fibers are not generally representative of field samples, it is not necessary to recommend the DM objective be used for routine fiber-counting. However, the DM objective does allow more very fine fibers to be counted and may provide counts of those fine fibers, if present, similar to that achievable with an electron microscope.