2IM.3

Analytical Method for Quantification of Airborne Respirable Crystalline Silica Using Raman Spectroscopy. VASILEIA VOGIAZI, Chen Wang, Pramod Kulkarni, *Centers for Disease Control and Prevention, NIOSH*

Inhalation exposure to crystalline silica aerosol poses significant health risks in many workplaces. There is a need for laboratory analytical methods as well field-portable methods for onsite analysis of trace level exposures, capable of measuring a few µg/m3. To address these needs, a standardized method using a portable probe-based Raman and an analytical confocal Raman microscope has been developed for the measurement of trace quantities of airborne respirable crystalline silica (RCS). This method is particularly useful to quantify trace level RCS exposures that can't be achieved with X-ray Diffraction (XRD) or Infrared (IR) methods. The method implements a microconcentration technique with point-measurement as well as a rastering scheme that covers a large sample spot area and provides detection limits much below the RCS exposure limit of 50 µg/m3. The method can quantify both α-quartz and cristobalite polymorphs in the range of 2 (± 0.1) - 50 (±2.8) µg per filter using point-measurement with a field-portable probe-based Raman spectrometer. The relative standard deviation of the probe-based method is in the range of 1-11%. Using a rastering scheme with a confocal Raman microscope the limit of detection as low as 0.1 µg could be achieved. Relative standard deviations of triplicate measurements were in the range 10-23% and the limit of quantification was estimated to be 0.5 (± 0.08) µg RCS per filter. The uncertainty of the method was better than that of the XRD method (NIOSH method 7500). The potential interferent materials, such as iron oxide and TiO2, can affect the quantification and measurement uncertainty, particularly at trace concentrations of RCS. The method is currently being extended to quantify RCS in field samples; the results will be compared with XRD measurements.

2IM.4

A Data Analysis Pipeline for Identification of Untargeted GC-EI-MS Spectra. DEBORAH F. MCGLYNN, Lindsay Yee, Lewis Geer, Yuri Mirokhin, Dmitrii Tchekhovskoi, Coty Jen, Allen Goldstein, Anthony J. Kearsley, Stephen E. Stein, *National Institute of Standards and Technology*

Despite the thousands of compounds represented in mass spectral (MS) libraries, a large fraction of spectra in complex mixtures cannot be identified. Environmentally sampled compounds pose a special challenge, as these compounds are commonly subjected to complex chemical reactions such as oxidation or pyrolysis. While matching chromatographic retention index data improves the confidence of any spectral identification, this information is not always available and typically requires significant manual effort to be incorporated in the analysis. In this work, we present a search method that considers MS similarity, retention indices, and molecular mass when scoring library matches. When retention indices are not available, AI-estimated values are provided. The new method was applied to a MS dataset containing 4833 Trimethylsilyl (TMS)-derivatized spectra collected by the University of California at Berkeley of particulate organic compounds emitted by wildland fires. The dataset was run against the NIST 2023 EI-MS dataset using the identity search method with retention index penalization. The RI threshold window used was ± 25 between the unknown and the library spectra. Based on NIST23 library matching, this analysis led to 181 new identifications in the dataset. 105 identities from previous work were confirmed while 34 previous IDs were changed. Following this, estimations of molecular mass were made for MS with high signal to noise ratios. This allowed the library-based 'hybrid' search method to be used to identify compounds similar to, but not present in, libraries. These new methods increase reproducibility, reduce analysis time, and increase identification frequency in these complex mixtures. Since the number of remaining unidentified spectra remains large (4530 spectra), this work concludes by identifying which spectra are most likely to be identified by additions to libraries or further analysis, and which are most likely to remain unidentifiable due to possible contamination or low signal strength.