Forensic analysis of surface fallout from low yield surface nuclear tests

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Five decades after the last US atmospheric test, fallout samples from low yield surface and near surface events are being re-examined for their forensic value using a combination of radiometric (counting) and mass spectrometry based techniques. Preliminary data from these studies indicate that soil samples collected along historical fallout plumes contain easily accessible information about key components of each test such as fuel isotopics and the elemental composition of structural components.

The distribution of fuel, activation product and fission product signatures as a function of soil grain size and distance from ground zero has been found to be nearly uniform among larger grain sizes of fallout soil (> 0.1 mm). Anthropogenic glasses extracted from the fallout soil samples constitute <5% of the bulk soil mass, yet contain >50% of the total activity. Order of magnitude increases in the concentrations of actinides such as ^{235}U or ^{239}Pu are observed in the glass relative to the soil. By gamma spectroscopy ^{235}U concentrations are estimated to be >60 μg/g and ^{239}Pu concentrations >20 μg/g in glasses recovered from two different tests. Preliminary analysis of the U bearing glasses by ICP-MS indicates a fuel with a minimum enrichment of 84% ^{235}U. Analysis of the stable isotope signal from ICP-MS measurements shows that Be is present in the glass at concentrations 4 times higher then the bulk soil, and that Cr, Co and Mo are present in the U bearing glasses at 3-20 times the bulk soil concentration.

These results demonstrate that fallout from low yield surface nuclear tests collected as surface deposits long after the detonation contains valuable information about device characteristics. This information is relevant to characterizing historical activities from surface based nuclear test programs and is applicable to nuclear forensics research. This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. LLNL-ABS-480552

Rapid esterifications for compound-specific stable isotope analysis of fatty acids

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Fatty acids are frequently found as abundant lipid molecules in biological and geological samples, and therefore have been employed as biomarkers in a number of studies particularly for organic geochemistry. However, to reduce polarity and enhance volatility of fatty acids, and resultingly to improve the shape and resolution of fatty acid peaks on gas chromatograms, derivatization such as esterification or silylation are generally required for identification and quantification as well as compound-specific stable isotope analysis (CSIA) of fatty acids.

Here we evaluate methyl (MCF) and ethyl chloroformate (ECF) derivatizations as a rapid and simple esterification for CSIA of fatty acids. These derivatizations are generally prepared with an admixture of MCF/H_2O/methanol/pyridine or ECF/H_2O/ethanol/pyridine at room temperature for 5 min (Fig. 1). In this study, we used 0.1M HCl aq., H_2O, 0.1M NaOH aq., or organic alcohol (methanol or ethanol) instead of H_2O to evaluate the efficiency of derivatization.

For both MCF and ECF derivatizations, fatty acids are esterified very rapidly (within 5 min) even at room temperature, and accuracy of the carbon isotope measurements is always less than 0.3‰ (1σ). Although the yield of derivatives would depend on the chemical constituents and pH of derivative reagents as well as on the carbon-chain length of fatty acids, quantitative esterification is observed by using ECF/ethanol/pyridine (2/60/5, v/v). Thus we conclude that these derivatizations are potentially suitable as a rapid and simple esterification tool for CSIA of fatty acids.

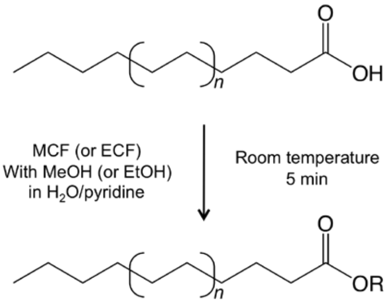

Figure 1: MCF or ECF derivatization of fatty acids.