

## **Current state-of-the-practice of applied forensic geochemistry within the environmental engineering/consulting community**

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Many environmental engineering/consulting companies now routinely conduct forensic geochemical investigations previously provided almost exclusively by academia. Such investigations include trace element and stable isotope analyses of soil/groundwater, fingerprinting of fuel and other hydrocarbon releases, and age dating contaminant releases and groundwater using stable and radioactive isotopes.

Trace and heavy elements and their respective isotopes are used to track surface and groundwater contaminant plumes; e.g., boron and  $\delta^{11}\text{B}$  can track municipal solid waste landfill leachate and rare earth elements have been used to track refinery effluent and sediment as well as waste soil dumped into rivers.

For more than a decade fuel release sources have successfully been identified by chromatography/mass spectrometry chromatogram pattern recognition. Polycyclic aromatic hydrocarbon (PAH) analysis and biomarker signature analysis have been used to determine diesel release age dates. Chlorinated volatile organic compound releases, such as trichloroethane, have been age dated using mass balance degradation or daughter product concentrations collected over time.

Stable isotope lead analyses have identified petroleum and gasoline source signatures and release age dates using the Anthropogenic Lead Archeostratigraphy (ALAS) model of Hurst (2000). Nitrogen and oxygen stable isotopes in dissolved nitrate can identify nitrate sources; oxygen-deuterium isotopes in water can distinguish water sources; and chlorine and oxygen isotopes in dissolved perchlorate are used to determine perchlorate sources (Sturchio, 2004). Tritium, coupled with  $^3\text{He}$ , can date groundwater to within several years.

### **References**

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Sturchio, N.C. (2004) GRA 11<sup>th</sup> Ann. Sym., Glendale, CA.

## **Application of heavy stable isotopes to forensic isotope geochemistry**

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Trace element geochemical tools have been applied for many years to understand sources of materials in the geological environment and in such diverse fields as anthropology, archaeology, biology and oceanography. Further advances have been made by the application of light stable isotopes (C, N, O, S, H). The advent of multiple-collector ICP-MS in addition to thermal ionization mass spectrometry have opened up the field considerably. It is now possible to use isotopic signatures of both radiogenic and stable isotopes for many elements in the periodic table to carry out fingerprinting studies.

Many of these systems rely on processes that give rise to different isotopic compositions either as a consequence of radiogenic decay or as a result of natural isotopic fractionation. A result of this is a range of different isotope ratios that may be used as tools to examine sources.

Whilst many of the "new" isotope systems offer new tools it is important not to forget the old tools such as Sr and Pb, which in many cases offer better fingerprinting techniques. This is partly a consequence that the systematics of these new isotope systems are not completely understood and that the extraction and purification of these "new" isotope systems can be troublesome.

This paper will give an overview of some of the applications of heavy stable isotopes in the field of forensic geochemistry with examples from archaeology, anthropology, palaeobiology and environmental studies amongst others.

## Enamel biomarker for assessing and tracing heavy metal exposure

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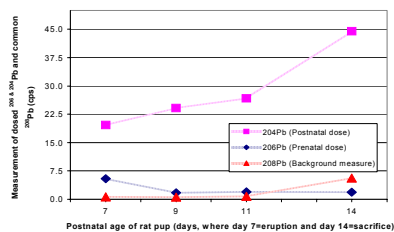
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### New Biomarker Method

We report on a new enamel biomarker for assessing and tracing heavy metal exposures with application to forensic geochemistry: The enamel crystals record a longitudinal record of ambient exposure to heavy metals, analogous to growth rate recorded in tree rings. Ion probe mass spectrometry (IMS) and laser ablation inductively coupled mass spectrometry (LAICPMS) have been used to reconstruct Pb and Mn exposures relative to Ca and Fe, respectively.

Pb isotopic tracers were used to demonstrate first principles of deposition and kinetics of Pb absorption in an animal model. Our IMS results are shown in figure below.



Instrumental neutron activation analysis (INAA) was conducted to measure Mn concentrations in target tissues in a controlled inhalation and ingestion experiment. Brain Mn was significantly associated with tooth concentrations ( $R^2=0.64$ ).

We report on strontium isotopic characterization of enamel as a geochemical tracer, having forensic applications.

## Environmental isotope forensics of perchlorate

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Widespread occurrence of perchlorate in surface water bodies and ground water aquifers has been recognized in recent years. This perchlorate, which may have both anthropogenic and natural sources, can be problematic because the highly soluble perchlorate anion is relatively inert and difficult to remove by conventional water treatment methods. Stable isotope ratios of Cl and O can potentially be used to distinguish the source(s) of perchlorate in a given location, and to evaluate the extent of biodegradation. However, few data yet exist regarding the stable isotopic composition of perchlorate from anthropogenic and natural sources. Before isotopic forensics of perchlorate can be widely applied, further isotopic characterization of perchlorate sources is necessary.

The stable isotope ratios of Cl and O in anthropogenic perchlorate salts can now be readily measured in milligram amounts, but it is difficult to extract and recover isotopically measurable amounts of perchlorate from natural waters in which perchlorate concentrations may be in the ppb range. The recent development of a new class of bifunctional anion exchange resins for efficient sorption and removal of perchlorate, along with a new resin regeneration technique for recovering sorbed perchlorate, provides a means for nearly quantitative recovery of perchlorate from water samples collected in the field for isotopic analysis.

We have begun a systematic characterization of the stable isotope ratios of Cl and O in perchlorate from different sources, starting with anthropogenic perchlorate reagents and natural perchlorate-bearing salt deposits. Preliminary results indicate that there are significant isotopic differences between anthropogenic and natural perchlorate sources, and that microbial perchlorate reduction has a large kinetic isotope effect. When the data set of isotopically characterized perchlorate sources becomes sufficiently representative, stable isotope forensics of perchlorate may become useful for resolving issues of source apportionment, natural attenuation, or monitoring of remediation efforts.

## Cr isotopes as indicators of Cr(VI) reduction and contaminant sources

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Chromium is a common contaminant released from industrial facilities such as plating shops, cooling towers, lumber treating operations, and pigment factories. In many industrial areas, multiple Cr sources exist, and soils derived from ultramafic rocks can also emit Cr, so determining "ownership" of plumes can be difficult. Cr is usually released as Cr(VI), which is soluble and toxic, but is readily reduced by a variety of mechanisms to Cr(III), which is insoluble and less toxic. This reduction often occurs naturally, or it can be induced by a variety of interventions that effectively remediate Cr plumes. Two major goals of forensic Cr geochemistry are tracing plumes back to sources and determining rates of Cr(VI) reduction.

Cr isotope (<sup>53</sup>Cr/<sup>52</sup>Cr) measurements show promise as indicators of Cr(VI) reduction. The kinetic isotope effect causes heavier isotopes to become increasingly enriched in the unreacted Cr(VI) with increasing extent of reduction. Two challenges are encountered with this approach: 1) The size of the  $\delta^{53}\text{Cr}$  shift for a given extent of reduction is expected to vary if reaction mechanisms vary from site to site, and 2) The original  $\delta^{53}\text{Cr}$  of the contaminant must be measured or estimated, and the sources are often gone. We did several laboratory experiments to determine the variability of the kinetic isotope effect. Cr(VI) reduction by *Shewanella oneidensis* MR-1 produces a range of fractionation, but under "lean" conditions similar to those in most aquifers, values of  $4.1 \pm 0.2\%$  were observed. Abiotic reduction by Fe(II) and several organic reductants yielded a range of 2.9 to 4.7%, though this work is not yet complete. This range translates into an uncertainty of  $\pm 25\%$  in extent of reduction calculated from  $\delta^{53}\text{Cr}$  data. Further work is needed to better define the range applicable to actual field sites.

Industrial Cr(VI) supplies probably have Cr isotope compositions close to those of the earth's mantle. However, various plumes may have differing  $\delta^{53}\text{Cr}$ , depending on factors such as reduction in soils at the spill site. Cr derived from ultramafic rocks may be distinct from anthropogenic sources (Ball et al. talk, this session).

## Tracing sources, movement, and fate of hexavalent Cr in ground water using Cr stable isotope variations

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Hexavalent Cr (Cr(VI)) in potable water supplies has become an increased health concern. Cr(VI) in ground water often has had an anthropogenic source. Substantial variations in <sup>53</sup>Cr/<sup>52</sup>Cr ratios occur in nature and may allow identification of sources or chemical processes involving Cr. In waters with high natural background concentrations, Cr isotopes may be used to determine the source of Cr(VI) and to distinguish between anthropogenic and naturally-occurring Cr(VI).

About 200 ground-water samples were collected in the western Mojave Desert in 2001 - 2002. Cr(VI) concentrations in samples from uncontaminated sources ranged from less than the detection limit (0.1  $\mu\text{g/L}$ ) to about 60  $\mu\text{g/L}$ , and were as high as 1,500  $\mu\text{g/L}$  in samples from contaminated sites. Cr(VI) concentrations were less than the detection limit in recharge areas near the front of the San Bernardino and San Gabriel Mountains and in discharge areas near dry lakes with low dissolved oxygen concentrations. The highest Cr(VI) concentrations in uncontaminated water (near 60  $\mu\text{g/L}$ ) were from wells in the Sheep Creek fan, composed of alluvium weathered from the dark, micaceous schist of the San Gabriel Mountains. Cr(VI) concentrations ranged from 0.9 to 28  $\mu\text{g/L}$  in water from deep wells in the granitic deposits underlying water-supply aquifers near Twenty-Nine Palms, California.

In the Twenty-Nine Palms and Sheep Creek fan areas, <sup>53</sup>Cr/<sup>52</sup>Cr ratios ranged from near 0.0 per mil to +4.6 per mil. The latter value is almost exactly the theoretical maximum expected to accompany oxidation of Cr(III) on MnO<sub>x</sub> substrates. Values near zero were from sites known to be contaminated. The <sup>53</sup>Cr/<sup>52</sup>Cr ratios were higher in water from aquifers pumped for water supply and highest in water from the underlying deposits. These waters may be of greater age, thus the <sup>53</sup>Cr/<sup>52</sup>Cr ratios may reflect greater reaction progress. The <sup>53</sup>Cr/<sup>52</sup>Cr ratios from uncontaminated sites can be compared with those from contamination sites and may allow determination of the extent of anthropogenic contamination.

## Using tritium-helium groundwater age to assess contamination vulnerability in California

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The California Water Resources Control Board, in collaboration with the US Geological Survey and Lawrence Livermore National Laboratory, has implemented a program to assess the susceptibility of groundwater resources. Advanced techniques such as groundwater age-dating using the tritium-helium method, extensive use of  $\delta^{18}\text{O}$  for recharge water provenance, and analysis of common volatile organic compounds (VOCs) at ultra-low levels are applied with the goal of assessing the contamination vulnerability of deep aquifers, which are frequently used for public drinking water supply. Over 1200 public drinking water wells have been tested to date, resulting in a very large, tightly spaced collection of groundwater ages in some of the heavily exploited groundwater basins of California. When employed on a basin-scale, groundwater ages are an effective tool for identifying recharge areas, defining flowpaths, and determining the rate of transport of water and entrained contaminants. De-convolution of mixed ages, using ancillary dissolved noble gas data, gives insight into the water age distribution drawn at a well, and into the effective dilution of contaminants at long-screened production wells. In combination with groundwater ages, low-level VOCs are used to assess the significance of vertical transport.

### Acknowledgement

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## Fuel/hydrocarbon fingerprinting

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There are several different types of fuel/hydrocarbon fingerprinting techniques or approaches that can be used to answer specific site concerns. Generally, there is a need to identify the parties responsible for some type of environmental contamination. The knee-jerk reaction to such a situation is to try to "age date" the contamination that is present at the site. Here, a request is made to determine the age or date when a release occurred. This approach is usually problematic for a number of reasons. For example, a slow release that occurs over a period of several years will often have fuel of different ages at different locations within the contamination plume. The oldest material is often near the edge of the plume and the newest material located close to the release point. An "age date" analysis can then vary based on the locations from which one collects a sample for analysis. Likewise, the mixing of fuel of two different ages can also confound age dating techniques. Here, any "age date" is often guaranteed to yield an unreliable result, one that is neither as old as the oldest release nor as new as the most recent release. These limitations will be discussed using an "age date" model.

Alternatives to the knee-jerk "age dating" approach involve the development of various hypotheses which can then be evaluated in a scientific manner. This approach greatly expands the techniques that can be used to address the site issues, as well as the certainty of the findings. Examples of how such an approach can be used will be discussed.

When using analytical testing data in forensic evaluations, it is usually important to develop the context within which the data are to be used. The most common approach is a simple error analysis. Without such an evaluation, it may prove impossible to establish the significance of chemical testing results.

## Estimating the age of leaded gasoline releases using stable lead isotopes: The ALAS Model

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Estimating the year gasoline was released into the environment has been difficult. Methods relying upon gasoline additive chronologies and/or presumed rates of biodegradation do not provide adequate age resolution. Lead isotopic analyses of gasoline-impacted southern California marine sediments by Patterson demonstrated the presence of systematic isotopic variations in gasoline lead between 1940 and 1978. Circa 1990, Hurst surmised that if these temporal variations in gasoline lead isotope ratios were real and could be calibrated, an improved method of age-dating gasoline releases might result.

By acquiring samples of archived leaded gasoline and gasoline-impacted sediment, a calibration curve, the ALAS Model (Anthropogenic Lead ArchaeoStratigraphy), has been developed, and has been used to estimate the age of leaded gasoline releases. Systematic increases in <sup>206</sup>Pb/<sup>207</sup>Pb ratios observed in the ALAS Model curve (1965-1990) are caused by the increased use of radiogenic lead from Mississippi Valley-Type ores (<sup>206</sup>Pb/<sup>207</sup>Pb ≈ 1.30) in the production of alkyllead additives. The ALAS Model calibration curve exhibits minimal scatter, which is attributed to the use of very similar proportions of lead by major alkyllead manufacturers (e.g. Ethyl Corporation, DuPont) from 1965 through 1990. Correlations between ALAS Model ages and known release ages are excellent ( $R^2 = 0.97$ ), warranting the continued investigation of lead isotopes as tracers of lead pollution by hydrocarbons.

## A forensic geochemical technique for estimating release dates of petroleum products

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### Background

In 1993 Christensen and Larsen (C-L Model) proposed that the degradation of normal heptadecane (nC17) relative to pristane (nC17/Pr ratio) could be used to estimate the age of diesel fuels released to the environment. The C-L Model indicated a linear relationship between the nC17/Pr ratio of the petroleum and the time since the release. Given the importance of accuracy and scientific defensibility of techniques for estimating the age of petroleum releases, it is not surprising the the C-L Model has been the subject of much debate. Concerns are that variations and unknowns in the subsurface conditions, starting (initial) nC17/Pr ratios of the petroleum released and the rate of degradation contribute to uncertainties in the C-L Model age determinations.

### Middle distillate degradation model

In order to address the uncertainties in the C-L Model, measured initial nC17/Pr ratios of ~4500 worldwide crude oils and 90 domestic petroleum products, the C-L Model was integrated with more than 50 additional data points for comparative linear regression analyses and new data from documented distillate and crude oil releases from diverse geographic regions to assess variations in subsurface temperature and conditions. The culmination of these data is the MDD Model for estimating the age of ditillates, fuel oils and crude oil releases up to ~20 years with substantially more certainty than the C-L Model.

### Conclusions

The MDD Model can be used to estimate petroleum release ages in ranges from plus or minus 1.5 years under optimal conditions to plus or minus 5 years in a worst case scenario. Where little information is available about the site and the initial nC17/Pr ratios is unknown, then it may only be possible to estimate that the release is older or younger than 10 years, whereas a 3 year window would be reasonable under optimal conditions using due diligence and care.

## Identifying polychlorinated biphenyl sources in environmental media

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Polychlorinated biphenyls (PCBs) are a class of chlorinated organic chemicals that were produced and marketed commercially from the 1930s to the 1970s. PCBs were used for a variety of industrial uses (e.g. dielectric fluids in electrical equipment). PCB products were produced commercially by chlorination of biphenyl. The resulting products were mixtures of a number of congeners in relatively stable proportions. Their manufacture and use ceased in the 1970s because of mounting scientific evidence that PCBs accumulated in the environment and could adversely impact biota. However, due to their persistence in the environment, PCBs continue to be an environmental contaminant of concern.

Environmental forensics investigations of sites contaminated with polychlorinated biphenyls (PCBs) present unique challenges. Source inference in environmental media requires a broad range of knowledge in a number of subdisciplines.

In this presentation we will review PCB industrial use/associations, PCB chemistry, analytical laboratory methods, and the congener patterns associated with commonly encountered sources. While PCBs are more recalcitrant to weathering and alteration, they can be altered in the environment. We will review known PCB alteration patterns and discuss how such alteration can be recognized in environmental field studies. The implications of these topics will be illustrated using case studies conducted in a variety of different environmental media (air, water, sediment and biota). Finally, we will discuss the importance of data preparation, data analysis and data visualization in environmental forensics investigations involving PCBs.

## Use of alkylated PAH source parameters in environmental forensic studies: Testing the paradigm

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The application of alkylated polycyclic aromatic hydrocarbon (PAH) analytical methods provide opportunities to more effectively characterize diesel-range petroleum mixtures released into the environment and to use this information for liability allocation in environmental forensic studies. Successful use of this information in forensic studies requires an understanding PAH distributions in various source materials and how these PAH distributions may change in respond to environmental weathering processes. While there are many studies documenting PAH distributions of various source materials, to date there are few published reports which document the effect of environmental weathering on the distributions and thus little available information on which to base conclusions regarding the use of PAH ratios as indicators of either source or diagenesis.

In this study, geochemical data are presented for eight crude oils obtained from a single oil field and potentially representing various stages of in-reservoir degradation. The objectives of the study were to (1) demonstrate a single common source for all eight oils, (2) define the oils as part of an in-reservoir weathering sequence, and (3) use these data to examine the effects of weathering on various PAH parameters commonly used as either source characterization or environmental weathering indicators.

Results of cluster and principal component analyses indicate that these oils have a common source and that differences are due to in-reservoir weathering processes. These weathering processes have resulted in the substantial addition of organic sulfur to the oils as evidenced in the various PAH ratios by a preferential preservation of the sulfur-containing PAHs (benzothiophenes, dibenzothiophenes, etc.) The significance of these results regarding source characterization and specific weathering processes will be discussed.

## The use of micro-Raman spectroscopy as a fingerprint in a forensic investigation

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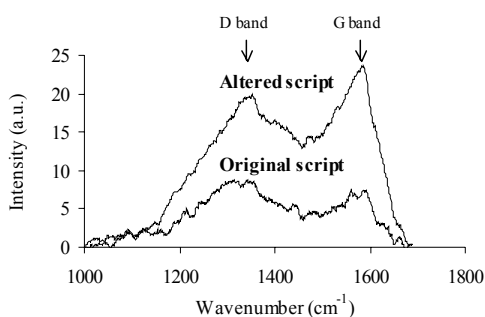
### Introduction

From the last years micro-Raman spectroscopy has revealed to be a powerful technique for the non-destructive physico-chemical characterisation of different materials related to the Forensic Science. A legal consulting in a cheque led to the investigation of the inks from the scripts using the micro-Raman spectroscopy. However there were no visible differences to the naked eye, the analysis were performed on different numbers and letters of the script.

### Results and conclusions

The spectra obtained (Fig. 1) reveal different Raman parameters ( $\nu$  and FWHM of the G and D bands; D/G intensity ratio) in the carbon component from the inks. This led us to the conclusion of the use of two different inks in the scripts (one used in the script 300 and another used in the number 1 of the 1300 euros).

Figure 1: The cheque investigated and the Raman spectra obtained in the original and altered script .



## A 270-year ice core record of atmospheric mercury deposition to western North America: An indicator of a partial success of the United States Clean Air Act of 1970

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The Upper Fremont Glacier (UFG), a mid-latitude glacier in the Wind River Range, Wyoming, U.S.A., contains a record of atmospheric mercury deposition. Although some polar ice-core studies have provided a limited record of past mercury deposition, polar cores are, at best, proxy indicators of historic mercury deposition in the mid-latitudes where 80 to 90 percent of the world's human population reside. Two ice cores removed from the UFG in 1991 and 1998 (each totaling 160 meters in length) provided a chronology and paleoenvironmental framework; essential to the interpretation of the mercury deposition record. A total of 97 ice core samples were selected using low-level processing methods and were analyzed to reconstruct a 270-year atmospheric mercury deposition record for the western United States.

The record shows that mercury concentrations are significantly elevated during periods corresponding to volcanic eruptions. This indicates that these natural events "punctuate" the record. Anthropogenic activities such as industrialization (global scale), gold mining and war-time manufacturing (regional scale), indicate that chronic levels of elevated mercury emissions have a greater influence on the historical atmospheric deposition record from the UFG. A declining trend in mercury concentrations is obvious during the past 20 years. This decline may be in response to the United States Clean Air Act of 1970.