International Workshop on Criminal and Environmental Soil Forensics 8th and 9th April 2006 Perth, Australia

A Post Symposium Workshop following the 18th International Symposium on the Forensic Sciences

PROGRAM & BOOK OF ABSTRACTS





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Venue:

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Dr Rob Fitzpatrick, Centre for Australian Forensic Soil Science (CAFSS)/ CSIRO Land and Water

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- Centre for Australian Forensic Soil Science (CAFSS)
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- Cooperative Research Centre for Landscape Environments and Mineral Exploration

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Photograph on front cover:

From CSIRO Land and Water, Pedology photograph set. Description: Detailed analysis of soil on the back of this shovel was used to help solve a homicide case. Photographer: Senior Constable Michael Heath. © 2004CSIRO

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International Workshop on Criminal and Environmental Soil Forensics Program

Saturday 8 April 2006				
8.30am – 9.00am	Registration			
9.00am – 9.10am (10 mins)	Welcome Message and Introduction Dr Rob Fitzpatrick - CAFSS and Richard Clarke - CCWA			
9.10am – 9.50am (40 mins)	Soil profiling (morphology) and mineralogy (X-ray, IR and microscopy) Forensic Soil Science: An overview with reference to case investigations and challenges Dr Rob Fitzpatrick – CSIRO Land and Water/CAFSS/CRC LEME (with M. Raven and M. McLaughlin)			
9.50am – 10.20am (30 mins)	The use of heavy mineral analysis to establish the provenance of suspect shellfish Richard Clarke – Chemistry Centre WA (with G. Bremner)			
10.20am – 10.30am (10 mins)	Topic Discussion			
10.30am – 10.50am (20 mins)	MORNING TEA			
10.50am – 11.20am (30 mins)	Soil as evidence in a Southern California forensic case and the development of a searchable soil profile database Marianne Stam - California Department of Justice, Riverside Crime Laboratory, USA			
11.20am – 11.50pm (30 mins)	Overview of x-ray methods and application to forensic soil examinations with case studies Mark Raven and Rob Fitzpatrick - CSIRO Land and Water/CAFSS			
11.50pm – 12.20pm (30 mins)	Mineral identification using the AutoGeoSEM Mike Verrall - CSIRO Exploration and Mining			
12.20pm – 1.20pm (60 mins)	LUNCH			
1.20pm – 1.50pm (30 mins)	Mid–Infrared spectroscopy (MIR): Overview of methods for rapid soil analyses in forensic soil examinations Sean Forrester - CSIRO Land and Water (with R.W Fitzpatrick, M. McLaughlin and L. Janik)			
1.50pm – 2.10pm (20 mins)	Topic Discussion			
2.10pm – 2.50pm (40 mins)	Soil taphonomy and grave excavation Forensic Taphonomy and Decomposition Processes in Gravesoils Dr Mark Tibbett - University of Western Australia			
2.50pm - 3.00pm (10 mins)	Topic Discussion			
3.00pm - 3.30pm (30 mins)	AFTERNOON TEA			

	Soil Geophysics
3.30pm – 4.15am (45 mins)	Clay vs Silt vs Sand: Does geophysical surveying of burials work all the time?
	Dr David Nobes - University of Canterbury, New Zealand
4.15pm – 4.30pm (15 mins)	Geophysical investigations at the Alleged Woolgar Massacre site, NW Queensland
	Ian Moffat - Ecophyte Technologies Pty. Ltd (with L.A. Wallis, P.C. Mill, and B.A. Keane and Woolgar Valley Aboriginal Corporation)
4.30pm – 4.40pm (10 mins)	Magnetic susceptibility and low altitude aerial photography using a helium balloon: applications to forensic soil examinations Dr Rob Fitzpatrick & Andrew Baker - CSIRO Land and Water/CAFSS/ Cooperative Research Centre for Landscape Environments and Mineral Exploration (CRC LEME)
4.40pm – 4.50pm (10 mins)	Topic Discussion
Posters 4.50pm – 5.00pm (10 mins)	From Kaikoura to Kaitorete: Non-invasive geophysical imaging of Maori burial sites
	Dr David Nobes - University of Canterbury, New Zealand (with L. Bateman, C. Butland, M. Flintoft, F. Gaiger, J. Lea, S. Wilkinson, H.M. Jol and M.A. Geer)
5.00pm	Close

	Sunday 9 April 2006
9.00am	Day 2 Commences
	Soil / Environmental Chemistry
9.00am – 9.10am (10 mins)	Overview and environmental case studies from a chemistry perspective Prof Mike McLaughlin - CSIRO Land and Water/CAFSS
9.10am – 9.30am (20 mins)	Case study involving lead and cadmium contamination around a lead smelter Bernie Zarcinas - CSIRO Land and Water
9.30am – 10.10am (40 mins)	Distinguishing, Anthropogenic and Geogenic Impacts of Environmental Contamination Prof Kirk Scheckel - United States Environmental Protection Agency
10.10am – 10.40am (30 mins)	Isotopes in soils: application to forensic science Dr Anita Andrew - Environmental Isotopes Pty Ltd
10.40am – 11.00am (20 mins)	MORNING TEA
11.00am – 11.30am (30 mins)	Marine Environmental Forensic Investigations: Case Studies of Very Wet, Very Salty Soil Assoc Prof Stewart Walker - Flinders University
11.30am – 11.40am (10 mins)	Terrestrial Environmental Forensic Investigation: Case Study of elevated levels of metals in drains in the WA wheatbelt Dr Rob Fitzpatrick - CRC LEME (with Andrew K. M. Baker, Mark Raven, Steve Rogers, Brad Degens, Richard George and Jason Kirby)
11.40am - 11.55am (15 mins)	Topic Discussion
	Soil biology and molecular diagnostics
11.55am – 12.35pm (40 mins)	DNA Fingerprinting Soils - Dirt, Death and DNA Dr Jacqui Horswell, ESR Ltd, New Zealand (with R. Parkinson, C. Macdonald, S. Cordiner, T. Speir, G. Chambers and A. Vass)
12.35pm – 1.05pm (30 mins)	Profiling and diversity estimations with soil DNA Prof Leigh Burgoyne - Flinders University of South Australia (with JM Waters, HA Chan, CDG Rogers, D.E.A Catcheside)
1.05pm – 2.05pm (60 mins)	LUNCH
2.05pm – 2.45pm (40 mins)	Identification of crime scene origin through use of soil organic evidence.
	Workshop Summary Dr Lorna Dawson - The Macaulay Institute, Scotland, UK (with L. MacDonald)
2.45pm – 3.05pm (20 mins)	Topic Discussion
3.05pm – 3.30pm (25 mins)	AFTERNOON TEA

3.30pm – 4.15pm (45 mins)	 General Topics Open for Discussion THE FUTURE – WHAT NEXT? 1. Networking / research projects in: Australia - Rob Fitzpatrick, Richard Clarke and others UK – Lorna Dawson USA - Kirk Scheckel (Environmental), Marianne Stam (Criminalistics) NZ - Jacqui Horswell, David Nobes and others 2. Soil Forensic Exhibits: Smithsonian Museum, International Soil Museum, Forensic Science SA. Rob Fitzpatrick – show draft display and discuss. 3. Forensic Soil Science – protocols. 4. Forensic Soil Science – field sampling kit.
Posters 4.15pm – 4.30pm (15 mins)	Mid–Infrared spectroscopy for rapid soil analyses Sean Forrester - CSIRO Land and Water
4.30pm	Workshop Close

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Forensic Soil Science: An Overview with Reference to Case Investigations and Challenges

<u>R.W Fitzpatrick¹</u>, Mark Raven¹ and Mike McLaughlin¹

¹Centre for Australian Forensic Soil Science, CSIRO Land and Water, Private Bag No 2, Glen Osmond, South Australia, 5064.

Environmental and forensic agencies in Australia do not have the capability to establish and maintain the expertise and infrastructure in soils and analytical soil science due to the focus on molecular forensic techniques. Hence, valuable evidence was often not collected and/or analysed, and new techniques in soil forensics were not being developed. At the request of the Australian forensic community the Centre for Australian Forensic Soil Science (CAFSS) was established to develop new techniques in soil forensics, and to provide a centre of expertise in soil forensics that could be used by Australian and International forensic communities. These communities also include environmental agencies and regulators, where sources and fate of contaminants in the environment could also be assessed using advanced analytical techniques.

We have developed and applied the following new systematic approach to discriminate soils using advanced soil morphology (e.g. colour, organic matter, diatoms), mineralogy, geochemistry (e.g. spectroscopy, magnetic susceptibility analyses) and wet chemical techniques:



New rapid spectroscopic methods, coupled with chemometrics, are being developed to rapidly screen and compare crime scene samples.

The following criminal and environmental forensic case investigations demonstrate how CAFSS has successfully used soil properties to both discriminate between and match soils for critical evidence in:

- Solving a double murder case by identifying the similarities between mineral assemblages in soils on a shovel and also from a quarry. The soils had a common provenance and revealed the location of two buried bodies.
- Identifying the locality of stolen ferns from a conservation park.
- Identifying provenance of soil removed from a site containing aboriginal artifacts.
- Sexual assault and kidnapping cases in which comparisons were made between soil minerals on clothing and a vehicle, with samples from the crime scene.
- Identifying the provenance of an industrial dust settling on parked vehicles. The mineralogy of these dusts identified them as coming from a cement works.
- Identifying the possible overseas and/or Australian provenance of soil on boots, rakes, shovels and mattocks belonging to suspected terrorists.
- Comparing dinosaur nest soil-like materials from imported samples and samples provided by Australian museums sourced from Henan Province, China.

CAFSS has been commissioned by national and international museums/ institutions to develop and supply interactive exhibits for visitors and the public to view how soil information can be used to interpret soil evidence to solve a crime.

While these advances described above have set new benchmarks in forensic soil science, the future will require still better understanding and linking of soil science, geophysics, mineralogy, chemistry and molecular biology information because these techniques will have a major future role to play in forensic searching- either to locate buried anomalies, blast residue or eliminate areas from searches.

The use of heavy mineral analysis to establish the provenance of suspect shellfish

Richard Clarke¹ and Graeme Bremner²

¹ Chemistry Centre (WA), 125 Hay Street, EAST PERTH, Western Australia 6004 ² Ministry of Fisheries, Private Bag 1926, DUNEDIN, New Zealand

Shellfish are sedentary, vulnerable to localized depletion, and often very valuable. Poaching is lucrative, and frequently attracts organized and transnational crime. Commercial fishing is usually constrained by catch or effort limits which are constrained by area; and straying into marine reserves or other closed areas is very profitable. The defence of closed areas against poaching is a difficult task and, unless a fisherman is observed in the act, the only means of detection is to establish the source of the catch after the event. This is possible if the animal in question was sedentary and acquired some characteristic mark of the environment in which it lived, a condition fulfilled by the abalone. Indicators of abalone provenance that have been used previously include population studies of encrusting organisms (e.g. seaweeds, corals) and heavy metal signatures obtained from chemical analysis of the soft tissue.

In a recently investigated case, mineralogical analysis of entrapped sediment has been used to locate the origin of a large catch of suspect abalone, landed by a commercial fishing boat in Nelson province, near the northern tip of the south island of New Zealand. The procedure involved the recovery of sand from worm tubes and other recesses on the shells, concentration and identification of the heavy minerals, and semi-quantitative analysis of the same fraction using X-ray powder diffraction. Comparison with sand recovered from abalone taken from a series of nine control sites over a 40 kilometre stretch of coast enabled the majority of suspect shells to be assigned unambiguously to a prohibited area of the fishery. A smaller number of animals were shown to be legally obtained. Interpretation of the data was based on visual pattern recognition using 2-D scatter plots, graphs and bar histograms, combined with simple logical deduction.

Soil as Evidence in a Southern California Forensic Case and the Development of a Searchable Soil Profile Database

Marianne Stam

California Department of Justice, Riverside Criminalistics Laboratory

Few California crime laboratories have analysts who are proficient in forensic soil analyses largely due to the perception that soil casework is too difficult, time consuming and provides little valuable information for the time spent to work the cases. Consequently, few attempts even the most rudimentary examinations and soils are often not collected or considered as valuable evidence at crime scenes.

This paper illustrates a Southern California soils case in which a detailed forensic soil profile database would have been useful in adding significance to the analytical results, and discusses the development of a searchable soil profile database that would make soil evidence more valuable as an investigative tool.

In the soils case, a suspect in San Diego County, California murdered two female acquaintances and buried them within 5 miles of each other in northern San Diego County. One victim was buried along a creek bed; and the other was buried in a citrus grove adjacent to a landscaped park. Investigators collected two shovels and a hoe from the suspect's garage. Soil from the shovels and hoe were examined and compared to soil from the two burial sites using basic analytical methods available in most crime laboratories. The soil on the shovels was similar to the soil from the citrus grove site, and dissimilar to the soil from the creek bed. The soil on the hoe was dissimilar to both burial sites.

The region where the burials were located is part of an extensive geomorphic province called the "Peninsular Range Batholith". This batholith includes Cretaceous granites, granodiorites, and gabbros, Mesozoic metasedimentary rocks, and Quaternary alluvial deposits. Results of the soil analyses on the shovels and at the citrus grove site showed the presence of quartz, plagioclase feldspars, alkali feldspars, a few zircons, biotite, hornblende, and a few pyroxenes; all quite common to batholithic environments. Consequently, although the soils on the shovels and at the citrus grove site were similar, the extent of the batholithic rocks, the lack of a detailed soil profile database, and the lack of better discriminatory analytical methods made it impossible to attach significance to this "match".

To improve the discriminatory abilities in California forensic soil cases, a University of California Soil Mineralogist was contacted about the idea of developing a searchable forensic soils database that would involve not only using the basic methods of soil analyses as discussed in the San Diego County case, but also more advanced analytical techniques, such as SEM/EDX. The concept and development of such a database will be discussed.

Overview of X-ray diffraction methods and application to forensic examinations

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The application of sophisticated analytical methods to forensic investigations is gaining increased importance due to advances in instrumentation and sampling techniques. Whilst X-ray diffraction (XRD) is a relatively old technique it is still of great importance due to its non-destructive nature; allowing further investigations of the original intact specimens. The major strength of XRD is its ability to identify and interpret crystalline components "directly". This is because XRD patterns are obtained from the interaction of the X-ray beam with the crystal structure of each component, and not "indirectly" derived from other information such as elemental analysis or chemical functional groups. XRD can be used to analyse all manner of crystalline materials from crime scenes such as; explosive residues, soil materials, paint chips, adhesive tapes, building materials, minerals, alloys, ceramics, gemstones and drugs. Various sample preparation techniques are employed depending on the quality and quantity of evidence available. Materials can either be analysed undisturbed on the carrier object or removed and analysed separately. Mounting the specimen in the instrument is also dependent on the size and shape of the items being investigated.

Several case studies will be presented outlining different aspects of XRD analysis applied to both criminal and environmental forensics. These will include; identifying the location of bodies from evidence taken from a suspect's vehicle, identifying the source of industrial dust recovered from parked vehicles, and analysis of burnt bone fragments.

Recent advances in X-ray diffraction instrumentation with direct application to forensics will also be covered along with advances in computer software for rapidly matching powder diffraction patterns of materials from crime scenes with databases of know materials.

Mineral identification using the AutoGeoSEM

Michael Verrall

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The AutoGeoSEM consists of a Philips XL40 Controlled Pressure SEM (Scanning Electron Microscope) fitted with an EDAX X-ray Spectrometer and custom written software for automation of the microscope and data processing. The SEM is operated in controlled pressure mode with a chamber pressure of between 0.1 to 0.7mBar. This reduces sample charging and allows non-conductive samples to be examined without a conductive coating. This is ideal for examining geological samples as well as other samples where coating the sample is undesirable or further optical work is required. The AutoGeoSEM technique has been used for applications including exploration for mineral sands, diamonds and oil. Samples used include polished sections of mineral grains and rough grains mounted on adhesive tape.

Samples to be analysed are typically mounted in resin, sectioned and polished. The polished sections are then placed in the SEM and the operator defines the area to be analysed and the parameters to be used for each sample. The AutoGeoSEM software will then drive the SEM stage to the first field of view, collect a BSE image and use image processing to distinguish the grains from the resin and to separate touching grains. Image analysis is then used to calculate grain attributes such as: area, width, equivalent circular diameter, perimeter, elongation, location of the centroid. Once the individual grains have been located a spectrum is collected from an included square within each grain. The spectra and images for each field of view are then stored and the process repeated for the next field of view.

For one sample, about 3000 grains are usually analysed and the total analysis time is just under an hour. The image acquisition time is usually about 5 seconds at a magnification of between x100 and x400 (depending on the grain size and detail required) and typically contain between 100 to 200 grains per field of view. The spectra acquisition time used varies from about 0.5 - 1 second.

The spectra collected are used to classify the grains into groups and determine the elemental composition of the grain. Each spectrum is compared to a database of standard spectra and a "Match Factor" from 0 to 100 is generated (100 indicates a perfect match). For most minerals Match Factors of 90-95 are typical. If a measured spectrum doesn't match any spectra in the database then a new group is created and any similar spectra will be assigned to this group. Typically anything with a match factor of less than 85 is considered not to match and will be assigned to a new group.

Once the grains have been classified the data is output in a text format that can be opened with standard spreadsheet programs such as Excel for further processing and presentation. These steps usually involve combining groups that are not of interest (such as the various minor silicate groups) and splitting the groups of interest in order to show more detail.

Mid–Infrared (MIR) spectroscopy: Overview of methods for rapid soil analyses in forensic soil examinations

<u>Sean Forrester¹</u>, R.W Fitzpatrick¹, Mike McLaughlin¹ and Les Janik²

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Mid-infrared (MIR) spectroscopy involves the acquisition of characteristic infrared spectra from an irradiated sample using an infrared spectrometer. The absorptions from fundamental and overtone vibrations of covalent bonding observed between various elements such as, carbon, oxygen, hydrogen, nitrogen, sulphur, phosphorus and silicon; allow for the identification of compounds by their spectra. The characteristic peaks in an infrared spectrum of soil can be related to specific soil physiochemical properties. Using partial least-squares (PLS), calibration sets may be built which relate soil spectra to laboratory data for these soils. The calibrations can then be used to predict soil characteristics for unknown samples.

This technique was initially developed at CSIRO to allow for the rapid investigation of soil mineralogy and carbon pools, utilising over twenty years of expertise in infrared soil analysis. The possible qualitative and quantitative applications of soil infrared spectra, led to a project to collate soil libraries and laboratory data into state-wide predictive datasets, and determine suitability for routine soil analysis. Work is continuing at present to combine state models into an Australia-wide calibration set, for the prediction of selected soil chemical and physical attributes.

The main advantages of the MIR-PLS technique over traditional laboratory analyses are speed, small sample requirements for an extensive suite of analytical results, also the procedure is sample non-destructive. Samples are analysed in minutes and all properties are predicted simultaneously, reducing the cost of the analysis. Hence, intensive sampling, analysis and soil mapping can be accomplished over large areas where this may have been time and cost prohibitive in the past.

MIR-PLS due to its rapid nature can be used as a "first-screening" for forensic soil sample analysis, typically predicting carbon pools, particle size distribution, CEC (cation exchange capacity), bulk density and pH. This gives an initial scan to determine samples that may require further analysis by other techniques (e.g. XRF, XRD, and ICP-MS). Soil spectra may also be analysed by PCA (principal components analysis), a reductionist statistical technique, to determine soil similarity.

Forensic Taphonomy and Decomposition Processes in Gravesoils

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Taphonomy was originally a branch of palaeontology and is literally the study of graves. From these historical roots, based in ancient graves (older than millennia), a new discipline of forensic taphonomy has emerged that relates to more recent graves (from days to millennia) and overlaps strongly with forensic archaeology and soil science. Forensic taphonomy today deals with the deterioration of cadavers, components of cadavers, and other materials of forensic importance. This is often with a view to determine the identity of the corpse or material, the manner and cause of death, or most commonly the residence time of the corpse (post-mortem interval) or material that has been buried (post-burial interval). To this end, this presentation will focus on the principles of forensic taphonomy and will review the recent advances in the science behind forensic taphonomy. The talk will develop a better understanding of the interface between the nature and properties of the burial environment and forensic materials that decompose within it with a particular reference to human remains and common mammalian analogues.

Clandestine burials of a cadavers introduce a high quality resource (high water content, narrow C:N ratio) that releases an intense, localised pulse of carbon and nutrients into the soil upon decomposition. This can have a marked affect on the physiochemical and biological properties on the surrounding soil commonly characterised by an increase in pH and the evolution of anaerobic gasses. Cadaveric materials are rapidly utilised by belowground floral and faunal communities and aboveground insect and scavenger communities. This results in the formation of a highly concentrated island of fertility, or Cadaver Decomposition Island (CDI). Each CDI is an ephemeral disturbance that, in addition to releasing water, carbon and nutrients to the wider ecosystem, acts as a hub by receiving these materials in the form of dead insects, insect exuvia and puparia, faecal matter (from scavengers, grazers and predators) and feathers (from avian scavengers and predators). Furthermore, CDIs are a specialised habitat for a number of flies, beetles, fungi and pioneer vegetation, which enhances biodiversity in terrestrial ecosystems. As we begin to understand the nature and succession physical, chemical and biological changes that occur in the CDI we can begin to estimate the residence time of cadavers and related materials in gravesoils.

Clay vs Silt vs Sand: Does geophysical surveying of burials work all the time?

David C. Nobes

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In the search for unmarked graves, we can eliminate methods, for example gravity and seismic, that lack the resolution (detail) to "see" the target graves. Infrared scanning can be used to detect the thermal anomaly of a burial, but the exponential decay of the thermal signal generally allows only younger graves to be detected. Electrical methods may work, but have practical limitations that may preclude their use. We have the greatest likelihood for detecting burials using magnetic field, electromagnetic (EM) and ground penetrating radar (GPR) methods.

Surveys of Maori ancestral burial sites, with both marked and unmarked graves, have allowed us to test when and where geophysical surveys are most likely to succeed. Results from five sites in three coastal settings along the east coast of the South Island of New Zealand show that burials in *clay* (Nobes, 1999) and *silt* (loess) (Bateman, 2003; Nobes *et al.*, 2006) can be identified using geophysical techniques, but burials in *sand* do <u>not</u> always yield anomalous responses (Nobes *et al.*, 2006).

Clay and loess are usually deposited as layers or massive beds so any disturbance due to burial is relatively clear. In contrast, near-shore, fluvial and dune sands contain sedimentary structures that can be difficult to distinguish from burials, and can mask the geophysical responses of the graves.

References

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- Nobes, David C., 1999. Geophysical surveys of burial sites: a case study of the Oaro urupa, *Geophysics*, **64**(2): 357-367.
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Geophysical Investigations at the Alleged Woolgar Massacre site, NW Queensland

lan Moffat^{1,2}, Wallis, L³, Mill, P¹, Keane, B¹, and Woolgar Valley Aboriginal Corporation

³ Department of Archaeology, Flinders University

Geophysical surveying was used in an attempt to define the location of the alleged Woolgar massacre site, northwest Queensland. The general location of a burial site (known as "Skull Camp") is well known among the locals, since Aboriginal skeletal remains were uncovered and reburied at the site during fencing operations in 1952. Oral histories from both the contemporary Wanamara people and members of the non-Indigenous pastoral community suggest these remains represented the victims of retaliatory massacres by the native mounted police for the fatal spearing of SubInspector Henry Kaye in 1881. The 1952 fence line (along which the remains were buried) is still observable and (with the guidance of the pastoralist who constructed the fence) a geophysical survey program incorporating ground penetrating radar, direct current resistivity, electromagnetic induction and a magnetometer was conducted with the aim of defining the location of skeletal material, changes in soil stratigraphy or a metal chain that was thought to have been used to restrain the victims during the massacre event. The survey yielded some anomalies that were investigated by digging a trench to a depth of 1.5m over the prospective area of the site; however no skeletal remains were located. Unfortunately since 1952 a section of the fence measuring approximately 250 m from within the middle of the survey area has been eroded by flood events, therefore the most parsimonious explanation for the lack of physical remains (given that skeletal material was definitely present during the fence's construction) is that they have been eroded from this area of missing fence line. Despite the lack of recovery of skeletal remains the project has demonstrated the utility of a multi-technique geophysical approach to potential massacre sites, particularly where the target location is well defined.

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Magnetic susceptibility and low altitude aerial photography using a helium balloon: applications to forensic soil examinations

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Mineral magnetic techniques (e.g. mass and volume magnetic susceptibility) are a relatively recent development (post 1971) and have now become a very powerful and widely used research tool to characterise natural and anthropogenic/polluted materials in landscapes (Thompson and Oldfield, 1986).

Magnetic susceptibility measurements can detect the presence of iron oxides in soils at lower concentrations than other methods such as X-ray diffraction analyses. In soils, their magnetic properties reflect the varied magnetic behaviour of the bulk of soil minerals present. In many soil samples, the magnetic susceptibility is largely determined by the ferrimagnetic mineral present such as magnetite and maghemite. Other major soil constituents may also affect magnetic susceptibility values. Quartz, calcium carbonate, orthoclase, organic matter and water are diamagnetic and, in most soils, these dilute the magnetic properties. In extreme cases, such as pure silica sands and pure limestone, the diamagnetic component will have a significant effect on the magnetic susceptibility of the sample. Paramagnetic soil minerals are those rich in iron but low in ferrimagnetic properties. They may make a significant contribution to bulk magnetic susceptibility. Antiferrimagnetic minerals will also increase magnetic susceptibility values. Of these, goethite and hematite are the most abundant and therefore can make an important contribution to the magnetic properties of soils.

Mass magnetic susceptibility (χ) was measured at low frequency (0.46 kHz; Xlf) and high frequency (4.6 kHz; χ_{hf}) using a Bartington magnetic susceptibility meter model MS2 (Bartington Instruments Ltd., Oxford, England) equipped with a 32 mm diameter dual frequency sensor, type MS2B in the laboratory. This enables frequency dependent susceptibility ($\%\chi_{fd}$) to be calculated ($\%\chi_{fd} = (\chi_{If^-} \chi_{hf})^*100/(\chi_{If})$). Volume magnetic susceptibility determinations were conducted in field also using a Bartington magnetic susceptibility instrument model MS2 but equipped with a MS2F probe, which determines concentrations of magnetic materials in the top 30 mm.

Mineral magnetic measurements have been most effectively applied to soils, soil parent materials, bedrock, river sediments and estuarine cores in studies of whole catchments. These techniques have also been used in a wide range of environmental studies such as sourcing sediments in reservoir catchments; establishing stream arm sediment contributions at river confluences; sourcing estuarine sediments; characterisation of soils; tracing overland soil movement; and identification of fire-induced magnetic oxides in soils and lake sediments. Soil magnetic properties, used in conjunction with other pedological and mineralogical methods, have been used to trace sources of alluvium to measure the extent of erosion and deposition in eroding landscapes.

We have developed a new method for hi-resolution, low altitude aerial photography using a Helium balloon. This involved using a 7.1 mega pixel digital camera (Canon PowerShot S70) being suspended on a gondola beneath a weather balloon filled with helium (Baker *et al.* 2004). A fine fishing net was used to distribute strain evenly over the balloon. The platform was tethered using two strong, light and highly visible cords attached to the net. A 5 kg weight was attached to each tether to anchor the equipment. The balloon was filled with approximately 3.0 m3 of helium providing 33 Newtons of lift capable of supporting three times the combined mass of camera, balloon gondola and tethers (0.9 kg). Excess lift, created by the high volume of helium, tensioned the tethers and stabilised the balloon and gondola. The latter incorporated a gimble that allowed the axis of the camera lens to remain perpendicular to the ground. A wireless video transmitter and infrared-extender were housed within the gondola. The camera's field of view was relayed to a handheld LCD screen and an infrared remote control facilitated ground-based shuttered release.



The method was used to acquire five sets of high resolution aerial photography (1 pixel = 2 cm) over a 12 month period in an Adelaide Hills focus catchment where volume magnetic susceptibility and electromagnetic data (EM 38) had also been acquired. The survey was carried out over a grid comprising GPS-located sample points along transects 20 m apart. The balloon was allowed to slowly ascend on its tethers to a maximum height of 50 m (Australia Civil Aviation Safety Regulations state - unmanned aircraft have a maximum ceiling of 122 m). Following processing all aerial photographs were analysed using ArcMap 9.1. These geophysical techniques (volume magnetic susceptibility and EM-38) - in conjunction with terrain information and soil analyses - have been used to successfully help interpret discreet soil-landscape patterns that have been attributed to erosion, salinity, waterlogging and soil forming conditions.

Hi-resolution, low altitude aerial photographic images were taken using the Helium balloon method at a murder scene in a partially vegetated sand dune in South Australia.

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From Kaikoura to Kaitorete: Non-invasive geophysical imaging of Maori burial sites

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Sacred sites need to be treated with care and sensitivity. Non-invasive, non-destructive geophysical surveying is an appropriate, optimal way to map the nature and extent of such sites, particularly Maori burial sites (*urupa*). Since 1997, geophysical methods have been successfully used on urupa (Nobes, 1999). The response varies site-to-site, depending primarily on the nature of the soil and the underlying bedrock.

The results from four sites represent three physical settings:

- (1) <u>Clay</u>: The Oaro urupa, south of Kaikoura on the east coast of the South Island of New Zealand, is on top of a hill overlooking the South Pacific, and was the first urupa to be surveyed using geophysical methods (Nobes, 1999). The site's clay soils overlie limestone bedrock. Magnetic, electromagnetic (EM) and ground penetrating radar (GPR) responses to the graves are clear and unequivocal, once the influences due to fences and the clay are removed.
- (2) <u>Loess</u>: The Koukourarata (Port Levy) and Wairewa (Little River) urupa are in loess overlying the volcanic bedrock of Banks Peninsula, near Christchurch, New Zealand. The EM responses are apparent but are not as obvious as for the magnetic surveys. The GPR surveys have clear anomalous responses that in most cases can be interpreted as graves (Bateman, 2003).
- (3) <u>Sand</u>: The Mangamaunu urupa is situated in coastal beach sands, just north of Kaikoura and near the hamlet of Hapuku. Anomalous geophysical responses are present but less obvious than for the Oaro, Koukourarata and Wairewa sites. We can identify some areas where the variable geophysical response is likely due to grave present, but some known, even marked graves have no anomalous response.

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A case study involving lead and cadmium contamination around a lead smelter

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Environmental forensics entails the study of earth materials and contaminants found on or in a receptor to possible source localities in order to establish the degree of probability that the material was or was not derived from a particular location. Many analytical methods are employed to examine and determine the characteristics necessary to differentiate the forensic environmental samples and establish the degree of probability that the material was or was not derived from a particular location.

Background

The maximum level (ML) for lead (Pb) in cereal grains was previously 0.5 mg Pb/kg. This ML expired in December 2002 and the new Australia New Zealand Food Standards Code ML (0.2 mg Pb/kg) took effect. Codex, which has standards for internationally traded food commodities, also has a ML of 0.2 mg Pb/kg for cereal grain.

Case Study

To determine the provenance of Pb and cadmium (Cd) in wheat and barley crops around a lead smelter, and the pathway of metal exposure.

Approach

- Determine the pathway of metal exposure i.e. was contamination occurring through root uptake of metals, or through direct dust/aerosol contamination of grain.
- Examination of elemental (Pb/As, Pb/Cd and Pb/Sb) and isotopic ratios (^{208/206}Pb, ^{207/206}Pb, ^{206/204}Pb, ^{207/204}Pb, ^{208/204}Pb) in dust contaminating grain, in soil, and in smelter emissions via high volume air samplers
- Determine if grain contamination was exacerbated by the physical process of harvesting (i.e. passage through a grain header) or by storage in silo.
- Examine the morphology of the Pb particles as this characteristic is strongly influenced by the temperature of the process from which it emanated.

Distinguishing Anthropogenic and Geogenic Impacts of Environmental Contamination

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The US EPA is charged with enforcement, regulatory, and research objectives to protect human health and to safeguard the natural environment. The research aspect of the US EPA employs techniques to determine how contamination occurred and its long-term impact with emphasis on remediation. Metal contaminated media provide extreme challenges in identifying the responsible source. Advanced analytical speciation tools at synchrotron research facilities such as X-ray absorption and X-ray fluorescence spectroscopies provide key pieces of evidence to discover the source of contamination and plan remediation activity. Aside from higher resolution and sensitivity of synchrotron techniques over traditional laboratory instruments, the distinguishing feature of synchrotron X-ray techniques will be provided to illustrate its utility to other related disciplines in environmental sciences and forensics.

At several metal contaminated sites within the US, the US EPA utilizes bench and field studies coupled with synchrotron speciaiton to understand the form, fate, and bioavailability of metals in addition to collecting forensics evidence for enforcement. At some sites, the origin of contamination is obvious and the responsible party is working with the US EPA to initiate remediation strategies. However, other sites where multiple parties may be responsible or where natural sources of contamination may exist, the ability to decipher culpability is tremendously complex without the correct approach. Through viable collaboration, a strong network of US EPA engineers, hydrologists, chemists, and soil scientists design and execute research to answer these multifaceted questions.

This presentation will highlight the synchrotron speciation efforts of the US EPA to identify the chemical forms of metals and how this information is utilized in remediation and enforcement protocols. A case study of a former military site near Boston, Massachusetts (USA), will illustrate the complex issues surrounding groundwater arsenic transport to a severely impacted nearby lake. At issue is the source of arsenic and its stability in the lake sediments. A large portion of the northeast United States has bedrock material with elevated levels of naturally occurring arsenic; however, scarce records of materials added to a large, historical landfill near the lake raises suspicion as a potential source. Likewise, past manufacturing activities in the area may be a distant source of contamination. Distinguishing the origin of arsenic between these possible sources is the primary objective but risk assessment to humans and wildlife will be the driver for remediation.

Isotopes in soils: application to forensic science

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The application of isotopic methods to forensic science has developed from an understanding of natural variations, with the behaviour of the light stable isotopes (H, C, N, O and S), generally well understood in geological and biological processes. Developments in analytical methods, such as coupling an elemental analyser or a gas chromatograph to an isotope ratio mass spectrometer, have led to automation and reduction in sample requirements, making these techniques widely applicable and cost effective.

Soil chemistry is complex, reflecting the effects of rock degradation, hydrological and biological processes and, commonly, anthropomorphic processes. Each of these processes has characteristic isotope signatures in one or more element that can be used to fingerprint soils in forensic investigations. Parent rock chemistry will be important in defining the S and N isotope values (as well as major and trace element compositions) of soils; hydrologic processes influence O, H and S isotope values in soils; biological processes dominate C and H signatures of organic matter; and anthropomorphic processes will be defined by the nature of the contaminant but will commonly be reflected in S, N, C isotope values or trace element compositions. Using multi-isotope signatures associated with different processes should yield unique identifiers.

Any isotopic approach needs to be considered within a sampling framework, including profile position, season, size fraction, selective leaches, sub samples and disturbance, and an analytical approach, including dissolution issues, bulk *vs.* compound specific methods. Examples from environmental and criminal forensic studies will be used to illustrate some of the issues.

Sediment – Very Wet Soil

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Sediment can be considered as very wet soil. Sediment is the material that is found under-water in streams, rivers, lagoons, billabongs or along the coast or under the sea.

Like soil sediment contains a range of particle material

- fragments of rocks, minerals such as quartz
- skeletal remains or shells whole diatoms
- organic material in various stated of decomposition.

The main differences between soil and sediment are

- moisture content
- varied level of salt
- restricted access to oxygen leading to oxic and anoxic layers
- size distribution.

Sediment coming from a river into the sea will be distributed depending on the energy of the river and sea and wave, wind and tidal regimes. For example a beach that is exposed may have no fine sand because the very fine particles have been washed away leaving large pebbles which require more energy than the water in the system had.

There are two main areas where forensic analysis of sediments are very important.

1) Sediment may be relevant to forensic cases when someone drives through a stream or walks over a beach leaving particular sediment on the tyre or on the sole of a shoe. Bodies may have been exposed to sediment either by sediment at the scene of the crime or because the body was dumped in a river or the sea or at a beach or weighted down in a reservoir.

2) In the field of environmental forensics sediment can be analysed to determine what is there, is in natural or has it been contaminated with a pollutant and, if so, how much pollutant is there and where this pollution came from. The ultimate aim of a forensic environmental contamination may be to present evidence that is acceptable in court that a specific amount of a specific chemical came from a specific factory and is now in a sediment.

The collection and analysis of sediment may require slightly different procedures or awareness of problems that may occur with sediment – for example highly saline sediment causing interferences in analysis.

This presentation will also give a case study where the collection and analysis and interpretation of sediment are discussed.

Terrestrial Environmental Forensic Investigation: Case Study of elevated levels of metals in drains in the WA wheatbelt

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Salinised land in the wheatbelt of Western Australia (WA) is expected to increase to over 3 million hectares if current trends continue and the construction of open drains has increasingly been seen as a major management option. Over 15 000 kilometres of drains have already been constructed in parts of the WA wheatbelt and generally without extensive regional linkages. Unfortunately most of the drains have been constructed with limited planning, design, and construction guidelines, and usually without an understanding of their effectiveness, stability, or their downstream impacts. However, despite these problems as well as the variability in drainage response and the relatively flat landscapes in the wheatbelt, deep drains are increasingly seen as a viable option.

Consequently, a research project to quantify the geochemistry of groundwaters and drain sediments in the WA Wheatbelt, particularly the Avon Basin, was developed to assess the causes and risks and to identify management options as part of the Engineering Evaluation Initiative (EEI). The specific objectives of this study were to:

- Assess the impacts of deep open drains on the production, export and fate of leachate and minerals.
- Develop strategies to appropriately manage the drained soils, acidic water and oxidation products.

A summary of some recent findings are:

- Acid groundwater is a natural phenomena in the WA wheatbelt. The highly acidic shallow groundwater, which is caused primarily by iron hydrolysis reactions. This situation develops when anoxic water containing dissolved ferrous ions is exposed to air and ferrous ions are oxidised to the ferric ions, which reacts with water to form reddish-brown precipitates of ferric oxyhydroxides, releasing free hydrogen ions in the process. If the water sample contains a substantial amount of dissolved iron and has a low buffering capacity, the pH of the solution may fall from a value of about 6-7 to 2-3.
- Drains accelerate generation of acid due to increased exposure and oxidation of groundwater Fe²⁺ to air.
- Drain sediments contain sulfidic and sulfuric materials (acid sulfate soils), which will increase the risk of additional acidification in drainage waters.
- Types of salt efflorescences are an integrated indicator of complex biogeochemical conditions and transformations occurring in the drains and play a critical role in drain erosion processes by salt fretting.
- Soluble sulfate/chloride-containing minerals play important roles in the transient storage of components (Na, Ca, Mg, Ba, Sr, Cl, Br, I & SO₄), which will dissolve during rainfall and contribute to formation of saline monosulfidic black ooze in drains.
- Types of iron oxyhydroxysulfates and oxyhydroxides are indicative of rapidly changing local environments and variations in redox, pH and rates of availability of S and other elements. As such, these minerals are indicators of soil-water processes operating in specific landscapes.
- Sulfuric horizons contain elevated trace element and metal concentrations (AI, Cu, Pb, Cr, Pb, Zn, Mg and rare earth elements).
- Anthropogenic burning results in the formation of irreversibly fused, particulate and discrete iron-rich artifacts, which can block drain pipes.

DNA Fingerprinting Soils - Dirt, Death and DNA

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Soils are complex and dynamic ecosystems developed from parent materials over time by the actions of plants, animals and microorganisms. Soil is frequently found on clothing, vehicle tires and interiors, in the tread of footwear and on carpets at crime scenes, and as trace evidence on objects. In addition, homicide victims are often discovered outdoors, on soil surfaces, or in shallow graves.

Recent developments in molecular biology are now applicable to soil investigation and have the potential to bring the molecular revolution to bear on soil in the same way that it has for human DNA profiling. DNA can now be extracted and profiled from the microbial communities that live in the soil. The method is sensitive enough to show whether soil samples are the same as each other or are different. This 'Soil Microbial Finger-Printing' can also be applied to "time since death" estimations.

After death, the human body decays in a succession of stages. For a body on top of soil, the products of decomposition seep into the ground. This input of substrates and microorganisms from the body causes the profiles of the microbial community in the soil to change significantly over the period of decomposition. We have been testing the hypothesis that microbial community profiles in soil reflect decomposition stages and hence provide a novel means of estimating PMI. Understanding homicide decomposition-associated microbiology based on DNA profiling may eventually lead to the development of a new technique to estimate the time elapsed since a person died, could confirm that a decompositional event had occurred at a specific location, even if no corpse is found, and could potentially link trace evidence to a crime scene.

The goal of this research project is to extend the development of molecular technology into new areas, to provide scientifically and legally robust DNA profiling/fingerprinting systems.

Profiling and diversity estimations with soil DNA

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In soil identification, diversity of methodology is always likely to be the norm so soil evidence is heading towards being both objective and combinable by Bayesian technique. The day of the expert-witness is ending. Mineral evidence, DNA evidence and other biological evidence will ultimately have to be combinable into a single, defendable probability-statement before presentation to a court.

When there is ample DNA, the problem is not so much a lack of methods to do this so much as an excess of methods. However there is usually quite limiting amounts of DNA from case work, reducing the breadth of choice of methods and choice is further reduced when we include the limitation that the selected technology(s) must be able to develop and yet be backwards-compatible like good software. We report that high gain amplification with unspecific priming is promising when DNA levels are highly limiting and very high gain amplifications become unavoidable. High-gain is commonly distrusted for ordinary PCR because it distorts the ratios of amplification products away from the ratios of the original templates. The use of unspecific priming is also distrusted because of the confusion from multiple initiation sites.

However, as Darwinian distortions are unavoidable with high-gain amplifications it was considered to be pragmatic to utilize their distinctive properties rather than to attempt to minimize or avoid them. In practice molecular-Darwinism during high-gain amplification was found to select almost the same few "winners" out of the complex populations of soil DNA making it a profiling strategy *in its own right* and, in theory, the multiple initiation site-problem with arbitrary primers should become greatly reduced by the very same selective effects. This deduction appears to be supported by the evidence.

As expected, strongly-selective amplifications appear to select from the menu of sequences in a template with negligible regard to the relative contributions of the template's molecular species and this effect has the practical advantage of erasing much variation arising from the well-known variability of soil-DNA-extraction procedures. Although not immediately obvious, the logical corresponding disadvantage is that minute traces of a highly diverse contaminating DNA, like human DNA, may overwhelm a profile.

Molecular Darwinism also provided an objective method for calibrating the relative diversity of a population against a standard that has no shared homology to the population being investigated. For example, a soil metagenome's diversity was scaled against the human genome.

As expected, the profiles of synthetic mixtures don't look simply like the additive consequences of their components which, again, is what might be expected from a competitive amplification model.

There are many other pros and cons: Current work still relies on arrays. Arrays make awkward data-bases but the technique is developing towards data-base-able sequences. Some back-compatibility to future developments can already be obtained by storage of DNA on say, FTA^(tm) but the future of back-compatibility probably lies with an electronic data-base of sequences so that the old records will only differ from new records by the numbers of sequences held within them.

Identification of crime scene origin through the use of soil organic evidence

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Forensic examinations at a crime scene can often involve the identification of soil materials. Soil is a complex material, the analysis of mineral and organic components providing information regarding geological origin, dominant vegetation and management. The ability to compare and match samples from different sources, providing clues to likely origin, is potentially a valuable tool in criminal investigations.

This paper considers the power of new fingerprinting methods, particularly those focusing on the organic component of soil, for comparing samples of different soil type and land-use. These methods include Fourier Transform Infra Red spectroscopy (FTIR), organic wax biomarker analysis and DNA fingerprinting, and are considered along with classic soil characterisation methods such as elemental analysis, mineralogy, palynology and optical methods. These techniques can both complement conventional forensic methods and provide new investigative or matching tools where previously none existed.

Individual analytical techniques will have different degrees of importance depending on the nature of the criminal case in question. Each method has its strengths for different situations and there is a great need to give more guidance on how to deploy the appropriate techniques for a given situation. As many more methods become quantitative, their use 'in combination' will help to characterise the soil more accurately and thus help to refine and narrow probable origin as well as give increasingly robust sample matches with defined probabilities.

One crucial component of applying a forensic approach to soil, is the development of a set of reference soils and databases, allowing estimation of probability of obtaining accurate soil matches. This would also pave the way in developing soil GIS tools. At present there is no reference 'population' to judge any soil analysis against. There are, however, significant sources of data and archived soils around the world that have been gathered for other purposes. There is therefore an opportunity to use some of these sources to generate the population data needed to test existing and new methods for their accuracy and resolution, and to determine the probability and certainty for the most promising methods. The SoilFit project will test many of these approaches.

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