

MFRC

Midwest Forensics Resource Center

Research and Development
Program Summary

April 2006

Acknowledgement

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Midwest Forensics Resource Center Research and Development Program

Introduction

The mission of the MFRC Research and Development Program, is to provide technological advances in forensic science for the benefit of our regional partners as well as the forensic community at large. Key areas of forensic science need are identified through our interactions with our Midwest partners and our R&D advisory group, as well as through our participation in national meetings in forensic science. Under the sponsorship of the National Institute of Justice and the U.S. Department of Justice COPS Program, the MFRC solicits proposals for the development of practical and useful technology, instrumentation, and methodology that address needs in areas related to forensic science and its application to operational crime laboratories. The MFRC facilitates proposal development by working to establish partnerships between researchers and our regional partners. The MFRC administers a peer-review of the proposals and then funds the selected projects at a cost of approximately \$55,000 each, with a 12-month period of performance.

The process for selection of these projects includes the following steps: 1) drafting of a call for proposals by MFRC staff, 2) review of the draft call by members of the R&D advisory committee, 3) review and approval of the call by NIJ, 4) issuance of the call to ISU, Ames Laboratory, regional partners, and research organizations, 5) receipt of proposals, 6) review of proposals by R&D advisory committee, 7) ranking and selection by MFRC staff using advisory committee reviews, with concurrence by NIJ, 8) notification of proposers, 9) receipt and review of progress reports by MFRC, 10) receipt and review of final reports by MFRC, R&D advisory committee, and NIJ.

The decision to fund any specific project is based upon a peer-reviewed call-for-proposal system administered by the MFRC. The reviewers are crime laboratory specialists and scientists who are asked to rate the proposals on four criteria areas including: 1) relevance to the mission of the MFRC, 2) technical approach and procedures, 3) capabilities, teaming, and leveraging, and 4) implementation plan. A successful proposal demonstrates knowledge of the background for the research and related work in the field and includes a research plan with a defined plan to implement the technology to benefit our partners at the crime laboratories.

Program Summary Technical Sheets

The following project summaries, while not a complete summary of all research areas, are meant to demonstrate the range of research funded by the MFRC. The project summaries describe the forensic need the projects serve as well as the benefits derived from the technology. The summaries provide a brief description of the technology and the accomplishments to date. In addition, the collaboration with regional partners and the status of the implementation of the technology are highlighted. These technical summaries represent the development and implementation of practical and useful technology for crime laboratories that the MFRC hopes to accomplish.

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Analysis of Forensic Soil Samples via High Performance Liquid Chromatography (HPLC) and Ion Chromatography (IC)

FORENSIC TECHNOLOGY NEED

Forensic soil comparison has traditionally been performed via analysis of color, texture, and mineralogical content, including an analysis of particle distribution by PLM and mineral identification by spectroscopy. Although these are useful methods for discrimination, they primarily target inorganic minerals in the soil while the organic and water-soluble constituents are largely neglected. Chromatographic methods could provide additional discrimination; the compounds examined by these methods (humic materials, organic and inorganic contaminants) are independent variables that are not examined in traditional mineralogical methods. Also, analyses of this type could be useful screening tools for soils, providing a quick method for eliminating soils from different sources before beginning any mineralogical analysis. This work is being conducted at the State of Michigan, Department of State Police, Lansing Forensic Laboratory.

TECHNOLOGY DESCRIPTION

Methods for HPLC analysis of organic and humic materials in soil have been developed but there is some variation in the forensic literature as to whether this type of analysis effectively discriminates soil samples. While the humic substances themselves may not discriminate soil samples, environmental contaminants can be detected by these methods and may prove to be an effective discriminator. IC has been widely utilized for the analysis of water-soluble inorganic ions in forensic explosive analysis but the application of this technique to forensic soil samples has not been explored. The purpose of this study is to evaluate the discriminatory power of HPLC and IC in forensic soil comparisons.

Qualitative HPLC and IC analyses of soil from 120 different locations will allow for an assessment of the evidentiary value of any similarities and/or differences in the organic and inorganic fractions of soil encountered during forensic analyses. Quantitative IC analysis will help determine baseline levels of anions (e.g. nitrate, chlorate, and perchlorate) in soil from various locations. Samples from six locations will be collected and analyzed over ten weeks to examine the temporal variation in organic and inorganic content of soils and the spatial variation of these constituents will be examined via grid sampling of these same six locations.

TECHNOLOGY BENEFITS

Quantitative IC analysis will help determine baseline levels of anions in soil from various locations and therefore aid in the forensic analysis of soils from cases involving low explosives; many of these devices contain some of the same anions detected by this method.

Any increased discrimination of soil samples via these methods will improve the analytical scheme for soil analysis by eliminating Type II errors (false inclusions). In cases where an inclusion is inferred, the improved discrimination will reduce the size of the class to which the samples belong and add significance to the association.

COLLABORATION/IMPLEMENTATION

Graduate students from Michigan State University are collaborating with Christopher Bommarito, Michigan State Police Forensic Science Division.

The results of the research will be disseminated through presentation at forensic meetings of the Midwestern Association of

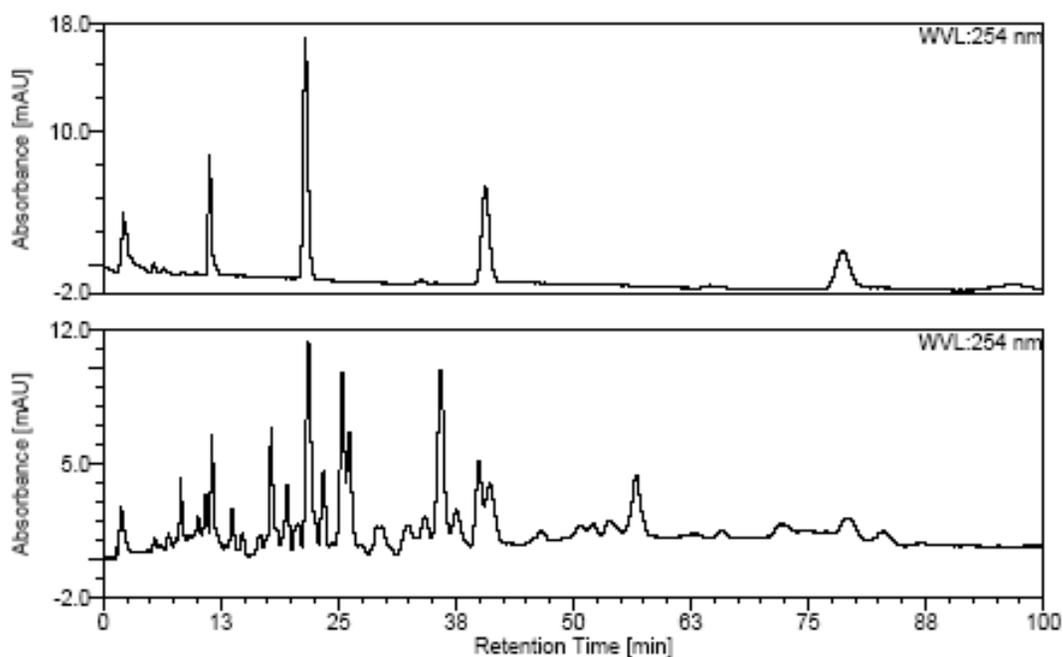


Figure 1. HPLC chromatograms of two samples showing qualitative differences in the number and location of components.

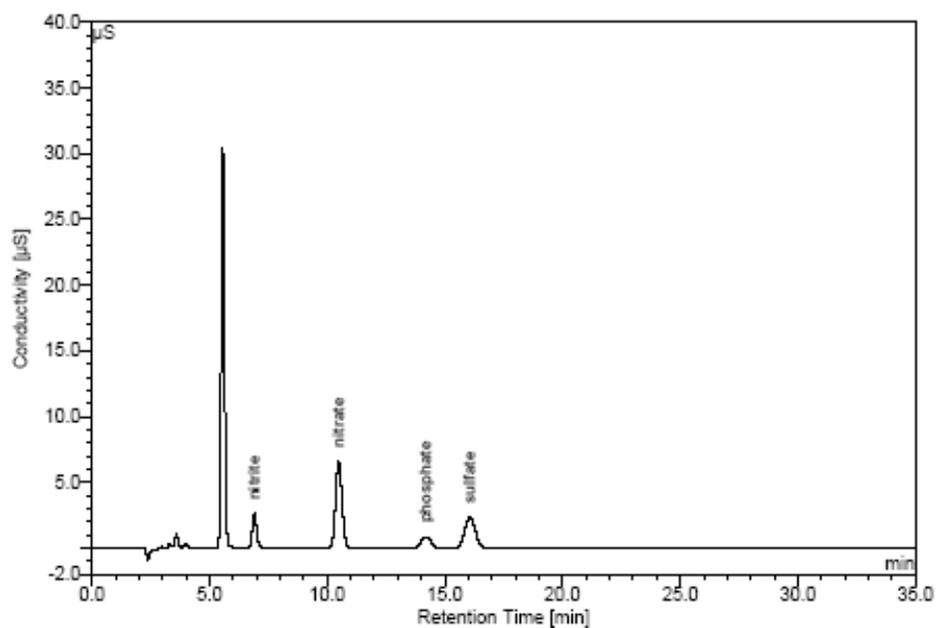


Figure 2. IC chromatogram of one sample showing detectable amounts of nitrite, nitrate, phosphate, and sulfate.

Forensic Scientists and the American Academy of Forensic Sciences. The research will be submitted for publication in the Journal of Forensic Sciences or other appropriate scientific journal.

ACCOMPLISHMENTS AND ONGOING WORK

The first two quarters of the project have been dedicated to sample preparation and instrumental analysis via both HPLC and IC. All 234 samples were prepared and have been analyzed using the developed methods. Examples of the resulting data are given in Figures 1 and 2. Figure 1 shows the HPLC Chromatograms of two samples and represents the magnitude of the qualitative differences in organic composition observed in the sample population. Figure 2 shows the IC chromatogram of one sample from the method used to detect nitrite, bromide, chlorate, nitrate, phosphate, and sulfate. The color of each sieved soil sample was determined according to the Munsell Soil Color Chart and Analyse-It statistical software was purchased for the quantitative analysis of the data.

Over the next few months the data from the sample population (n=120) will be examined in detail to determine the discriminating power of each of the two methods developed earlier in the study. The data from the spatial (n=54) and weekly (n=60) samples will be examined to determine the variation in Mid-Michigan topsoil composition over space and time. The first step in these examinations will be a qualitative comparison of the location and number of constituents in HPLC and the overall anion composition in IC. Then, a semi-quantitative comparison of the relative ratios in HPLC and a quantitative comparison of the anion concentrations in IC will provide statistical support for any visual differentiations.

The statistical analysis will be completed utilizing the Analyse-It program. This program

generates box plots that graphically show the median, upper and lower quartiles, inter-quartile ranges (IQRs) (middle half of the data), and overall spread for parametric and non-parametric statistics; the organic component ratios and inorganic anion concentrations in soil are non-parametric variables. Outlying observations are identified by the program as either "near" (between 1.5 and 3.0 IQRs from the upper or lower quartile) or "far" (more than 3.0 IQRs from the upper or lower quartile) outliers; soil samples identified as far outliers will be considered to be statistically different. When variation in component ratios (HPLC) or amounts (IC) by visual inspection can be quantitatively confirmed as statistically significant, samples will be considered differentiable.

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Detection of Substituted PAH Residues by SPME in Arson Debris Analysis

FORENSIC TECHNOLOGY NEED

Weathered arson debris samples and/or complex matrix interferences may confound the detection of gasoline residues in arson debris. SPME provides a means of increased sensitivity in the pre-concentration of a wide variety of compounds including the aromatic hydrocarbons used in the determination of gasoline residues in arson debris. The majority of literature and discussion to date has focused on SPME-ACS comparisons from often a competitive viewpoint; however, this research demonstrates that SPME can be used in conjunction with the existing ACS protocols to form a more comprehensive analysis. While the ASTM accepts SPME as a screen tool for accelerants in arson debris, the technology could play a much more significant role in validation methods as an augmentation to traditional ACS protocols. This project is collaboration between the University of Wisconsin – Platteville and the Wisconsin State Crime Laboratory – Madison.

TECHNOLOGY DESCRIPTION

This project offers an in-depth assessment of SPME's usefulness in bridging the gap between the forensic community's capabilities in detecting gasoline accelerants in arson residues by current methods and the need for a reliable, inexpensive, readily available technology with improved sensitivity for aromatic compounds in an environment containing pyrolytically degraded polymers.

SPME is a widely accepted technique in the analytical community and is accepted by ASTM as a screening analysis for accelerants in arson debris. The focus of this research proposal is the assessment of SPME capability in three specific hypotheses. The first hypothesis is that SPME can accurately

and reliably detect C1-substituted naphthalene / C2-substituted naphthalene ratios in gasoline, evaporated gasoline, and a wide variety of complex arson matrices that includes polymeric material with high concentrations of aromatic monomers such as polystyrene and reproducibly distinguish these samples based on the ratio of these polyaromatics. The second hypothesis is that SPME can provide reliable detection of heavier semi-volatiles in gasoline such as anthracene as well as more highly substituted aromatics such as C5 alkylbenzenes. The third hypothesis is that a hybrid solvent extraction-fiber immersion SPME technique provides an optimal recover of heavier polyaromatics from complex matrices. These experiments represent three potential windfalls in advancing the trace evidence community's ability to detect aromatic compounds associated with gasoline in very difficult matrices.

The primary practical objective of this project is to provide an enhanced detection of gasoline residues in arson debris that may contain contaminants due to the pyrolytic decomposition of polymeric materials. The goal of this research project is to bridge the gap between the current status of techniques used in the analysis of arson residues and the need of the forensic community for method development for recovery of ignitable liquid residues from a variety of matrices as described in *Forensic Sciences: Review of Status and Needs* (1). This project bridges this gap by thoroughly assessing the applicability of a highly sensitive, pre-concentration technique known as solid-phase microextraction (SPME) for determining the presence of gasoline residues in a complex matrix of arson debris that includes high levels of pyrolytically degraded polymeric materials.

TECHNOLOGY BENEFITS

- Increased sensitivity for substituted polyaromatic hydrocarbons common to gasoline
- Augmented detection when combined as a screening tool with ACS protocols

ACCOMPLISHMENTS/STATUS

In a comparison of SPME and ACS on two manufacturer's GC-MS units, there are desirable utilities in each technique. The SPME is much more sensitive than the ACS method as previously reported in the literature. This sensitivity is compromised however by the relatively rapid saturation of the SPME fiber with aromatic and polyaromatic hydrocarbons. After initial experiments demonstrated that the optimal desorption time was 1.0 minutes in a GC inlet at 250°C, SPME fibers were optimally "loaded" with 1.0 hour headspace exposure. Longer periods of time such as "overnight" resulted in a loss of resolution caused by fiber saturation. Subsequent experiments demonstrated that the 100 μ m PDMS fiber was better suited than the 7 μ m PDMS ($p < 0.05$) for detection of polyaromatic compounds which ran contrary to one previous report in the literature.

There was an observed tendency for heavier aromatic compounds displacing lighter aromatic compounds with increased exposure times; however, at 1.0 hour fiber exposure there was no loss of the classic "crown" of alkylbenzenes with SPME. It should also be noted that the crown remains and there is a detection of C2, C3, and C4-naphthalenes (Figure 1) by SPME that is not present using ACS and overnight exposures (Figure 2). A similar phenomenon was observed with anthracene and C1-anthracene.

With respect to polyaromatic (PAH) peak ratios, the increased sensitivity of SPME did not carry over into better defined peak ratios (i.e. C1-naphthalene to C2-naphthalene) in a

consistent manner. Sample to sample variability was high when comparing the evaporated series of gasoline standards, and the introduction of a burn matrix further complicated straightforward attempts at quantification. As part of these experiments a wide variety of polymer standards were burned under various conditions (i.e. varied air intake). While these burns produced pyrolysis products that were chromatographic interferences, the mass spectral identification of these compounds did not indicate the formation of naphthalene, anthracene, or simple alkyl derivatives of either PAH.

Perhaps the most significant, encompassing result of this project is the potential for SPME to augment the trace evidence toolbox for arson debris analysis. The results of the SPME analysis of gasoline residue in various building material matrices demonstrated that in a vast majority of trials SPME did produce the "key" structural elements (i.e. "the crown" of alkylbenzenes in gasoline) **and** reliably detected more highly substituted forms of PAH in all spiked samples and many control burns.

One of the significant arguments against the use of SPME in crime laboratory validations is the cost of the fiber vs. the need to archive a sample. ACS method provides a rather inexpensive, easy-to-store archival sample (part of the strip or the strip itself). If SPME were to replace ACS, the cost of using a SPME fiber for one analysis would be impossible to meet; however, if SPME methodologies were validated and used as a screening/analytical tool specifically for PAH, SPME could provide a relatively affordable method to obtain potentially vital information in a portion of the difficult cases. Because samples falling under these criteria (confounded or inconclusive ACS analysis) may only be between 5-10%, the cost of archiving a fiber becomes less of an impact on the budget of a laboratory.

File : C:\MSDCHEM\1\DATA\S009.D
 Operator : JRW
 Acquired : 24 May 2005 14:07 using AcqMethod SPME1
 Instrument : Instrument
 Sample Name : 75% EVAP'D-UWP
 Misc Info : 100u PDMS/5 MIN EXP
 Vial Number : 1

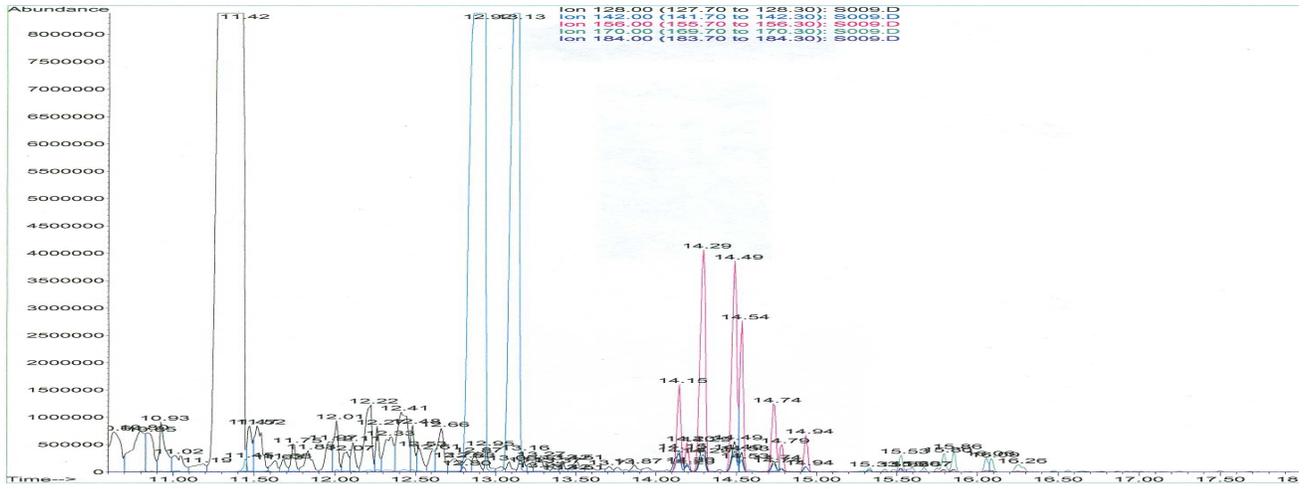


Figure 1. Naphthalene Detection and Resolution Via SPME (naphthalene at 11.42 minutes, C1-nap. At 12.00 min., C2-nap at 14.29 min, and C3-nap at 15.53 min).

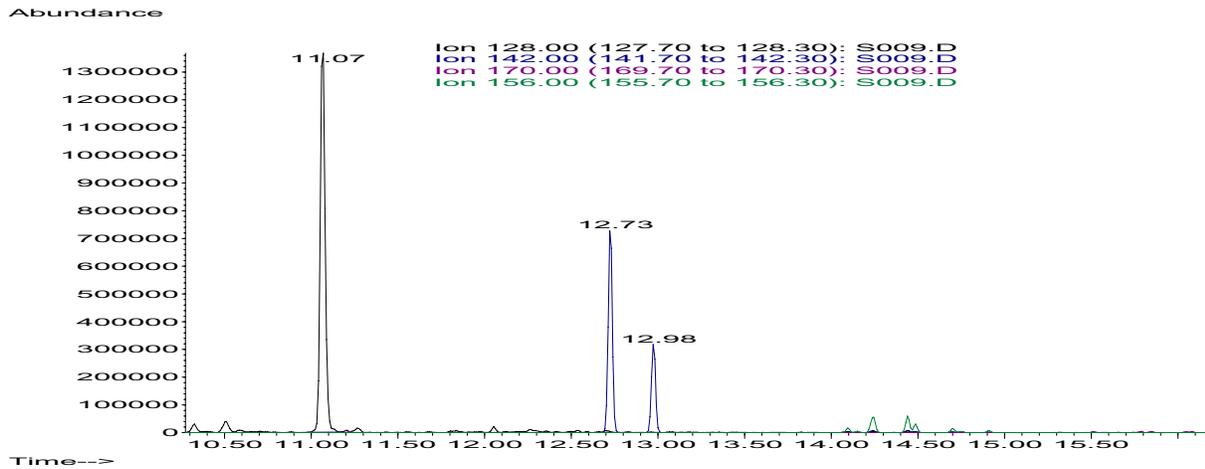


Figure 2. Naphthalene and C1-Naphthalene Detection and Resolution Via ACS

Figures 3 and 4 illustrate the usefulness of SPME in a complex matrix. These mass spectra clearly show C2-naphthalene in the gasoline (75% evaporate standard) containing asphalt shingle matrix whereas the compound is not present in the asphalt shingle matrix alone (n = 10). The ACS method (n = 8) could not provide sufficient analyte to detect the presence of gasoline in this environment.

Thus the primary conclusion of this work is that SPME can be used to effectively augment ACS methods employed in the vast majority of crime laboratories today. Further work needs to be done to ascertain the percentage of cases where this method would yield useful data, to search for a fiber coating that provides a more uniform retention of all molecular mass PAH, and to automate the technology.

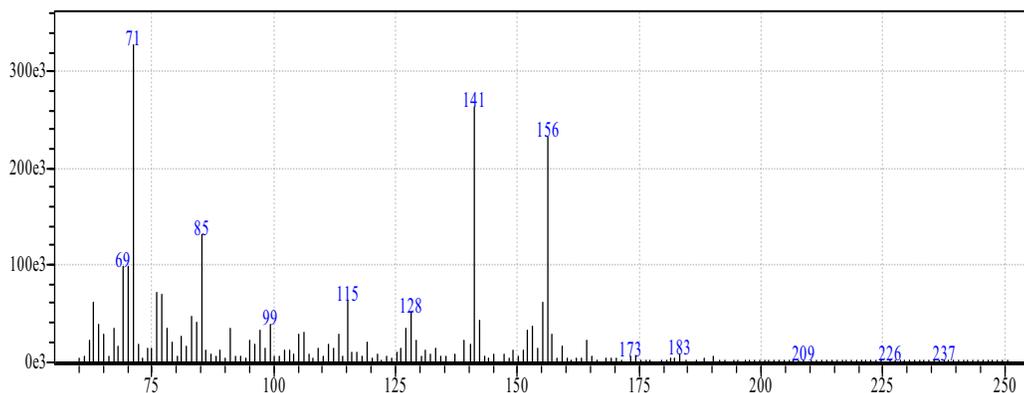


Figure 3. Mass spectrum of 16.65-minute retention compound from the analysis of gasoline and asphalt shingle pyrolyzate.

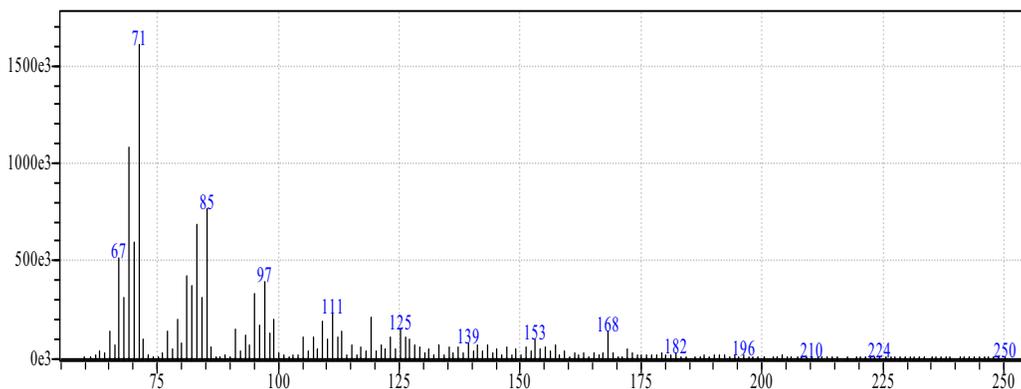


Figure 4. Mass spectrum of 16.65-minute retention compound from the analysis of asphalt shingle pyrolyzate.

COLLABORATION/IMPLEMENTATION

This project involves a collaboration between the University of Wisconsin – Platteville and the Wisconsin State Crime Laboratory – Madison. The project had a secondary success in advancing the overall collaborations of these two institutions. In addition to furthering the working relationship between Dr. Cornett and Dr. Wermeling (which resulted in planning for future projects in trace element analysis and further work in this arena) Dr. Cornett attending the Advanced Fire Debris Training Course offered by the Midwest Forensics Resource Center in August 2005. At this training course, there was ample opportunity to involve analysts from around the Midwest and course instructor Carl Chasteen in conversations related to SPME in arson debris analysis and this particular project.

PUBLICATIONS AND PRESENTATIONS

- Wermeling, J., Cornett, C.R., “ Detection of Substituted PAH Residues by SPME in Arson Debris Analysis “. Annual Meeting of the Midwest Association of Forensic Scientists, St. Louis, MO, October 5-7, 2005.
- Cornett, C.R., Wermeling, J., “Detection of Substituted PAH Residues by SPME in Arson Debris Analysis,” *Midwest Forensics Resource Center/Ames Laboratory Report IS-5177* (2005).

Project dissemination led to an oral presentation and undergraduate research poster at the Midwest Association of Forensic Scientists annual meeting in St. Louis, MO October 5-7, 2005. In addition, an oral presentation and research poster have been presented at the March 13-17,2006, Pittsburgh Conference in Orlando, FL. A manuscript is being prepared for submission to the Journal of Forensic Sciences.

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Development of a Rapid, HPLC-Based Intoxicant Screening Approach

FORENSIC TECHNOLOGY NEED

The overall objective of this work is to develop an ultra-fast, highly reliable gradient elution screening method for detecting and identifying drugs of abuse. The availability of such a screening technique will both significantly reduce the burden placed on the much slower and more expensive, harder to maintain LC-MS method, and it will provide valuable information to LC-MS operators so that they can set the system parameters to obtain more reliable information.

TECHNOLOGY DESCRIPTION

HPLC has become, over the past two decades, the dominant analytical methodology in the pharmaceutical industry due to its superiority to GC in avoiding the need for derivative formation; it is proving increasingly important in forensic drug analysis as attested to by the numbers of papers focusing on HPLC at the upcoming The Society of Forensic Toxicologists (SOFT) meeting. It is becoming a common technique in clinical toxicology. The specific aims of this work are:

- To develop a Rapid, HPLC Intoxicant Screening method (RLCIS) based upon our earlier Ultra-Fast Gradient Elution HPLC (UFGELC) work.
- To identify 2 or 3 alternative, highly stable reversed phase HPLC columns with different selectivity to be used for analytes which cannot be identified due to incomplete chromatographic resolution from endogenous materials.
- To apply chemometric techniques including various established forms of Principal Components Analysis (PCA) and Multivariate Curve Resolution Alternating Least Squares (MCR-ALS) as a means maximizing the use of the

data that will be obtained by the RLCIS approach. These techniques will be used to establish detection limits, identify specific analytes, which are insufficiently resolved (chromatographically and spectroscopically) from endogenous species and licit pharmaceuticals.

- To evaluate the discriminating power (DP) and mean list length (MLL) provided by the measured retention times of the target analytes, and assess detection limits for the target analytes using the optimized UFGELC method. Both the DP and the detection limits of the UFGELC methodology will be compared at the U of M and the Minnesota BCA to the much slower LC methods currently in use in forensic labs, as well as capillary electrophoretic screening methods under development at the Minnesota BCA.
- To determine the sensitivity and specificity of the methodology by evaluating real samples whose composition is known based on previous analysis using established techniques (e.g. LC-MS). After demonstration of adequate predictive capability, the methodology will then be transferred to the Minnesota Bureau of Criminal Apprehension Forensic Science Laboratory (BCA Lab), where the technique will be used more routinely in order to establish performance statistics.

TECHNOLOGY BENEFITS

A major virtue of this UFGELC methodology is that several retention standards can be run repeatedly throughout a day at very low cost

in time thereby greatly improving retention and thus identification reliability.

COLLABORATION/IMPLEMENTATION

This proposal involves collaborative work between Professor Carr's laboratory and Glenn Hardin's toxicology group at the Minnesota Bureau of Criminal Apprehension (BCA) Forensic Science Laboratory (St. Paul, MN). They have made exchange visits, communicate weekly and Glenn Hardin has participated in the Carr research group weekly meetings. The Carr group has visited the BCA on numerous occasions to discuss this work. After the initial work Carr's group will obtain a minimum of 100 real (but blind) samples which have been analyzed by Mr. Hardin's team using LC/MS or GC/MS for analysis by UFGELC.

Collaborative work will be published in the Journal of Forensic Sciences and Journal of Analytical Toxicology and presented at the American Academy of Forensic Sciences (AAFS) and The Society of Forensic Toxicologists (SOFT). Dwight Stoll of the University of Minnesota will work with the MN BCA toxicology group to transfer the methodology to them.

ACCOMPLISHMENTS AND ONGOING WORK

The first quarter of the project will begin by April 2006.

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Elemental Characterization of Automobile Body Fillers, Putties, and Caulk by Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry for Matching of Evidence

FORENSIC TECHNOLOGY NEED

Fillers, putties, and caulks are common in our everyday lives, yet very little can be found regarding the analytical characterization of these materials, either organic or inorganic. Automobile body fillers, putties, and caulking-type materials generally are comprised of 60% to 90% organic materials with the balance being inert, inorganic filler. These inorganic fillers may differ from different manufacturers and the composition of the fillers may contain trace inorganic impurities that could provide additional discrimination criteria for these material types.

The presence, absence, and relative abundance of elements in specific association patterns provides a unique means of display and comparison of the trace elemental signature for samples and is easily understandable. Elemental compositional analysis has frequently been considered as the best approach to classify and differentiate between samples that are similar. The implementation of laser ablation as a sampling technique prevents destruction of the sample by dissolution and extends this elemental analysis technique to much smaller sizes, typical of trace samples encountered in forensic cases.

TECHNOLOGY DESCRIPTION

This project involves the utilization of laser ablation-inductively coupled plasma-mass spectrometry as an analysis technique, which can differentiate samples of similar composition based on the unique trace elemental signatures. Additionally, criteria and protocols for the comparison and differentiation of these sample types, based on multivariate analysis techniques, will be

developed. Multivariate analysis techniques allow use of the full acquired mass spectrum without any prior knowledge of the chemical composition of the sample.

A number of trace elemental analysis methods could be used to characterize these materials. These methods include atomic absorption spectrometry, neutron activation, mass spectrometry, X-ray fluorescence, as well as solution nebulization-inductively coupled plasma atomic emission spectroscopy and mass spectrometry (ICP-AES and ICP-MS). Each method has its strengths, but only ICP methods have the precision, sensitivity, multi-element detection capability, and dynamic range suited for trace elemental analysis of forensic samples. Compared to ICP-AES, ICP-MS has better detection limits (a factor of 100 for many elements) and has the capability to provide isotopic information for most elements.

TECHNOLOGY BENEFITS

Although the majority of crime laboratories do not have the instrumentation used in this study, the results of this study will provide the framework for elemental analysis schemes employing other techniques and fill an apparent void in the literature on the inorganic analysis of these types of materials. Furthermore, this study will demonstrate another type of forensic sample that can be readily analyzed by LA-ICP-MS, further validating the technique in the forensic community. This project is an extension of previous work from the PI's laboratory, the development of LA-ICP-MS as a technique to analyze and discriminate forensic glass fragments (see Publications).

ICP-MS provides a high level of discrimination due to excellent detection limits (10-100 times better than AES), practically unlimited element range coverage, and isotopic information. Laser ablation-ICP-MS is rapid, eliminates the need for extensive sample preparation, and is virtually a nondestructive technique allowing for the possibility of the questioned samples to be further analyzed by corroborative techniques. Furthermore, laser ablation promises to increase the number of analytically useful elements detectable by standard ICP-MS techniques by eliminating problems with some elements due to poor dissolution and contamination. Additionally, smaller samples may be analyzed making the technique applicable to more cases.

Sample comparisons in this project are done by using multivariate analysis techniques, in particular principal component analysis (PCA). PCA allows the use of the full mass spectrum without requiring any pre-selection or elimination of elements. PCA is a multivariate data reduction method that examines the variance patterns within a multidimensional dataset. PCA reduces the dimensionality of the dataset to a few simple variables while retaining a major portion of the mass spectral information. These new variables are then used for sample comparisons and to derive a statistical significance for the analysis.

COLLABORATION/ IMPLEMENTATION

The analysis protocols being developed for this project are based on a previous MFRC project evaluating LA-ICP-MS for glass analysis. This project is being carried out in collaboration with the Wisconsin State Crime Laboratory, Milwaukee, which is providing guidance in the development of sampling and analysis protocols for these types of samples. The developed technique and protocols from the project will be made available to all MFRC partners for casework assistance

PUBLICATIONS AND PRESENTATIONS

- Baldwin, D. P., Houk, R. S., Bajic, S. J., "Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry of Forensic Glass Samples –Final Report," MFRC/ Ames Laboratory Report IS-5163 (2004).

ACCOMPLISHMENTS AND ONGOING WORK

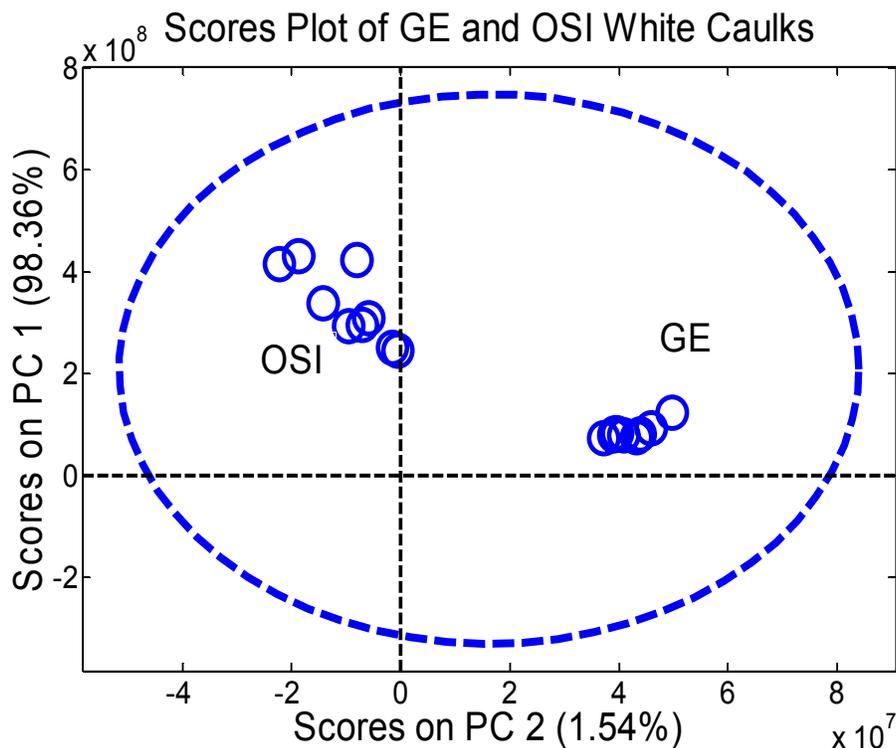
Work to date has focused on establishing optimum laser ablation parameters for analysis and sample presentation for fillers and caulks and establishing an analysis protocol. Survey studies of several caulks and body fillers were conducted to investigate any potential issues or effects during the curing process of off-gasses of volatile organics on analysis. Work continues on establishing which elements can be used for comparison and differentiation of the different sample types and determining the inorganic compositional variations between manufacturers.

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This plot illustrates the Principal Component Analysis (PCA) of the mass spectra acquired by LA-ICP-MS from two white caulks from two different manufacturers that are not differentiable based on their color. The entire mass spectra (elemental fingerprints of the samples) are used in the analysis. PCA reduces the mass spectra to a few variables, which are used for sample comparisons. When these variables are plotted, the repetitions of the samples cluster together, but the samples occupy different areas on the plot indicating that the different caulk samples are of distinguishable elemental composition.

Fast Gradient Elution Liquid Chromatography for Rapid Screening of Drugs of Abuse in Blood

FORENSIC TECHNOLOGY NEED

Delivering competent analytical judgment on samples in a timely manner is becoming more difficult as the sample load in forensic laboratories continues to increase, while economic pressures do not permit a concomitant increase in staff size. These pressures are influencing the development of new analytical technologies, which can deliver high quality qualitative and quantitative information in a high throughput environment. Historically gradient elution HPLC with diode array detection was an often-used method for screening and even identification of drugs of abuse in biological samples because the hardware is relatively inexpensive and easy to run in comparison to mass spectrometric methods, has tremendous chemical selectivity, and can produce precise retention time data under well-controlled conditions; however, it is typically quite slow (20-30 minutes per run). A collaboration between Peter Carr at the University of Minnesota and Glenn Hardin at the Minnesota Bureau of Criminal Apprehension developed a technique for rapid screening of drugs of abuse in blood.

TECHNOLOGY DESCRIPTION

In gradient elution HPLC, the time required to complete an entire analysis (cycle time) before beginning subsequent analysis must include not only the time to increase the percentage of the strong solvent and cause elution of analytes from the column (gradient time), but must also include the time required to re-equilibrate the column with the initial solvent before beginning the next analysis. As the gradient time is made shorter to improve throughput, the re-equilibration time represents an increasing fraction of the total cycle time.

To reduce the required re-equilibration time, a standard HPLC system (P 1090) was augmented with an additional binary pump and 6-port 2-position valve. The modified system is shown schematically in Figure 1, where the function of Valve A is simply to allow delivery of the desired solvent composition from either of the two binary pumps A or B as dictated by the operator. The function of Valve B is to deliver the sample to be analyzed to the column head at the time of injection. Gradient elution in subsequent injections was performed by alternately selecting either pump A or B (using Valve A) as the pump delivering the solvent gradient. The first solvent gradient is delivered by pump A, followed by actuation of valve A to allow delivery of solvent from pump B to the column head. This arrangement allows immediate re-equilibration of the column with the initial eluent, without waiting for strong solvent from the first gradient to be flushed from the system. This greatly reduces the dwell volume of the system. Initial solvent is delivered from pump B through the column for a specified re-equilibration time, which is then followed by injection of the second sample and delivery of the second solvent gradient to the column using pump B. This process of delivering solvent gradients from alternating pumps can then be repeated as many times as necessary to complete the analysis of several samples.

TECHNOLOGY BENEFITS

A novel instrument configuration was used to significantly reduce the time needed to re-equilibrate the HPLC column between gradients runs, thereby reducing the total time for each complete gradient elution analysis. By developing Fast Gradient Elution HPLC with total analysis times ideally less than 3 minutes (acceptably less than 4 minutes) capable of providing content comparable to

the much longer (20-30 minutes) standard method. HPLC analyses are currently in use as screening tools. Included in this development process was the assessment of reproducibility of retention time for the target intoxicants.

COLLABORATION/IMPLEMENTATION

Most of this work was carried out in continual collaboration with Mr. Glenn Hardin of the Minnesota Bureau of Criminal Apprehension in Saint Paul, MN. They meet on a regular basis to exchange information. A paper will be submitted this quarter for publication. The work will be presented at the next Society of Forensic Toxicologists by Glenn Hardin.

ACCOMPLISHMENTS AND ONGOING WORK

By minimizing the HPLC instrument dwell volume and maintaining precise cycle times we are able to screen blood extracts for drugs of abuse using a gradient elution method with

an analysis time of 4.5 minutes per sample, including a gradient time of 2.5 minutes and a 2-minute column equilibration time. Compound identifications are made by matching diode-array data (spectra from 200-400 nm) and retention times against a spectral library constructed in-house using Chemstation software (Agilent). The average standard deviation in retention time for morphine, oxycodone, methamphetamine, benzoylecgonine, zolpidem, diazepam, alprazolam and amitriptyline was 1.2 seconds for 60 successive injections ranging from 2 to 200 ng of each drug. Limits of detection range from 3 to 17 ng, with the lower limit for spectral matching (at 95% spectral match) being closer to 17 ng. This strategy is easily implemented on standard HPLC systems with minimal modification and serves to greatly increase laboratory throughput while reducing solvent waste. The long term stability of the columns has been assessed and a novel retention index scheme developed to compensate for column drift and errors in eluent composition.

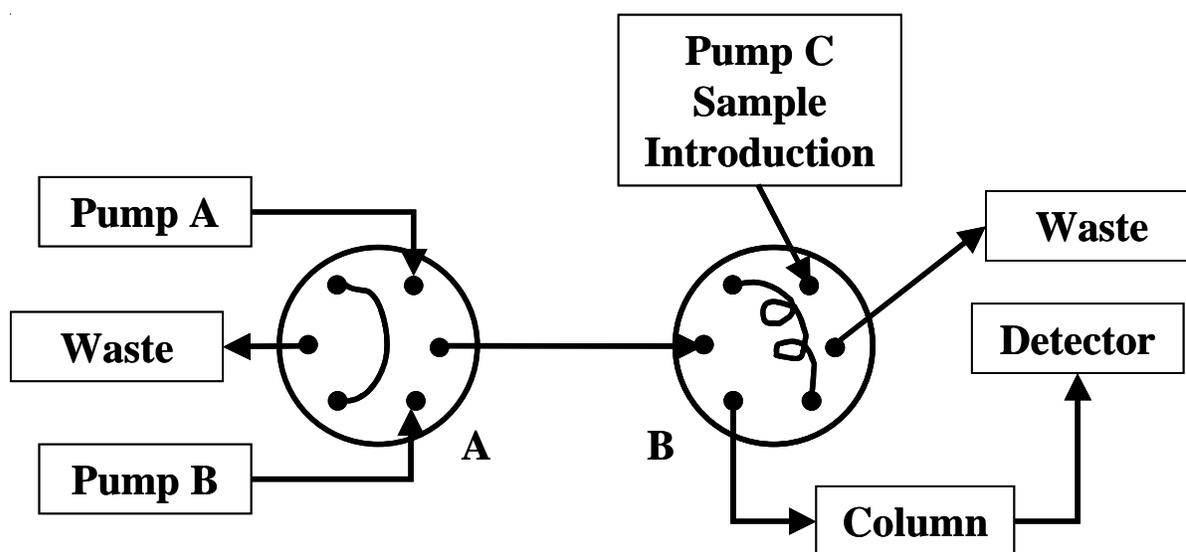


Figure 1. Schematic representation of a modified HPLC system that allows fast column re-equilibration between gradient analyses.

PUBLICATIONS AND PRESENTATIONS

- Fuller, KM, Stoll, DR, Steinkraus, LD, Pasha, MS, Hardin, GG, Carr, PW. "Fast Gradient Elution Liquid Chromatography for Rapid Screening of Drugs of Abuse in Blood". Annual Meeting of the Society of Forensic Toxicologists, Nashville, TN, October, 2005.
- Carr, P.W., Fuller, KM, Stoll, DR, Steinkraus, LD, Pasha, MS, Hardin, GG "Fast Gradient Elution Reversed-Phase HPLC with Diode-Array Detection as a High Throughput Screening Method for Drugs of Abuse," *Midwest Forensics Resource Center /Ames Laboratory Report IS-5179* (2006).

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Forensic Evaluation of Soils for Presence of Decaying Flesh

FORENSIC TECHNOLOGY NEED

Locating clandestine graves for forensic analysis is currently being solved in many ways. One primary method is with dogs, but the supply and availability of properly trained canines is limited. A handheld portable instrument capable of finding clues associated with clandestine graves could supplement or even supplant the use of dogs for this activity. Portable instruments ship much easier than dogs, required less maintenance, produce less waste, and can be potentially operated by someone with minimal training.

The proposed research concerns a new instrument to detect the presence and precise location of clandestinely buried bodies. Human decomposition produces a range of chemical by-products, which can serve as chemical signatures to the presence of human remains. Traditionally detection of such a broad range of chemicals was performed in a laboratory environment using complicated bench-top instruments such as GC-MS, HPLC, etc. This project is devoted to the development of a portable chemical analyzer for these chemical signatures using pyrolysis, low power portable sensors, and/or pattern recognition to detect and identify these chemicals. The final instrument will have both a Surface Sweep for the analysis of general surface area and Penetrometer for sub-surface analysis of specific locations. The Penetrometer can serve as verification of locations identified in the Sweep mode.

The expense associated with a full forensic exhumation is significant; this project aims to ensure that none of this labor will be wasted on empty sites or sites with non-human remains.

In this research the Sensor Research Group at Illinois Institute of Technology along with Scott Rochowicz at the Illinois State Police

Forensics Lab in Chicago and John F. Schneider and his group at Argonne National Labs, will investigate the best sensors for detecting the signature chemicals produced by decaying bodies. The identity of many of these chemical signatures has already been tabulated by Vass.

TECHNOLOGY DESCRIPTION

Many of the identified specific chemical products produced from the decomposition of decaying humans can be detected using chemical sensors, either directly or via an automated, controlled pretreatment (e.g., pyrolysis). A laboratory study will be performed to determine the optimal sensor systems for the detection of appropriate signature compounds. An array of sensors will be used as the detector for the proposed analyzer. Previous work by members of our group has shown that multiple sensors, with proper calibration and data processing, can be used to identify specific compounds or classes of compounds. Such a sensor-based analyzer is often called an "electronic nose". Figure 1 shows an instrument developed to perform near real time, in-situ detection of subsurface soil contamination of explosives. Conceptually, this probe will be similar, but will be able to be operated by a single user. The final product will use the best (clearest signal without interference) sensors in a custom electronic nose package.

On-site measurements using portable chemical analyzers can increase the reliability of locating buried human remains. Currently the best technology for locating clandestine gravesites is with forensic dogs. Other nondestructive methods include geographical tests such as ground penetrating radar or depression evaluation, botanical evaluation, and entomological evaluation.

By the end of Year 1, the investigators will have identified sensor types and operational parameters for the detection of appropriate chemicals. This information will guide the development of the actual field-portable instrument. The final product will be more portable and require less maintenance than dogs. Operationally, it will be easier to use than geophysical probes and will be less subjective than botanical and/or depression evaluations. It will insure that forensic exhumations are performed in the right place.

TECHNOLOGY BENEFITS

The overall objective of the project is to evaluate the feasibility of using chemical analysis to facilitate locating buried human remains and to ultimately develop a field portable chemical analyzer for this purpose. The analyzer is envisioned to have both surface sweep and subsurface probe capabilities. The technology can improve operating efficiency with a battery-powered, cane-size instrument with a handle to facilitate manual deployment beneath the soil surface.

Forensic exhumations are costly endeavors, and the cost would be incurred regardless of whether or not the suspect site contained human remains. Potentially significant cost

savings should be obtained with respect to the use of trained forensic exhumers simply by eliminating “false digs”.

Although it is not yet known what sensors will work best, the past experience with instrument development by members of the research team have produced similar instruments in the \$2000 to \$3000 range.

COLLABORATION/IMPLEMENTATION

Dr. William Buttner/IIT as project PI and will manage the administrative and technical project requirements. Dr. Joseph Stetter/IIT will provide technical assistance to Dr. Buttner. Dr. Stetter and Dr. Buttner manage the International Center for Sensor Science and Engineering at IIT and each have extensive experience in bringing innovative sensor systems to market in a timely, cost effective, manner and with developing field analytical methods. Dr. John Schneider (Argonne) has extensive field analytical chemistry experience and has appropriate clearance for working with restricted materials. Mr. Nathan Schattke has a joint appointment with Argonne and IIT and will perform most of the technical experimentation and design. He is a Chemical Engineer working towards a Ph.D. in Analytical Chemistry. Mr. Scott Rochowicz of

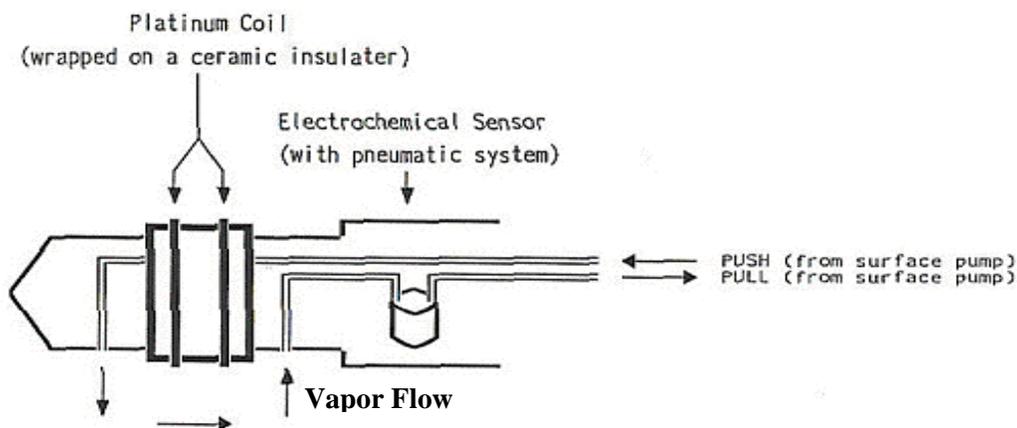


Figure 1. Probe used for subsoil detection of explosives that will serve as the concept of probe to detect chemical by-products of human decomposition.

the Illinois State Police Forensic Science Center at Chicago is interested in evaluating this technology and will provide critical technical input into the design requirements for the field portable Forensic Analyzer. Mr. Rochowicz will also help select sites for field deployments.

This concept will be marketed to the fifty state police offices around the USA, as well as other interested state, federal, and municipal agencies. This equipment is the type that will not require a manufacturing facility and long production runs, instead it will be a small number (less than 300) custom built units; the IIT SRG has a partnership with a small instrument-development company that could provide sensor-based instrumentation (Transducer Technology, Inc.). IIT will closely interact with Mr. Scott Rochowicz (Forensic Sciences Center at Chicago) to provide feedback and guidance on instrument design and to facilitate dissemination of this technology to the forensic community. Results of the laboratory testing will be published in the chemical and forensic science journals. Field testing performance will be published in a forensic journal and technical presentations at a forensic symposium are also envisioned.

ACCOMPLISHMENTS AND ONGOING WORK

The project will begin April 2006. The project is just being initiated pending implementation of the contract between IIT and the MFRC. Major project activity is expected to begin in the summer. Preparations have progressed in the following areas. The pyrolysis systems for laboratory investigations have been investigated. A literature review was performed cross correlating compounds on the list of chemical associated with human decomposition with the types of chemicals found in soil, thus identifying interferants and undesirable detection products.

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Identifying Co-ops and Farmers as Illicit Sources of Anhydrous Ammonia for Meth Makers

FORENSIC TECHNOLOGY NEED

The theft of liquid ammonia for illicit use in the manufacture of methamphetamine is increasing in Iowa and its neighboring states as well as in western states such as Washington and Oregon. At this point in time the majority of thieves steal anhydrous directly from co-op facilities or nurse tanks in the fields of farmers. However, we are now beginning to experience problems with employees of co-ops and farmers who are selling anhydrous to meth makers.

In general, meth makers do not have access to the sophisticated distillation equipment necessary to pre-purify anhydrous adulterated with the salts proposed here or the meth synthesis inhibitor we have discovered. A large part of the problem posed by ammonia thieves is that there are thousands of portable anhydrous ammonia storage tanks in farmers' fields plus many dozens of large (100,000 gallon) storage tanks at the co-ops, which are targets. Thieves have little regard for safety issues, removing the anhydrous ammonia with thermos bottles, gas cans, and sometimes simple insulated carriers.

TECHNOLOGY DESCRIPTION

The objectives of this project are:

- To develop a salt that is soluble in anhydrous ammonia that in very small quantities would make a detection compound when lithium, a key component of the illicit meth synthesis, is added.

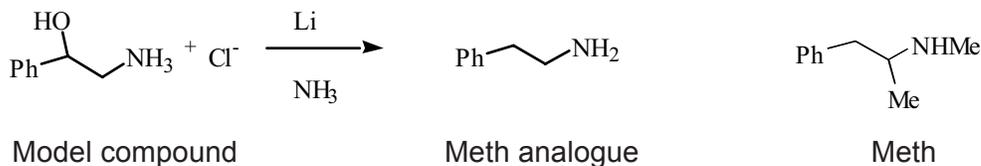
- To detect the detection compound liberated in the meth synthesis by High-Performance Liquid Chromatography (HPLC).
- To generate a variety of salts, each having a different HPLC signature for the detection compound. A given salt would then identify the co-op from which the ammonia was obtained.
- To develop salts that are not harmful to humans and farmers' fields.

TECHNOLOGY BENEFITS

In summary, if anhydrous ammonia were to contain an effective detection compound which rendered the methamphetamine produced by the lithium/ammonia reaction traceable, the theft and farm safety issues would be significantly diminished. The detection compound will be inseparable from the methamphetamine without very sophisticated and expensive equipment. In addition to identifying the detection compound, we need to be concerned with the effect of the detection compound on farmers' fields, since the vast majority of the anhydrous ammonia will be used for agriculture. This potential problem will be addressed in a broader proposal after a suite of viable candidate salts is established.

COLLABORATION/IMPLEMENTATION

The investigators remain in close contact with the Iowa Department of Criminal Investigation (contact: Nila Bremer) and the Iowa Meth Task Force (contact: Marvin Van Haften). Once they find an effective salt, the Iowa DCI



laboratory will subject the salt to the conditions used in the actual meth synthesis reaction (using ephedrine hydrochloride).

ACCOMPLISHMENTS AND ONGOING WORK

We have identified two salts which meet the criteria for detection in the model compound by HPLC. These salts are now in the hands of the Iowa DCI lab for testing in an actual meth synthesis.

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Long-Term Stability Studies of Liquid from Clandestine Methamphetamine Laboratories

FORENSIC TECHNOLOGY NEED

Maintaining evidence integrity is a pillar principle of any forensic science laboratory. The ASCLD/LAB accreditation process requires laboratories to prove the presence of laboratory controls which preserve the integrity of submitted evidentiary items. Generally, issues of integrity involve proper sealing and unbroken chains of custody; however, environmental conditions also play a vital role in the stability and integrity of evidence.

At the current time, very few long-term stability studies have been performed for evidence containing controlled substances outside of biological fluids. Specifically, there is a lack of data regarding the stability of methamphetamine and its precursors in solutions from clandestine laboratories. The Johnson County Sheriff's Office Criminalistics Laboratory (Mission, Kansas) will conduct a long -term stability study of liquids from clandestine methamphetamine laboratories in collaboration with five (5) additional forensic laboratories.

TECHNOLOGY DESCRIPTION

Solution stability data will be collected for liquid samples from various stages of methamphetamine production using the Iodine/Red phosphorus reaction scheme. Solution aliquots will be stored at four distinct storage conditions: Refrigerated (5°C), Ambient, Controlled Room Temperature (25°C/60% RH), and Elevated Room Temperature (40°C). Sample solutions will be pulled at defined time-points up to twelve (12) months and analyzed by gas chromatography – mass spectrometry (GC-MS) to determine concentration trends of ephedrine, pseudoephedrine, and methamphetamine. Results of the analyses will be used to

evaluate the precursor and final product stability based on specific sample storage conditions coupled with length of storage.

Results of the analyses will be used to evaluate whether the original levels of precursors and methamphetamine remain stable based on specific sample storage conditions coupled with storage time. Actual temperature/humidity trends of several forensic laboratory evidence lockers will be collected using continuous temperature/humidity circular chart records for up to 12 months.

Forensic scientists currently lack knowledge regarding the stability of clandestine laboratory solutions. Without this knowledge, there are significant ramifications to the testing and reporting processes. These include evidence awaiting analysis for long periods of time, which may significantly degrade and change the evidence in qualitative or quantitative composition. Forensic scientists' results from degraded samples could be successfully challenged.

TECHNOLOGY BENEFITS

The implications of the collected data on clandestine laboratory liquid sample storage are vast. The results will determine if precursors and final products degrade or convert over time based on the specific solution, the storage condition, and the time of storage. This will answer potential questions concerning the integrity of these solutions while in storage. The results will also determine if collected solutions require specialized storage (i.e., refrigeration) or expedited analysis. Finally, the results will also assist the forensic analysts in proper interpretation of data collected from solutions stored for extensive periods of time following collection and submission.

Table 1: Specific solutions and their respective storage conditions to be studied.

Component	Matrix	5°C	Ambient	25°C/60% RH	40°C
Pseudoephedrine (pure)	Deionized water	X	X	X	X
Pseudoephedrine (pure)	Methanol	X	X	X	X
Pseudoephedrine (tablet)	Deionized water	X	X	X	X
Pseudoephedrine (tablet)	Methanol	X	X	X	X
Pseudoephedrine (liquid capsule)	Aqueous (high pH)	X	X	X	X
Pseudoephedrine (liquid capsule)	Aqueous (high pH) and Diethyl ether	X	X	X	X
Ephedrine (pure)	Deionized water	X	X	X	X
Ephedrine (pure)	Methanol	X	X	X	X
Ephedrine (pure), Iodine, Red Phosphorus	Deionized water	X	X	X	X
Pseudoephedrine (pure), Iodine, Red Phosphorus	Deionized water	X	X	X	X
Methamphetamine, residual reaction products (unfiltered)	Aqueous (low pH)	X	X	X	X
Methamphetamine, residual reaction products (filtered)	Aqueous (low pH)	X	X	X	X
Methamphetamine, residual reaction products (filtered)	Aqueous (high pH)	X	X	X	X
Methamphetamine, residual reaction products (filtered)	Aqueous (high pH) and Diethyl ether	X	X	X	X
Methamphetamine free base	Diethyl ether	X	X	X	X
Methamphetamine HCl	Diethyl ether	X	X	X	X

COLLABORATION/IMPLEMENTATION

The Johnson County Sheriff's Office Criminalistics Laboratory (Mission, Kansas) will conduct a long-term stability study of liquids from clandestine methamphetamine laboratories in collaboration with the Kansas Bureau of Investigation in Topeka, Kansas; the Kansas City Police Department in Kansas City, Missouri; the Missouri State Highway Patrol in Jefferson City, Missouri; the Wisconsin State Crime Lab in Milwaukee, Wisconsin; and the Western Forensic Law Enforcement Training Center in Pueblo, Colorado. The temperature/humidity trends of evidence storage facilities at these other forensic laboratories are valuable data. The sponsoring laboratory and the five (5) additional forensic laboratories will maintain a temperature/humidity circular chart recorder in their respective evidence storage facilities for the entire duration of the twelve (12) month study. These recorders will document the actual temperature/humidity conditions of evidence storage facilities over the course of a year. The data will show trends, fluctuations, and other individual events that affect temperature or humidity. It is anticipated that temperature fluctuations will be extreme in some labs based on the fact that a good number of crime labs do not incorporate HVAC systems into their evidence storage facilities.

The results of the research will be disseminated through presentation at forensic meetings of the Midwestern Association of Forensic Scientists and the Clandestine Laboratory Investigating Chemists Association. The research will be submitted for publication in the Journal of Forensic Sciences or other appropriate scientific journal.

ACCOMPLISHMENTS AND ONGOING WORK

The first quarter of the project began February 24th.

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Micromechanical Cantilever (MC) based Sensors for Chemical Species Detection

FORENSIC TECHNOLOGY NEED

Successful completion of this research will help advance the mission of MFRC towards development of new and improved instrumentation capable of sensitive detection and drug identification. Current sensor systems require extensive sample preparation or specialized instrumentation to identify molecules of controlled substances such as cocaine with high specificity and sensitivity. MC-based sensors will provide an invaluable tool for forensic science because of its portability, capability for detection, and capability for identification with high sensitivity and specificity. Successful completion of the project will buttress our research efforts towards developing portable sensors for chemical species detection by integration of receptor layer coated micro-cantilevers and high resolution interferometry into a single microfabricated chip.

TECHNOLOGY DESCRIPTION

In the proposed work, we will develop a proof-of-concept for our sensing approach by building a MC-based sensor that is functionalized with aptamer molecules for sensitive and specific detection of cocaine molecules. Accordingly, the specific aims are to:

- Develop robust miniature sensors for high-resolution measurement of surface stress associated with formation of affinity complexes on the sensitized surface of micro-cantilevers. Figure 1 shows the schematic of a surface stress sensor.
- Functionalize micro-cantilevers with an aptamer that is capable of sensitive and specific detection of cocaine molecules.

- Characterize the sensitivity and specificity of cocaine detection with MC-based sensors functionalized with suitable receptor layers of aptamer molecules.

TECHNOLOGY BENEFITS

Successful completion of the research will serve to advance the mission of MFRC towards development of new and improved instrumentation capable of sensitive detection and drug identification. In addition, successful demonstration of this sensing approach will set the stage for rapid development of MC-based sensors for a wide variety of molecules of forensic interest including detection of DNA, explosives, and toxic chemical species. Development of MC-based sensors will be an important break-through for forensic science

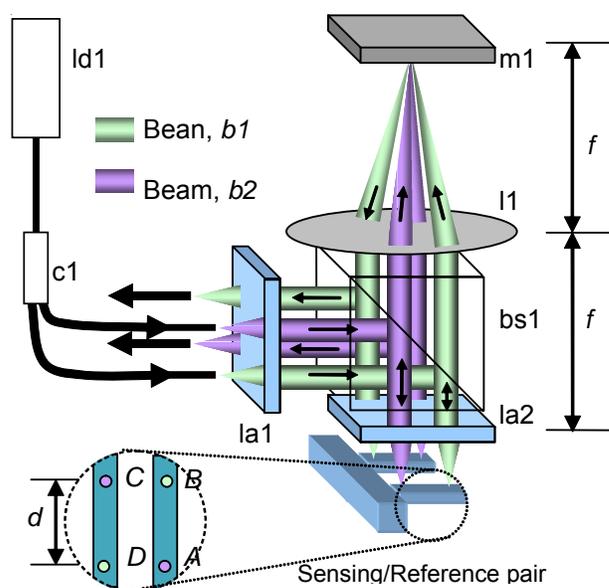


Figure 1: Schematic of surface stress sensor.

because of the tremendous sensitivity that can be achieved by aptamer-coated micro-cantilevers and the specificity imparted by aptamers for identification of chemical species at concentrations of parts-per-billion. Such sensitivity will be revolutionary for forensic analysis of controlled substances, explosives, toxic species, biological molecules, and DNA matching required for crime-scene identification.

COLLABORATION/IMPLEMENTATION

The PI's will work with MFRC to develop partnerships with partner crime laboratories and share the developed sensor technology. As a part of this effort, they have established contact with Dr. James Siefert (Forensic Science Division, Michigan State Police, email: siefertj@michigan.gov and ph: 989-777-9300) and will explore the possibility of establishing a collaboration in order to leverage his expertise in the area of controlled substance identification for the sensor development. The investigators will utilize the results of this project to seek further funding from NIJ, NSF, DOE and the Homeland Security Advanced Research Projects Agency for development of portable MEMS sensors.

Results of the research will be disseminated through peer-reviewed journal publications in the Journal of Forensic Sciences and presentations at annual meetings of American Academy of Forensic Science (AAFS) and Midwestern Association of Forensic Scientists (MAFS). In order to facilitate wide dissemination of research finding in the forensic community, the investigators will work with the MFRC to share the developed sensor technology with partnering crime laboratories. The long term goal is development of inexpensive, portable, and precise sensors that will be extremely useful for rapid forensic detection and identification of chemical and biological species. The development of MC-based sensors will be accomplished by integration of receptor layer coated micro-

cantilevers and curvature interferometry into a single microfabricated chip.

ACCOMPLISHMENTS AND ONGOING WORK

The first quarter of the project (February 1, 2006 to April 30, 2006) has been dedicated to developing the aptamer molecules for sensitivity and specificity for detection of cocaine, as well as preliminary design of the micro-cantilever system.

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Trace Metal Analysis of Ecstasy by Microwave-Assisted Digestion and Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES)

FORENSIC TECHNOLOGY NEED

A common technique among drug enforcement officials for obtaining information about and combating the illegal trafficking of drugs of abuse is known as signature profiling. In this method, an appropriate analytical method is employed to determine the composition of certain characteristic impurities in a seized drug sample, and then attempts are made to match more than one sample to a common origin—a manufacturer or a dealer, for example. In light of the dangerous effects of the drug ecstasy and the prevalence of its trafficking and use, the ability to gather intelligence that might lead drug enforcement authorities to a manufacturer or a dealer is of paramount importance. This technology, if effective, would be directly applicable in the effort to combat this dangerous and illegal drug in the United States and abroad.

This project continues a trace metal profiling project previously, employing more sophisticated chemometric methods of data analysis and attempting to establish a rational scientific basis for variations in trace metal content of ecstasy tablets. The synthetic work was performed in a synthetic organic chemistry laboratory at Southeast Missouri State University. The trace metal analysis of the resulting products, homologs of MDMA, was performed using ICP-OES in our atomic spectroscopy laboratory. Microwave-assisted digestion of seized illicit ecstasy tablets is currently being performed in the Southeast Missouri Regional Crime Laboratory with analysis following in the atomic spectroscopy laboratory. The results of this work will be communicated in the forensic literature, presented at professional meetings, and otherwise shared with other researchers in the field. The findings of this study have the greatest potential for impact at the federal

level and could prove useful to the United States Drug Enforcement Agency as an intelligence tool.

TECHNOLOGY DESCRIPTION

Trace elements in ecstasy samples obtained from law enforcement agencies are being determined by inductively coupled plasma-optical emission spectrometry using microwave-assisted digestion. Elemental patterns or signatures are being analyzed for the purpose of comparing and differentiating between ecstasy samples. The emission data will be analyzed using statistical multivariate methods, such as principal component analysis, in order to determine if the results allow for discrimination between different categories of samples. In addition, ICP-OES of an unscheduled ecstasy homolog synthesized in the lab has been performed to determine if samples from the same preparative batch can be positively linked together and distinguished from separate batches based upon their trace metal profiles. This second dimension of the project attempts to establish the scientific underpinning for trace metal profiling as a method of discriminating the origins of ecstasy samples.

A great deal of past attention and effort has been dedicated to the task of creating a database of all known ecstasy tablet logos as a means of tracking traffic of the drug. This method may be seriously limited in that it does not take into consideration variations between synthetic batches of ecstasy that are ultimately pressed using the same pill press. Another common technique among drug enforcement officials for obtaining information about and combating the illegal trafficking of drugs of abuse is known as signature profiling. In this method, an appropriate analytical

method is employed to determine the composition of certain characteristic impurities in a seized drug sample, and then attempts are made to match more than one sample to a common origin—a manufacturer or a dealer, for example. In the past several years, the main thrust of effort toward that end has been in examining the organic impurity profiles of ecstasy tablets in efforts to determine common origins or synthetic route of production. Since only two main synthetic routes predominate the production of MDMA, this approach is limited in the discriminating information it can provide about a sample of the drug. The project approach which has received a dearth of attention, is the examination of trace metal impurity profiles for seized ecstasy samples.

TECHNOLOGY BENEFITS

The technology being developed in this project offers the potential capability to state with confidence whether or not a number of ecstasy tablets were manufactured in the same synthetic batch and/or by the same synthetic method employing the same chemical reagents. The capability to link two or more seized ecstasy tablets together would be a powerful tool in the hands of drug enforcement officers and forensic analysts. The results thus far indicate that such statements can be made.

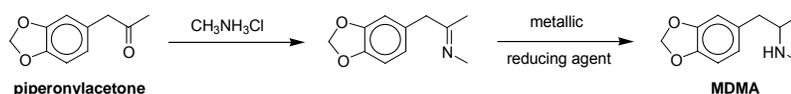
The development of a rational scientific foundation for the trace metal profiling method

would meet the need of forensic scientists to be able to defend their findings in a court of law under the Federal Rules of Evidence, the so-called Daubert admissibility standard.

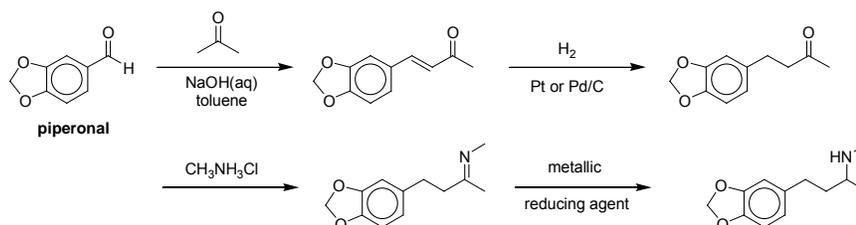
COLLABORATION/IMPLEMENTATION

This project is being carried out in collaboration with the Southeast Missouri Regional Crime Lab in Cape Girardeau, Missouri, and the Drug Enforcement Agency (DEA). In order to obtain samples from the DEA, a DEA research license was applied for and obtained during 2005. Samples from the DEA are received by and stored at the Crime Lab for security and legal purposes, and all microwave digestions are done there prior to trace metal analysis at the Department of Chemistry at Southeast Missouri State University. Dissemination of this research will be conducted through publication in peer-reviewed journals and presentations at professional meetings. Results will be shared with interested parties at other universities, crime labs, the FBI, the DEA, and MFRC member organizations. For example, the principal investigators have opened a dialogue with researchers at Michigan State University who are active in this research area. They intend to establish a collaboration in which the results of their work are shared in order to gain additional insights into this problem and to avoid unnecessary duplication of work. This collaboration was initiated through the DEA and the MFRC as a direct result of the MFRC Forensic Research and Development program.

MDMA Synthesis by the Reductive Amination Route from Piperonylacetone



Synthesis of an N+1 Carbon Homolog of MDMA by a Reductive Amination Route from Piperonal



ACCOMPLISHMENTS AND ONGOING WORK

This project is ongoing and will continue beyond the grant period. Several approaches are being used for this work. The first involves the trace metal analysis via inductively coupled plasma-optical emission spectrometry (ICP-OES) of an MDMA homolog synthesized in the lab to determine if samples from the same preparative batch can be positively linked together and distinguished from separate batches based upon their trace metal profiles. Trace metal concentrations were analyzed in a synthetic N + 1 homolog of MDMA, 4-(3, 4-methylenedioxyphenyl) butan-2-methylamine. This derivative of MDMA was prepared via the reductive amination synthesis using different reducing agents: aluminum-amalgam, sodium borohydride, sodium cyanoborohydride, and hydrogen over palladium, platinum, and platinum oxide.

Samples of the synthesized product from each reductive route were digested in acidic media and analyzed using inductively coupled plasma-optical emission spectrometry (ICP-OES). The emission intensities of all elements tested in each sample were then subjected to principal component analysis to determine if the results allow for discrimination between the reductive routes. These results thus far demonstrated that samples could be distinguished by reducing agent with a 90% confidence interval based on the concentrations of titanium, aluminum, and sodium observed in the samples. Trace metal analysis has not yet been completed for the hydrogenation reactions using metal catalysts.

Although additional analyses will be carried out on synthetic samples produced in Southeast Missouri State University labs, the major focus of our continued efforts will be the trace metal analysis of ecstasy samples received from the DEA. The first samples from the DEA were received late in 2005. Currently, 200-300 samples have been received. These samples were obtained from

different seizures, and contain a variety of different ecstasy tablet logos. Work is currently underway to analyze the trace metal contents of these samples via ICP-OES using microwave-assisted digestion. Although this work is not yet complete, The results do seem to indicate that certain metals are present in distinctive concentrations for ecstasy tablets from the same seizure. Once enough emission data have been generated, principal component analysis will be applied in order to determine if the results allow for discrimination between different categories of samples.

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AN ARTIFICIAL NEURAL NETWORK FOR WAVELET STEGANALYSIS

FORENSIC TECHNOLOGY NEED

Steganography is the science of embedding a message into an innocent file, so that there is no obvious trace that a secret message is hidden there. Steganography software can be used for illicit purposes to hide or transmit sensitive information. Steganalysis is of increasing importance to cyber security as it allows analysis of suspicious data to confirm or deny hidden message content. This project, conducted as a joint project between the Mathematics Department at Iowa State University and the MFRC in Ames, Iowa, developed new steganalysis software that allows message detection in currently used data types as well as data types that will become more common in the future, namely, in JPEG2000 image files. JPEG2000 is not yet widely used, but its use is expected to become more prevalent in the next 2-5 years. The software package produced by this project shows proof of concept that with steganalysis methods this new data type can be successfully detected, using a modular pattern recognition system, the artificial neural network (ANN). It will thus prepare cyber forensics personnel with tools for analyzing this new data type as it becomes increasingly prevalent and also allow additional analysis methods to be added into the ANN system. Mr. Steve Bogle, Assistant Director of Iowa Division of Criminal Investigation, will beta test the software in his labs.

TECHNOLOGY DESCRIPTION

The steganalysis technique under development utilizes a pattern-recognition system called an *artificial neural network* (ANN). An ANN is a tool that distinguishes between two classes of objects. The ANN is first trained by feeding it a large set of objects of each of the two classes. Through the

training, the ANN develops a rule for distinguishing the classes. Once trained, the neural net can apply its rule to candidate objects and classify them.

In the present project, the objects to be classified are images. One class contains innocent images, the other, stegoimages. The tool being developed will be a software application that runs on an ordinary personal computer. The user will start the program, select the image file to analyze, and the software will determine whether the image contains hidden data. However, this tool will not attempt to extract hidden data.

There are few steganalytic tools available at the present time. The commercial packages on the market use algorithms that are proprietary and secret, so it is difficult to assess their effectiveness. Furthermore, these tools are based on older technology and do not detect the most recent steganographic techniques. In particular, they are not designed for images encoded in jpeg2000 format. Our tools are intended to show proof of concept that both of these shortcomings can be overcome.

TECHNOLOGY BENEFITS

The technology provided by this software package shows that analysis of new data types (JPEG2000) can be successful. The software package also analyzes several current data types and current hiding algorithms using the ANN. The ANN is a modular analysis system, and new analysis methods that arise in the future can be added onto the ANN system in a modular way. Old ones can also be removed or modified. The state of the art in forensic practice is advanced by this software because currently available software does not seem to provide analysis techniques for the new data types, nor the modularity of analysis tools.

COLLABORATION/INNOVATION

Most of the coding for this project has been completed. Only the GUI front-end remains to be written. Furthermore, almost all of the computation required for training the neural net is finished. We have been in touch with Steve Bogle of the Iowa DCI who has offered to help field-test the software.

PUBLICATIONS AND PRESENTATIONS

- Davidson, J., Bergman, C., and Bartlett, E. "An Artificial Neural Network for Wavelet Steganalysis," *Proceedings of SPIE - The International Society for Optical Engineering, Mathematical Methods in Pattern and Image Analysis* 5916 (2005): 1-10.

ACCOMPLISHMENTS AND ONGOING WORK

At this time, all of the data for training and testing of the neural net has been created. This involved running several different steganographic tools on a starting set of images, and for each original image, creating 8 stego images. This resulted in a data set of over 10,000 images. One nontrivial aspect of this task was the creation of a simple steganographic tool that operates in the wavelet domain, anticipating stego techniques that will target jpeg2000 images. All of the feature-extraction routines have been coded and the code run on the training images.

Some preliminary tests were run on a subset of the training data and announced in the paper mentioned above. In this test the neural net recognized 92% of the stegoimages and flagged 10% of the "innocent" images as being suspicious. This amounts to a false negative rate of 8% and a false positive rate of 10%. This is at least as good as results announced by other researchers.

Mathematically, we should achieve even better results using the full set of features.

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Development of Forensic Imaging Technique for Fast Analysis of Magnetic Tapes with High Spatial Resolution

FORENSIC TECHNOLOGY NEED

Currently video cameras are widely used as tools for recording criminal acts. Due to recent enormous advances in analog and digital editing technologies, digital editing or fabrication of videotape evidence has become possible. Today fine magnetic particles or ferrofluids are used to visualize any artificial alteration on magnetic tapes such as tape recorder 'start' and 'stop' marks and over-recordings. However, the use of magnetic particles and ferrofluids is time consuming, inaccurate, and can easily cause contamination or even damage on the surface of tape evidence.

R.C. Sanders at the Wisconsin State Crime Laboratory's Forensic Imaging Unit and S.J. Lee at Iowa State University's Center for Nondestructive evaluation are collaborating to develop an improved technique for analysis of magnetic tapes.

TECHNOLOGY DESCRIPTION

This project is developing a new forensic imaging technique which will allow fast and accurate analysis of magnetic tapes using a transparent magnetic garnet film. The garnet film replaces the magnetic particles or ferrofluids for visualization of magnetic fields and gives a non-invasive method of detection with a high spatial resolution and fast inspection time. The forensic imaging technique (shown in Figure 1) will be based on a magneto-optic (MO) sensor film, which allows fast analysis of magnetic tapes with a high spatial resolution. When placing a transparent magneto-optical film onto a magnetic tape such as audio or video tape, the recorded patterns on the tape will magnetize the magneto-optical film up or down locally in accordance with the tape recording. This domain magnetization pattern can be readily visualized using the Faraday Effect and the image can be recorded using a CCD camera for storage and processing.

This technique will have significant impact on forensic analysis on magnetic recording media by offering a nondestructive method for imaging magnetic pattern with improved spatial resolution and speed.

TECHNOLOGY BENEFITS

When video evidence is damaged by cutting it into strips, this technique would be useful in visualizing the recording to properly align the strips so they can be spliced back together to be able to play the damaged videotape. At the present time this requires the use of a magnetic tape developer such as Kyread dip, which is basically a fine powder suspended in a liquid. This is a potentially destructive and time consuming process.

COLLABORATION/IMPLEMENTATION

S.J. Lee has expertise in magneto-optic measurements, in particular measurement techniques for nondestructive evaluation applications, and instrumentation for signal processing and equipment control. Lee has developed a magneto-optic spectrometer for investigation of magneto-optic properties of rare-earth transition intermetallic compounds for his Ph.D. thesis at the magneto-optic laboratory in the Department of Physics of Iowa State University. He has acquired a broad knowledge of magnetic particle inspection techniques and advantages and disadvantages of the techniques.

This project will be carried out in collaboration with R. C. Sanders, who is the Forensic Imaging Unit Leader of Wisconsin State Crime Laboratory. R.C. Sanders has extensive expertise and experience in forensic analysis of tape media. He will provide test samples for validation of the proposed technique and will visit Ames Laboratory to discuss the research plan and to assess research progress. Sanders will also be responsible for evaluating

the proposed magneto-optic techniques, developing strategic plans for technology transfer through conducting field trials at forensic laboratories, and developing commercial instrumentation based on the proposed technique.

The investigators in coordination with the forensic laboratory partner will develop and publish a validation protocol that individual crime laboratories can utilize for ASCLD-LAB required validation testing of new procedures and equipment, as well as quality control tests that can reasonably be performed by a forensic scientist before examining evidence to verify that the equipment and procedures performed as expected with a known standard. The results of this work will be disseminated in international journals in forensic sciences in particular *the Journal of Forensic Sciences* and *Forensic Science International*, as well as in journals of nondestructive evaluation and magnetics research such as *Review of Progress in Quantitative Nondestructive Evaluation* and *IEEE Transactions on Magnetics*.

ACCOMPLISHMENTS AND ONGOING WORK

The first quarter of the project is underway. Work has been dedicated to building the

proposed MO imaging system is shown in Figure 1. This system consists of two main components: a magneto-optic garnet film and the optical source and detection system. In order to make a user friendly system, we will develop an imager stage equipped with a film holder and a tape mount. A magnetic tape located under the film will be mounted on a linear x-y motion stage for alignment.

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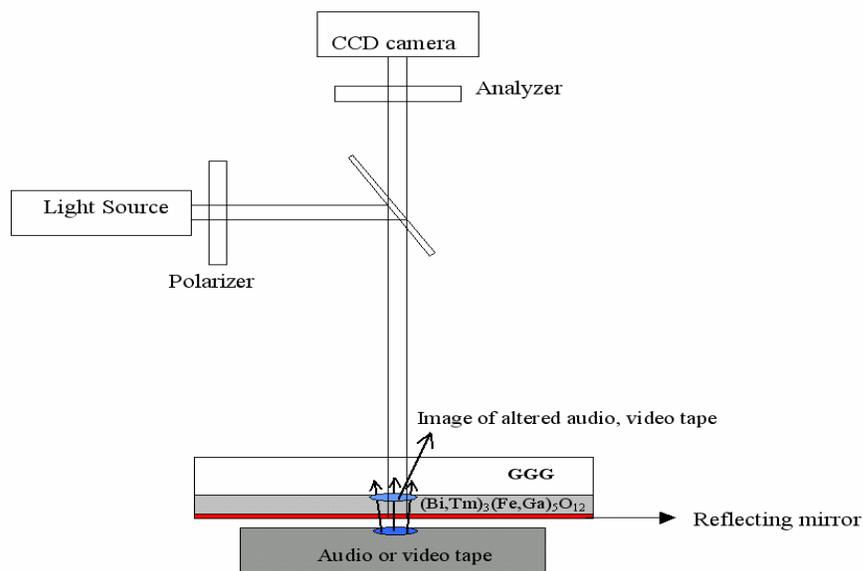


Figure 1. A schematic diagram of forensic magneto-optic imaging system.

Application of a Crystal Orientation Method to Forensic Physical Matching of Metal Surfaces along a Fracture Line

FORENSIC TECHNOLOGY NEED

Examples of metal fragments found at a crime scene include knife tips, pieces from weapons, pry tools, or automobile parts such as antennas or trim pieces. A physical match between two surfaces is routinely viewed as definitive proof that the fracture surfaces were generated from the same part. In the AFTE Journal, Summer 2005 issue, Katterwe indicated that there were current “trial challenges” to the practice of fracture matching examinations by tool mark examiners. There has been little systematic effort to establish a basis for the uniqueness of a physical match of surfaces. Rocky Stone in the AFTE Journal, Fall 2004 issue, presented a mathematical probability model with a random surface generated to duplicate the complex nature of a fracture surface. However, he indicated that the calculations did not account for the class characteristics that are present in actual fractures such as the nature and the characteristics of the alloy that would further individualize a particular fracture.

There is a need for a method to measure the nature and characteristics of materials with a known error rate that can be applied to performing physical matches. This study will examine the feasibility of using surface crystal orientation to associate, or differentiate, metal fracture fragments. This study will investigate the feasibility of using Electron Backscatter Diffraction (EBSD) in combination with conventional forensic methods to physically associate broken surfaces of metal pieces to determine whether the broken pieces were at one time one piece. This study may provide an additional method to establish individual characteristics based on atomic arrangements, and it is possible that the high degree of resolution that our instruments will bring to bear on this problem may contribute

new information to the ongoing debate about the uniqueness and characterization of fracture patterns. This work is being conducted at the Ames Laboratory in collaboration with M. James Kreiser, retired tool mark examiner from the Illinois State Police.

TECHNOLOGY DESCRIPTION

The overall objective of this study is to evaluate whether surface crystal orientation can be used to associate metal fracture fragments. This study will test if the orientations of the fractured crystals across the fracture plane for two surfaces, determined by a forensic examiner by conventional methods, is in fact unique and can be relied on to determine that the two pieces sharing the fracture plane were at one time one piece.

This study will examine steel knife fractures. Test pieces will be made using three alloys of carbon steel such as 1060, stainless steel 440C, and hardened tool steel O1. For the sample size there will be several hundreds of crystals to measure assuming an average crystal size of 20 μm . The samples will be fractured with Instron fixtures. The test knife samples will be fractured by wedging the samples into a fixture and fracturing the metal. The load type will be both tension and bending stress to failure.

The first part of the study will examine known fracture matches to test the orientation analysis. In the second part of this study, the set of fractured pieces will be physically matched followed by orientation analysis. Forensic examiner, M. James Kreiser, will collaborate with the investigators on the sample preparation and physical matching of fracture specimens. Mr. Kreiser will perform the preliminary match of the test fracture pieces prior to the EBSD testing. If casting of surfaces is required, a high quality casting

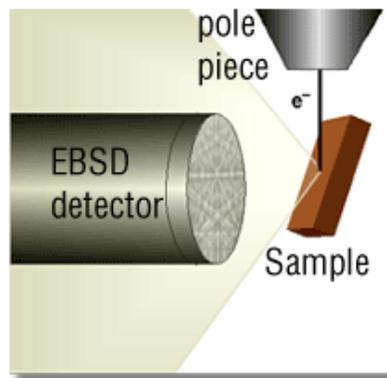
silicon rubber will be used. The basis for physical matches is the assumption that there are an undefined number of matches all along the fracture break. The factors used in physical matches include 1) dimensional consistency, 2) color, 3) shape of the line of the break i.e., zigzag, curved, straight, etc.; 4) irregularities from point to point along the line of the break; 5) any toolmarks or imperfections crossing the broken edge, and 6) fracture surface pattern matching. Measurements of the crystal structure for the physically matched pieces will scientifically complement the physical-match technique. The crystal orientation measurements and matching will provide a way to validate the current physical match process.

The study will use Electron Back-Scattered Diffraction (EBSD). EBSD is a point to point

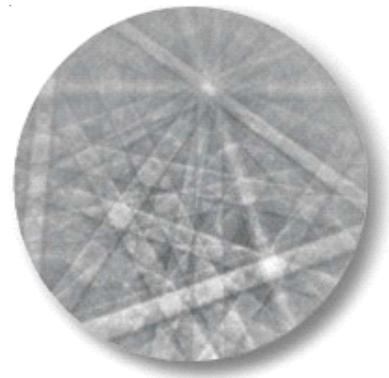
(grain to grain) measurement on the surface of the metal. EBSD basic operation (Figure 1) collects electron back scattered patterns using a specialized collector on the Scanning Electron Microscope. Orientation patterns are generated by sample interaction with the electron beam (Figure 1). A schematic of the process and of resulting patterns of orientation is given in Figures 1 and 2.

TECHNOLOGY BENEFITS

Electron Back-Scattered Diffraction is used to determine the orientation of the individual crystals. One of the objectives of this study will be to determine the feasibility of obtaining suitable patterns from “real” fracture surfaces. The outcome of this project will be a workable measure of the orientation of crystals along the fracture line of many materials such as



Specialized collector on SEM



Kikuchi Bands

Figure 1. Backscattered electron patterns are collected from positions on sample surface.[1]

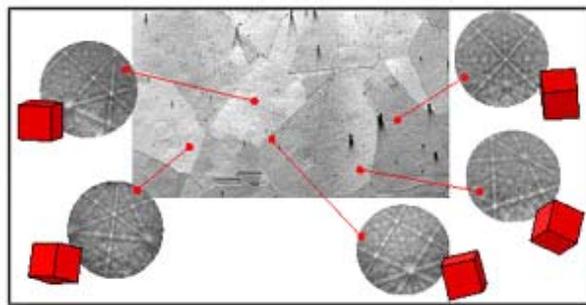


Figure 2. An example of various orientations of adjoining crystals on the metal surface.[1]

[1] Ametek, “EBSD Technologies and Applications Introduction to Orientation Imaging Microscopy”, www.edax.com/technology/EBSD/OIM.html.

metal fractures of knives, pry tools and car antennas.

This study will also serve as the basis for establishing the unique combination of crystal orientation patterns along both sides of the fracture line. These experimental studies will test the core assumptions that if the pattern matches, the surfaces were originally together because of the assumption that a fracture surface is stochastically complex and, therefore, unique. This data collection will strengthen the scientific foundation of the fracture match practice with data to begin the task of estimating error rates.

COLLABORATION/IMPLEMENTATION

M. James Kreiser will perform the preliminary match of the test materials prior to the EBSD measurements. The principal investigators, Barbara Lograsso and Thomas Lograsso of Ames Laboratory, will generate the EBSD experimental data. The PIs will disseminate their findings in presentations at the MFRC Annual Meeting, the Midwestern Association of Forensic Scientists and the American Academy of Forensic Sciences and AFTE annual meetings and regional meetings. The results will be developed into a written paper to be submitted to forensic journals such as Journal of Forensic Sciences. The results will be disseminated in report form on the MFRC web site.

ACCOMPLISHMENTS AND ONGOING WORK

The first quarter of the project (February 1, 2006 to April 30, 2006) has been dedicated to sample preparation and EBSD pattern generation. Samples of typical knife materials such as 440 Stainless steel, tool steel 01, and a carbon steel 1045 have been prepared for pattern examination.

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PROPOSED CD-ROM BASED DIGITAL INFORMATION DATABASE ON PIPE AND TUBING UTILIZED IN IMPROVISED EXPLOSIVE DEVICES

FORENSIC TECHNOLOGY NEED

The rise in terrorist bombings has made the identification of bomb materials a priority for the forensic scientist. The volume of evidence to be analyzed and the importance of such testing for preventative and investigative purposes have increased the need for tools to aid identification of the specific tubing material used for bombs that are reliable, fast, and cost effective. This project is a collaboration between the Western Law Enforcement Training Center (WFLETC), Missouri State Highway Patrol, and Lake County Crime Laboratory in Painesville, Ohio to develop a CD-ROM database for the forensic identification of bomb pipe and tubing.

TECHNOLOGY DESCRIPTION

The objectives of this project are to document all known types of pipes and tubing, the various pipe and tubing manufactures worldwide, any manufacturing identification marks on the pipes and tubing, any companies that import pipes and tubing into the United States, and companies that regularly stock or market each type of pipe and tubing. The first part of the project is documenting as many manufactures of pipes and tubing as can be found. This can be done primarily by internet searches. The next step is to contact them to request information regarding any identification markings. Samples of as many types as possible of pipe and tubing will be purchased. This includes purchasing samples in various regions of the United States. The samples will be measured, digitally photographed and all data entered into the database. All companies that import pipes and tubing into the United States will be identified and documented. Additionally, all companies that commercially sell pipes and

tubing will be documented, as well as any identifying sales tags. All of this information will be placed into a searchable database. Once the samples have been documented, they will be disposed of in an appropriate manner. The database will be forwarded to the Midwest Forensics Resource Center (MFRC) for final compilation into a searchable CD format. As the CD's are completed, the MFRC will be responsible for their distribution.

Currently this information is either outdated or incomplete. Additionally, the data that has been gathered is either in printed format or in handwritten notes. No digital captures of this information is currently available. By putting this type of information in a searchable database, it will cut down on time spent looking for information of this type by investigators and forensic laboratories at all levels of law enforcement.

TECHNOLOGY BENEFITS

This type of database will be of great use to law enforcement agencies nationwide. From 1992 to 2002 the following Midwestern states reported the following number of bombing incidents involving Improvised Explosive Devices (IED's) that can be classified as pipe bombs: Iowa 98 pipe bombs, Missouri 241 pipe bombs, Wisconsin 67 pipe bombs, Minnesota 57 pipe bombs, Nebraska 27 pipe bombs, Kansas 73 pipe bombs, Oklahoma 78 pipe bombs, Arkansas 40 pipe bombs, Tennessee 111 pipe bombs, Kentucky 56 pipe bombs, Indiana 106 pipe bombs, Michigan 201 pipe bombs and Ohio 219 pipe bombs. These amounted to a total of 1,374 pipe bombs in the Midwest Region (26% of nation total). During this same period, there were a total of 5,307 pipe bombs nationwide. The availability of such a database should greatly reduce the amount of time and effort that is

required by forensic scientists to process bomb samples.

COLLABORATION/IMPLEMENTATION

The initial phases of this program will be conducted by internet searches and direct contact with manufacturers, distributors and retailers of pipe and tubing that could be used to construct pipe bombs. These contacts will be made by personnel from the WFLETC, Lake County Regional Crime Lab and the Missouri State Highway Patrol laboratory that are involved in the project. The data obtained from these contacts will be forwarded to the WFLETC. All samples of pipe and tubing purchased for the project will be shipped to the WFLETC for documentation. The equipment needed to digitally document the pipe/tubing will be housed at the WFLETC.

The final CD-ROM product is intended for distribution nationwide to all levels of law enforcement. This would include both the investigative and forensic laboratory levels. The MFRC will be the disseminating agency for this product. Credit for the final CD-ROM will be shared by the MFRC, the WFLETC, the Lake County Regional Crime Lab and the Missouri State Highway Patrol laboratory.

ACCOMPLISHMENTS AND ONGOINGWORK

The PI's have started to procure pipe and end caps. The PI's from the WFLETC, the Lake County Regional Crime Lab, and the Missouri State Highway Patrol laboratory are investigating pipe manufacturing. The video documentation of the purchased pipe lots has started. David Green from Lake County Crime Laboratory plans to tour a pipe manufacturing facility in Ohio.

Future work will focus on research on different manufacturers of pipe materials. Once this research is completed, samples from several areas will be evaluated.

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Quantification of the Individual Characteristics of the Human Dentition

FORENSIC TECHNOLOGY NEED

Daubert vs. Merrell Dow Pharmaceuticals has raised the bar for admissibility of comparative science testimony. Although it is generally accepted in the dental community that the human dentition is unique, there has been little research generating statistical evidence to support this supposition.

Partnering with the Wisconsin Department of Justice, Crime Laboratory – Milwaukee Imaging Specialists and a Professor of Evidence from Marquette University's Law School, this pilot study will begin the development of a data base on the frequency distribution of six dental characteristics in the population. It will also generate statistical evidence for an inter-operator and intra-operator error rate for those analysts conducting the research, answering another of the Daubert factors.

A literature search on the individuality of the human dentition reveals only a few studies on individuality as it relates to patterned injuries (bitemarks). MacFarlane (1974) authored a study which is frequently cited, followed by Rawson's (1984) research and a project limited to arch width by Barsley (1987).

Currently forensic odontologists, in the analysis of bitemark evidence, are not able to quantitatively state the frequency that a given set of dental characteristics occurs in the population. Without the ability to quantify the frequency, their conclusion lacks a legal scientific basis and is limited to a subjective opinion. In this project, Dr. Thomas Johnson at the Marquette University School of Dentistry in collaboration with the Wisconsin State Crime Laboratory will use an appropriate technique for examining several remarkable characteristics of the teeth.

TECHNOLOGY DESCRIPTION

Utilizing professional imaging and statistical software this protocol will vastly improve the accuracy of observing, recording and calculating the measurements and frequency of the attributes being studied in this random, ethnically diverse sample population. Further expansion of the study would include the use of a hand held laser surface scanner, only recently available, enabling the direct computer capture of three dimensional dental characteristics. This will eliminate the need for registering an exemplar which then has to be scanned into the analysis software.

This project has two specific aims:

- Investigate using six measurements, the pattern of each of 400 samples, and calculating the probability that any two would be judged to be the same.
- Begin the development of a database that can be used to define the frequency that a given set of dental characteristics occurs in the population.

This study will utilize tooth exemplars, recorded in an ADA-accepted dental bite registration material, of 400 male volunteers between the ages of 18 and 44. This sample size (n=400) was derived from power calculations by a biostatistician using nQuery Advisor®. Final calculations will be accomplished using SAS® Statistical Analysis Software. The volunteer samples will be randomly derived from dental clinic patients, representing a diverse population composed of Caucasians, Blacks, Asians, and Hispanics that mirrors the general population. To limit the size of the study, only males will be sampled. A total of 500 exemplars will be collected

throughout the life of this research to allow for as many as 100 drop-outs (exemplars that don't meet the standards required).

All information will be recorded using an alpha numeric designation to protect identity and preserve confidentiality. Imprints of the biting edges of the dentition will be recorded in an American Dental Association (ADA)-approved bite registration material, scanned at 100% at a resolution of 400 pixels/ inch and saved as read-only images in a postscript file format. The scans will be accomplished using an Epson Expressions model 1680 Pro. The accuracy and reliability of the scanner will be verified before and periodically during the scanning. Original scans will be archived on an external hard drive and backed-up on compact disks. Adherence to the Scientific Working Group on Imaging Technology guidelines will be verified by two imaging specialists from the Wisconsin State Crime Laboratory - Milwaukee.

TECHNOLOGY BENEFITS

- The rapid development of technology has enabled the direct capture of data, eliminating several intervening steps during which error could be introduced. Earlier studies were highly labor

intensive, requiring several steps between capturing the dental characteristics and the analysis.

- Until the comparative science of bitemark analysis is able to demonstrate hard science for opinion and establish an error rate, the admissibility of this valuable tool is in jeopardy.
- The development of this technology will enable odontologists not only to express an opinion of probability based upon statistics, but also apply this analytic technique to the examination of actual patterned injuries, greatly reducing the element of subjectivity.

The ability to scientifically express the linkage between a human bitemark and the perpetrator will establish the credibility of forensic odontology. Creating a database for this comparative science reduces the reliance on subjective assessments. It will also discourage the attempted analysis of indistinct bitemark patterns, since the characteristics necessary to apply the database must be observable. Presently observations are expressed only as the objective assessment of specific characteristics observed and a

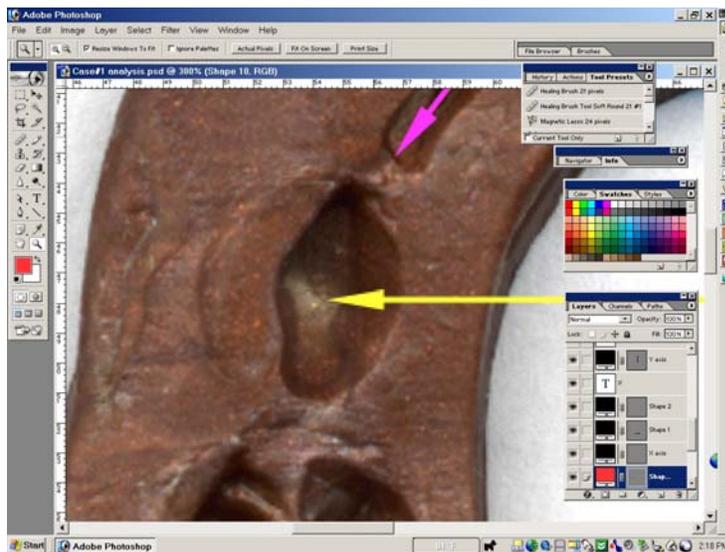


Figure 1. The tools in Adobe Photoshop CS2 are utilized to measure and record size and angles of rotation from the X axis.

subjective evaluation of their evidentiary value. This project will investigate if a logical basis of science can be determined, thus establishing credibility for the bitemark analysis. The long-term goal for this initial project is to continue to expand the sample size, involving other sources of exemplars, and thus expand the database.

COLLABORATION/IMPLEMENTATION

- This is a multi-disciplinary team composed of a Professor of Evidence from the Law School, a biostatistician and two imaging specialists from the Wisconsin State Crime Laboratory. The Law School Professor will provide the expertise on the admissibility and case law pertaining to this type of evidence. The Imaging specialists will assist in following the SWGIT guidelines and the biostatistician will calculate the statistical data and provide expertise in preparing the charts and graphs for dissemination and publication.

- A paper entitled *Quantification of the Individual Characteristics of the Human Dentition; a Preliminary Report*, was presented at the American Academy of Forensic Sciences annual session in Seattle, Washington, February 24, 2006.
- Partnering with the Wisconsin State Crime Laboratory – Milwaukee, the study will utilize two imaging specialists as consultants on computer generated digital imaging techniques in conformity with the FBI Scientific Working Group on Imaging Technology (SWGIT) standards.
- A paper on the completed research will be submitted for presentation at the annual meeting of the American Academy of Forensic Sciences, (AAFS), and will be submitted for publication in the *Journal of Forensic Sciences*. Publication in this refereed journal of the AAFS will provide broad dissemination, reaching a membership of 6000, representing the entire United

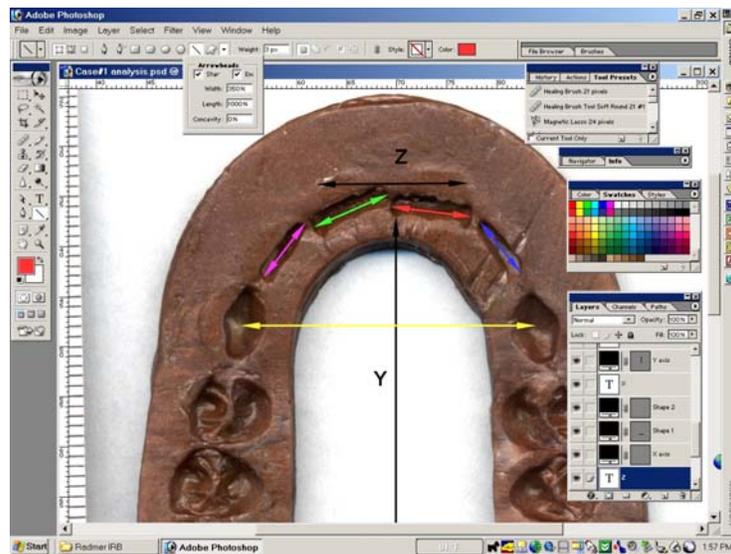


Figure 2. By establishing the start and end point of each area to be measured, with the line tool with arrow tips, the measure tool can be more accurately placed.

States, Canada and fifty-six other countries worldwide. The completed research paper will also be submitted for publication to the *Journal of Forensic Identification*. Additional presentations are anticipated by the dental student research assistant at the International and American Association of Dental Research (IADR/AADR) meeting.

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ACCOMPLISHMENTS AND ONGOING WORK

The first quarter of the project began by March 1, 2006. One hundred exemplars have been registered and scanned into the master file of read only images.. A file hierarchy has been set up, with separate working files of duplicate images for each of the six student researchers. An Access data entry sheet has been developed by the biostatistician for the direct transfer of recorded measurements into the Excel spreadsheet. A protocol has been established to accuracy and repeated reliability of the scanner. Imaging specialists from the State Crime Laboratory – Milwaukee have been available to insure that the protocol conforms to the guidelines of the Scientific Working Group on Imaging Technology (SWGIT).

By the end of the grant year, January 31, 2007, we anticipate having data on two hundred of the sample, (“n”=400), accomplished and be able to report a statistical trend. It is our expectation that NIJ will by then have acted upon a grant proposal submitted on January 13, 2006 which will then allow completion and an extension of this research.

STATISTICAL VALIDATION STUDY OF TOOLMARK UNIQUENESS

FORENSIC TECHNOLOGY NEED

Recovery and comparison of toolmarks, footprint impressions, and fractured surfaces connected to a crime scene are of great importance in forensic science. It is generally accepted that particular manufacturing methods produce marks on tools that are substantially different from tool to tool. This premise is based on a handful of limited studies in the literature, training, and on years of experience of the toolmark examiner in making relative correspondences and associations of known matches and non-matches to develop a knowledge of uniqueness.

The forensic community is seeking studies to provide validation of the proposition of tool uniqueness because of recent court challenges. There have been several studies showing the impact of various tool manufacturing methods on the individuality of toolmarks and striations produced on tools. These studies have also shown that similar and/or sequentially-produced tools make toolmarks that are distinctly distinguishable. The major shortcomings of these studies is that they concentrated on only a single manufacturing method (e.g., broaching) and typically used a small number of samples (<10).

This project attempts to provide the statistical tools necessary for the validation of the proposition that particular manufacturing processes produce marks on the work-product (or tool) that are substantially different from tool to tool. This work extends these previous studies by conducting a more comprehensive statistical analysis of toolmark variation produced by several manufacturing methods (filing, grinding, whetstoning, broaching, and milling).

TECHNOLOGY DESCRIPTION

The overall objective of this project is to provide the basis for and conduct a numerical validation study of the proposition that particular manufacturing processes produce marks on tools that are substantially different from tool to tool. The approach to validation involves the development of statistical methods for data reduction and the analysis of a collection of digital images of toolmarks produced by various tool manufacturing methods on produced work-products. The study is being performed on an existing database of over 8,000 digital images representing grinding, milling, broaching, whetstoning, and filing manufacturing processes on commercial tools as well as consecutively-made samples. In order to achieve this objective, the project extends and augments work that was initiated under a project funded by an FBI grant to develop statistical tools for the validation of toolmark uniqueness. That work (see Publications and Presentations), conducted by the PI's of this project, provided the proof of concept demonstrating that a "degree of association" or degree of similarity, relative to a particular manufacturing process, can be obtained from digital images of toolmarks and developed statistical algorithms that were designed to mimic the comparison process of a forensic scientist.

The initial work from the FBI grant laid the groundwork for this more detailed and comprehensive study on validation. Since the initial feasibility study was based on preliminary algorithms, tested on a limited number of digital images, and optimized for ground surfaces, several refinements and issues of the original work need to be addressed in order to achieve the objectives of this project. Briefly, these refinements are: i) accelerating image comparisons either by

exporting the algorithms to a compiled language or integrating the algorithms with commercial pattern-matching software; ii) optimizing algorithms for surfaces from other manufacturing processes (e.g., broach, milled, and filed), since the initial algorithms were optimized using images of ground surfaces; and iii) applying faster and more advanced algorithms to an extensive image database for more refined estimation of error rates for various surfaces.

TECHNOLOGY BENEFITS

Implementation of this work will provide formal and objective support to the premise of toolmark uniqueness, which is empirically based on a handful of limited studies in the literature, training, and years of proficiency testing and experience of toolmark examiners in making comparisons of known matches and non-matches to develop a knowledge of uniqueness. An objective validation of tool uniqueness, consistent with sound scientific methods, supporting an examiner's assigned identification based on alignment of striae of a questioned mark made with a suspect tool, is particularly important in light of some recent court challenges.

COLLABORATION / IMPLEMENTATION

This work is being carried out in collaboration with Mr. James Kreiser, an experienced forensic toolmark examiner. The results of this work will be presented at forensic science meetings and published in relevant forensic science journals. Communication of preliminary results from the previous study generated significant interest in the community.

PUBLICATIONS AND PRESENTATIONS

- Baldwin, D. P., Morris, M., Bajic, S. J., Zhou, A., "Statistical Tools for Forensic Analysis of Toolmarks," Poster Presentation at American Academy of Forensic Sciences, 56th Annual Meeting, 2004, Dallas, TX, Paper B95.
- Baldwin, D. P., Morris, M., Bajic, S. J., Zhou, A., Kreiser, M. J., "Statistical Tools for Forensic Analysis of Toolmarks- Final Report," FBI/Ames Laboratory Report IS-5160 (2004).

ACCOMPLISHMENTS AND ONGOING WORK

This project, under a previous FBI grant, demonstrated that a validation study can be performed using digital images and statistical algorithms to produce a well-defined metric (i.e., index value) to determine the level of differences between known matches and known non-matches for a particular tool manufacturing method. Refinement and reimplementing of the original algorithms into a compiled language has been completed. A more extensive validation experiment, testing the refinements and investigating the assignment of "critical" index value for a particular surface type, has begun. Investigation of employing and integrating a commercial pattern-matching software package for comparisons continues. Additionally, strategies to objectively identify 'areas of interest' in images for comparison have begun to be identified and examined.

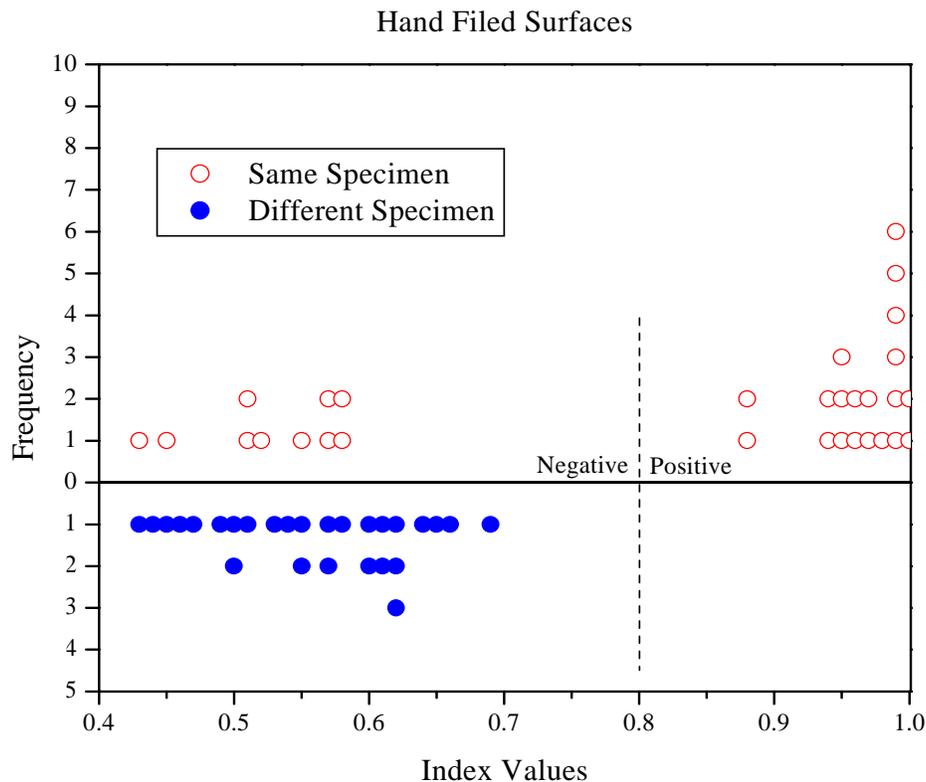
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Frequency chart showing results from applying the experimental algorithm to a 30-image data set of hand-filed specimens acquired at 15x magnification. The dotted line may be indicative of a characteristic “break-point” for this surface type. Same specimen index values to the left of the dotted line are false-negative results. Note that there are no false positives. The assigned index value indicates the “quality” of a match or non-match and involves the comparison of the degree of match for points at predetermined distances and angles to randomly selected points around the image. Sets of three replicate images were used for each specimen from a collection of ten samples to test the experimental algorithm in a controlled study. Images of the same object were classified as “known matches”, while images of similar objects of the same surface type were labeled as “known non-matches.” The resulting 30-images, compared two at a time, provide 30 image pairs that should match (those taken from the same replicate set) and a very large number of possible pairs that should not match. The generated index values for same-object pairs are larger than those for different-object pairs; the degree to which these groups separate (good) or overlap (bad) is an indication of how well the algorithm performs.

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