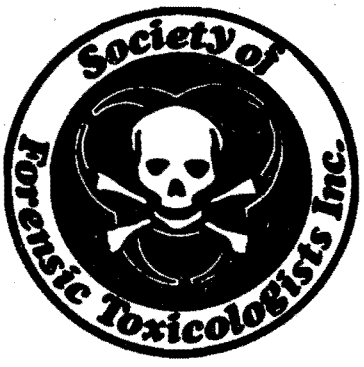

SOFT/CAT MEETING

SOCIETY OF FORENSIC TOXICOLOGISTS
CALIFORNIA ASSOCIATION OF TOXICOLOGISTS

MGM GRAND HOTEL
RENO / LAKE TAHOE NEVADA
OCTOBER 29 – NOVEMBER 1, 1986

PARTICIPANTS FROM THROUGHOUT THE
UNITED STATES AND CANADA

A REVIEW OF TANDEM MASS SPECTROMETRY (MS/MS) IN TOXICOLOGY, Dean D. Fetterolf, Forensic Science Research Unit, FBI Academy, Quantico, Va 22135

The routine identification of drugs and xenobiotic substances in physiological samples by toxicology laboratories typically involves a two step screening and confirmation procedure. MS/MS combines these two stages of analysis in one instrument.

This presentation will review quadrupole MS/MS instrumentation and its application to clinical, forensic, and environmental toxicology.

The forensic determination of drugs of abuse at the nanogram level will be presented. For example, cocaine and its metabolites can be simultaneously detected in urine at the 10 ng/ml level in only one minute.

To determine environmental exposure to toxic chemicals rapid, sensitive, and selective methods are required. An MS/MS procedure to determine chlorinated hydrocarbons in human urine and blood at the sub-ppb level at the rate of 100 samples/hr has been developed.

Clinical applications such as the determination of femtogram levels of tryptomines and anticonvulsants in brain tissue extracts will be discussed.

A NOVEL METHOD FOR THE CALIBRATION OF KOVATS RETENTION INDICES USING n-ALKANES WITH A THERMIONIC NITROGEN-PHOSPHORUS SPECIFIC DETECTOR

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This paper describes a novel method for the calibration of gas chromatographic (GC) columns with nitrogen-phosphorus detectors (NPD) using n-alkanes rather than nitrogen containing drug mixtures. This results in a more accurate and rapid calibration for the calculation of relative retention times (RRI), such as Kovats indices, than has been possible using NPD. The method describes the temporary conversion of the NPD into a detector with properties much like a flame-ionization detector (FID).

By deliberately increasing the H_2 gas flow rate to the NPD bead from 4 ml/min to 8 ml/min, the n-alkanes can now be detected by the NPD and, therefore, used as RRI calibrators. Once the GC has been calibrated, the H_2 gas flow is lowered back to the normal flow of 4 ml/min. The detector now behaves as a NPD, no longer detecting n-alkanes.

LEGAL ISSUES OF PREPLACEMENT DRUG SCREENING TOXICITY AND LAW

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Preplacement and employment related drug screening of toxicology likely to be the subject of legislation and regulation in the near future. Government policy at the Federal level is encouraging preplacement and employment drug screening of employees in safety or health sensitive areas. Illicit drug control measures are expanding from interdiction of supplies and suppliers to surveillance of the user population.

This talk will address toxicological issues in employment related analyses, particularly assurances of sample integrity, reasonable chain of custody, licensure or certfication of laboratories and personnel, methodologies of screening and confirmation, the legal evidentiary value of scientific opinion testimony, retention of positives under lock and key, legal mechanisms for discovery of reports and results, and subpoena of specimens. Medical confidentiality and clinical laboratory regulations in California require particular sensitivity towards confidentiality of results, release of reports or specimens, and efforts to prevent publication (dissemenation to others) which may be defamatory or slanderous.

EXCRETION OF BENZOYLECGONINE FOLLOWING INGESTION OF COCA TEA

W. L. Hearn, T. Hall and L. Totton, Toxicology Testing Service, Inc., Miami, Fla.

Health Inca Tea, a supposedly decocainized coca leaf tea, has been sold in health food stores throughout the United States for several years. When the product was reported to contain cocaine, some stores stopped selling it, but it remains available in some locations.

The effect of drinking Health Inca Tea on the urinary excretion of benzoylecgonine (BE) was investigated in three volunteers who drank one, two and three cups of the tea respectively. Urine samples collected before and for several days following consumption of the tea were screened by EMIT-DAU and EMIT-Q-ST assays for BE, and selected samples were confirmed and quantified for BE by GC-MS using an ethylated derivative and trideuterated BE internal standard. The three doses yielded maximum BE concentrations of 5800, 12,000, and 14,800ng/ml respectively. Times to first negative urine were estimated from graphs of EMIT $\triangle$ values vs. time. These data show that a single cup of coca tea can yield positive ($>$300ng/ml) test results for 28 hours, while consumption of three cups yielded EMIT positive urines for over 55 hours.

EVALUATION OF EMIT AND RIA HIGH VOLUME TEST PROCEDURES FOR THC METABOLITES IN URINE UTILIZING GC/MS CONFIRMATION

M. L. Abercrombie, M.S. and J.S. Jewell, Ph.D., Forensic Toxicology Drug Testing Lab (WRAMC), Ft Meade, MD 20755

Results of EMIT, Abuscreen RIA and GC/MS tests for THC metabolites in a high volume random urinalysis program are compared. Samples were field tested by non-laboratory personnel with an EMIT system using a 100 ng/mL cutoff. Samples were then sent to the Army Forensic Toxicology Drug Testing Laboratory (WRAMC) at Ft Meade, MD, where they were tested by RIA (Abuscreen) using a statistical 100 ng/mL cutoff. Confirmations of all RIA positives were accomplished using a GC/MS procedure. EMIT and RIA results agreed for 91% of samples. Data indicated a 4% false positive rate and a 10% false negative rate for EMIT field testing. In a related study, results for samples which tested positive by RIA for THC metabolites using a statistical 100 ng/mL cutoff were compared with results by GC/MS utilizing a 20 ng/mL cutoff for the THCA metabolite. Presence of THCA metabolite was detected in 99.7% of RIA positive samples. No relationship between quantitations determined by the two tests was found.

RAPID SCREENING OF BIOLOGICAL SPECIMENS FOR DRUGS USING TOXI-LAB® EXTRACTIONS AND CAPILLARY GC/MS.

Ricky P. Bateh, PhD (Consolidated Laboratory Services, 2549 Park Street, Jacksonville, FL 32204)

As a result of drug screens gaining popularity in the public domain, many clinical and forensic laboratories performing these tests have had increased workloads. To compensate for these increased workloads, new methods have been evaluated to increase throughput and to decrease turnaround time for screens and confirmations.

Reported here is a method in which drugs are extracted from biological materials using commercially available Toxi-Tubes® and analyzed by TLC followed by GC/MS. The protocol will be discussed. GC/MS analysis is performed on a Hewlett-Packard 5970B system. A variety of drugs and metabolites (~75 compounds) are eluted in a 15-minute program and are readily identified by their mass spectra.

The combination of these two methodologies provides a rapid and efficient means by which specimens are screened/confirmed for the presence of drugs.

"URINE DRUG TESTING — ITS DESIGN, IMPACT AND LIMITATIONS"

NARESH C. JAIN
Rancho Los Amigos Medical Center, Downey, CA., 90242 and USC School of Medicine, Los Angeles, CA 90033

Drug abuse has become one of the most compelling realities of contemporary society It has penetrated every segment of our population: from schools to sports and from organized crime to board rooms. Drugs in the workplace allegedly cost government agencies and business millions of dollars each year in increased absenteeism, poor work performance, thefts, accidents and wasted time. The President's Commission on Organized Crime is in favor of urine drug testing. In fact, many employers are now resorting to urine drug testing on current and prospective employees.

This paper discusses main aspects of urine drug testing, including design and security requirements of the toxicology lab. Random drug testing requires that urine samples be collected under direct observation and chain of custody maintained at all times. Records of quality control, proficiency testing, training and certification of lab personnel and calibration/maintenance of scientific equipment must be made available in an arbitration or legal proceeding. Quantum of proof and interpretation of both positive and negative urine drug tests will be discussed.

PHENOTHIAZINE TRANQUILIZERS AND ENVIRONMENTAL AIR POLLUTANTS

Hendrik Keyzer and Wayne Plumtree
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Los Angeles, CA 90032

Phenothiazine drugs administered as adducts of electron acceptors induce significant changes in duration of sedation and hypothermia compared to the drugs in the pure form. Sulfur dioxide, nitrogen dioxide and ozone, common air pollutants, altered these physiological effects of the drugs considerably whether administered as part of the adduct or as sensitizers of test animals. Age, condition and sex of the test animals, in this case Webster Swiss albino mice, had a vital bearing on the results of administration of the adducts and of the pure drugs to sensitized test subjects. The lethal dosage of the drugs was also affected by the pollutants. The observations are discussed in terms of various hypotheses.

PLACENTAL TRANSFER OF IMIPRAMINE TO A NEWBORN INFANT, Robinson, C.A., Upton, K., and Scott, J.W., Dept. of Pathology, The University of Alabama at Birmingham, AL 35294.

A case of imipramine toxicity in a newborn infant is described demonstrating the ability of imipramine to cross the placenta following overdose of the mother.

A 20 year old woman 30 weeks pregnant was admitted to the hospital with premature uterine contractions. The patient had ingested 900 mg of imipramine-HCl over an 8 hour period prior to admission. The patient's admission laboratory data were within normal limits; ritodrine and magnesium sulfate were administered to inhibit uterine contractions. Amniocentesis was performed; the amniotic fluid showed an L:S ratio of 1.7. Despite efforts to inhibit uterine contractions a 1000 gm female was delivered, breech, 72 hours post admission. The infant developed hyaline membrane disease and expired at 2 weeks of age.

The total TCA level was 359 ng/ml mother and 134 ng/ml infant. Imipramine has generally been accepted as safe during pregnancy and to our knowledge has not been reported to cross the placenta; nor is the effect of imipramine on uterine contractions known.

THE QUANTITATIVE DETERMINATION OF SERUM CAFFEINE LEVELS IN NEONATES RECEIVING THEOPHYLLINE FOR PRIMARY APNEA, Basso MJ, Ristuccia P, Carson S, Bidanset JH, Department of Pharmaceutical Sciences, St. John's University, Jamaica, NY, 11439

Primary apnea, suggested as a cause of central nervous system damage, occurs frequently during the neonatal period. Traditional management of apnea has involved the administration of low doses of theophylline. The neonate uniquely metabolises theophylline to caffeine.

This study compared two methods for quantitating caffeine, high performance liquid chromatography (HPLC) and enzyme multiplied immunoassay technique (EMIT), in neonatal serum from infants receiving theophylline. Sixty infants with primary apnea, admitted to the Intensive Care Nursery of Winthrop-University Hospital, each received an initial dose of 3mg/kg aminophylline. Blood samples were obtained by heelstick. Serum was separated and used for the determination of theophylline (fluorescence polarization immunoassay-FPIA). Remaining serum specimens were frozen and stored at -80 C. until analyzed for caffeine.

A STUDY ON THE INTERACTION OF ETHANOL DIAZEPAM AND PROPOXYPHENE, James Ruger, Ph.D., Jesse H. Bidanset, Department of Pharmaceutical Sciences St. John's University, Jamaica, New York 11439

Ethanol, diazepam and propoxyphene, each at four dosages (nine combinations) were orally administered to Sprague-Dawley male rats. Three performance tests were correlated with the dosages and brain concentrations of the drugs. The propoxyphene-ethanol interaction was found to be antagonistic. The interaction of ethanol-diazepam was synergistic for all three tests, at most doses and times. The propoxyphene-diazepam combinations produced significantly greater effects than from either agent alone.

One hour post-dose, rats were sacrificed by decapitation, the cerebellum was removed within one minute and flash frozen in liquid nitrogen. Cyclic-GMP was measured by RIA. Ethanol, diazepam and propoxyphene concentrations were assayed by gas chromatography. Generally, c-GMP activity was depressed in a dose-related manner, but correlated poorly with the performance tests in all but the ethanol-diazepam interaction.

DETECTION OF BARBITURATE IN URINE BY LATEX IMMUNOASSAY.
Joseph Wu, Lucia Franco, Salvatore Salamone, and Magdalena Usategui.
Roche Diagnostic Systems, Nutley, NJ 07110.

A homogeneous latex immunoassay has been developed for qualitative and quantitative detection of barbiturate in urine. The assay is based on agglutination between barbiturate antibody and a barbiturate-latex conjugate. When free barbiturate reacts with antibody, no visible agglutination is produced. The presence of free barbiturate in urine and barbiturate-latex conjugate in the reaction mixture results in competition for antibody binding, so that agglutination is decreased with increasing concentration of free barbiturate. The assay procedure involved the transfer of a mixture of conjugate, urine sample and antibody to a slide. The reaction was then measured from an analog-digital device. The assay is completed in 3 minutes. Coefficient of variation for within and between day studies are well below 10%. Results obtained from 60 patient samples with the present method as compared to those with radioimmunoassay were good. This method is simple, fast, and needs no sample dilution or pretreatment.

CONFIRMATION OF POSITIVE EMIT-D.A.U. (TM) ASSAYS BY THIN LAYER CHROMATOGRAPHY. Gerald Clement, Deneen Pieri, Joann Havassy and Susan Liska, Health-East Laboratories, P.O. Box LAB, Allentown, PA, 18105.

We analyzed 800 urine specimens for drugs of abuse using a battery of EMIT assays and a modification of the Davidow thin layer chromatographic procedure. The confirmation ratios were (no. confirmed/no. positive by EMIT): Amphetamines (21/82), Barbiturates (19/31), Benzodiazepines (11/62), Cocaine metabolite (79/124), Methadone (18/22), Opiates (33/57), Phencyclidine (1/1) and Propoxyphene (7/11). When possible, unconfirmed positive specimens were reanalyzed by a third method. The low ratio of confirmation for amphetamines was due to cross-reacting substances in the EMIT assay. Many unconfirmed positives for benzodiazepines and opiates tested positive after hydrolysis of the specimens. Most unconfirmed positives for cocaine metabolite were beneath the detection limit of the TLC method. A summary of the analyses of unconfirmed positives, a discussion of detection limits and the preliminary results of our in-house blind proficiency testing program will be presented.

GUIDELINES AND SPECIFICATIONS FOR QUALITY ASSURANCE PROGRAMS. Raymond J. Bath Ph.D., Bath Toxicology Group. Inc., 17 Stone Lane, Marlboro, New Jersey 07746

Quality Assurance Programs and Quality Control Procedures are the prerequisites of laboratories generating data for forensic science investigations. Courts and governmental agencies are now requiring active quality assurance programs that provide the policies, organization, objectives, functional activities and specific QA and QC procedures designed to achieve data quality goals. This presentation provides the guidelines and specifications of the sixteen essential elements of a QA Project Plan, recommends a format to be followed, and specifies how plans are to be reviewed and approved.

SITE DEPENDENCE OF DRUG LEVELS IN POSTMORTEM BLOOD - AN EXTENSIVE CASE STUDY
G.R. Jones and D.J. Pounder
Office of the Chief Medical Examiner, Edmonton, Alberta, Canada.

There is an increasing awareness that blood levels of some drugs increase rapidly and extensively after death. Postmortem increases of 2-5 fold have already been reported for some drugs. Furthermore, postmortem concentrations of many drugs are markedly site dependent.

We wish to present extensive distribution data gathered on a 25 year old female who overdosed on imipramine, acetaminophen, codeine, diphenhydramine and ethanol. These substances, plus desipramine, were quantitated in blood from 10 different sites, tissue from 25 sites involving the major organs and three muscles, plus CSF, vitreous humor and bile.

Blood imipramine levels differed by up to 760% (2.1-16.0 mg/l) whereas blood concentrations of acetaminophen differed by less than 20% (55-65 mg/l). An intermediate distribution pattern was seen for codeine (range 0.33-0.89 mg/l) and diphenhyramine (range 0.34-2.04 mg/l). The blood ethanol levels ranged from 1.51-1.75 g/l (mean 1.64).

The data obtained demonstrates the wide intra-subject variability of blood levels possible for some drugs, and allows preliminary conclusions to be drawn regarding a mechanism for the phenomenon.

SIMULTANEOUS DRUG SCREENING AND CONFIRMATION USING WIDE BORE CAPILLARY GAS CHROMATOGRAPHY

R.E. Shirey, R.J. Bartram, W.J. Pinnick, and J.E. Doyle*
Supelco, Inc., Supelco Park, Bellefonte, PA 16823-0048 (814)359-3441

By using a tee and two 30m x 0.75mm glass columns of distinctly different polarity (SPB-1 and SPB-35 columns), an analyst can confirm the identification of drugs in a sample rapidly and with a minimum of expense. The newly designed tee provides a mixing chamber that ensures complete volatilization of the sample. This eliminates sample discrimination and uniformly divides the sample between the two columns. The SPB-1 and SPB-35 columns are bonded, samples can be analyzed on both columns under identical run conditions. The analysis time on the higher polarity SPB-35 column is not significantly longer than on the SPB-1 column.

COCAINE METABOLITE ASSAY IN DRUG ABUSE PATIENTS USING ABBOTT TDx.

B.M. Kapur, D. Meadows and M. Anderson
Addiction Research Foundation,
33 Russell Street, Toronto, Canada,
M5S 2S1

During a 6 week period calibration curve stability and precision studies were performed on the Abbott TDx. A comparison was done on 100 methadone maintenance and suspected drug abuse patient urine samples, screened by TLC (26 positive, 24 questionable and 50 negative (randomly selected)) for cocaine were tested with the Abbott's TDx and Syva QST cocaine metabolite assay. A few positive samples were also serially diluted and compared. All immunoassay on patient samples were done in duplicate.

Results: Calibration curve deviation ranged between 1-3% for the various calibration points over the fourteen day period. CV for the low control was 4% whereas for the high control was 2%. All TLC positive and questionable samples gave positive result with both the immunoassay procedures. All TLC negative samples were also confirmed with these procedures. Serial dilutions of patients urines demonstrated linearity within the TDx calibration range. This was not possible with Syva's QST procedure.

EVALUATION OF TDx DAU ASSAY FOR PCP

M. Calderone, D. Somers, R. Stephon, R. Foery, Toxicology Department, Reference Laboratory, Newbury Park, CA 91320

The TDx (Abbott Laboratories) DAU Phencyclidine Assay is a reagent system for the detection of 1-phenylcyclohexylpiperidine (PCP) in urine. Using 50 uL of human urine, at pH 5.0 to 8.0, the assay exhibited a dynamic range of 25 to 500 ng/ml. Within run and day-to-day precision were 2.3% and 4.7% at 25 ng/ml, 2.8% and 6.0% at 35 ng/ml, and 2.2% and 2.9% at 250 ng/ml.

Recovery studies in the assay's dynamic range were 97.7 to 101.5%. The calibration curve was stable for 10-14 days at ± 10% of target concentrations. Comparison of TDx vs EMIT and TDx vs GLC data showed 96 of 101 specimens were positive by all three methods at conc. >75 ng/ml, 5 of 101 specimens were positive by TDx and GLC at conc. <75 ng/ml, and 97 PCP-free specimens were negative by TDx, EMIT, and GLC.

The assay provides acceptable accuracy and precision with minimum cross-reactivity to potentially interferring substances.

DRUGS OF ABUSE: DATA COLLECTION SYSTEMS OF DEA AND RECENT TRENDS

Richard S. Frank, Chief
Forensic Sciences Section
Office of Science and Technology
Drug Enforcement Administration
1405 Eye Street, NW
Washington, D.C. 20537

The U.S. Drug Enforcement Administration has several different systems for collecting drugs of abuse. One is an early warning system, which collects information to provide scientific data and interpretation on drug abuse. Another is the collection of data from evidence submitted to DEA laboratories. This presentation will review the significance of the data in each system, and examine some of the trends observed from the data.

"YOU BET YOUR LIFE!"

Jerry D. Nelson
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National City, CA. 92050

We all know that "strung-out" junkies are playing their own form of "Russian roulette." Some of us recognize that clandestine drug laboratory operations represent a threat to the safety of the drug manufacturer as well as enforcement personnel. But did you know that these operators threaten your safety and security as well? You bet your life!

Through photographic slides we will re-visit clandestine drug laboratories to demonstrate lack of interest in good manufacturing practices, quality control and safety.

RECOGNIZING ECSTASY: ADAM AND EVE, THE MDA DERIVATIVES - ANALYTICAL PROFILES

W.L. Hearn, G. Hime & W. Andollo,
Toxicology Testing Service, Miami, Fla.

In the first half of 1986 the news media discovered the drug MDMA (Ecstacy, Adam, 3,4-methylenedioxy-N-methylamphetamine), and publicized it so efficiently that it is now in great demand. As judged by the number of samples submitted to Up Front Drug Information's S.P. Lab; MDMA, MDEA (Eve, 3,4-methylenedioxy-N-ethylamphetamine) and MDA (3,4-methylenedioxyamphetamine) have become increasingly plentiful "on the street." In anticipation that they will be encountered in forensic specimens, analytical data have been developed to assist in the identification of these three drugs. Thin layer chromatographic characteristics, gas chromatographic retention indices and mass spectral data will be presented. Cross reactivity with the EMIT Amphetamine Assay was also investigated and will be discussed.

FENTANYL ANALOGS-SCREENING TECHNIQUES

Donald P. Cox, Ph.D.
Janssen Life Sciences Products
Piscataway, NJ 08854

Tritium-based Radioimmunoassay procedures for fentanyl, alfentanil and sufentanil were developed for determining body fluids levels (primarily in serum) of the appropriate fentanyl analog and/or metabolite. The procedures are sensitive to the low nanogram range for the parent compounds and have been successfully employed for studying the pharmacokinetics and metabolism of the respective drugs. Certain non-commercialized analogs have been shown to cross-react with fentanyl antisera and sufentanil antisera which provides a potential use for illicit fentanyl analog screening. Currently, these radioimmunoassay kits are being modified and tested for rapid screening of urine samples. As data become available, appropriate modifications in the product information sheet will be made and submitted to regulatory agencies to support approval of an indication for presumptive evidence for abusing a fentanyl analog (in the absence of authorized use).

Confirmatory procedures will be necessary to support the data and recommended techniques are available.

WITNESS EFFECTIVENESS TRAINING

Many articles have been written on the subject of the expert witness. Most of the articles appear in the forensic journals and the contributors include other forensic scientists, lawyers, judges and psychologists. The subject matter covers a broad range of topics from how one should present certain types of evidence at trial - through being a good expert witness and onto discussions about legal versus scientific truth. These articles and the infamous 'mock trial' have been to date the basis for expert witness training. In reality, the only training most experts receive is when they are 'on-the-job', that is, at trial. And usually there is little or no critique provided to the witness after their testimony. Learning how to be an effective witness, that is, communicating ones believability should not be left to 'practice'. The purpose of this paper is to present a unique approach to enhancing expert testimony by training people to be effective witnesses rather than training people how to testify.

Raymond J. Davis
Director
Quantum Analytical Laboratory

AN ETHANOL-RELATED HEMOGLOBIN ADDUCT IN CHRONIC ALCOHOLICS: IDENTIFICATION AND CHARACTERIZATION

Teri Stockham, M.S.*, and
Robert Blanke, Ph.D.,
Department of Pharmacology & Toxicology, Medical College of VA.
Richmond, Virginia 23298

Frequently, chronic exposure to chemicals is difficult to document by analysis. Further, exposure to an agent at some time in the past may be troublesome to prove since the agent may be metabolized or excreted quickly. Others have shown that selected chemicals can form adducts with macromolecules such as DNA or proteins. These persist for the life of the macromolecule. It has been proposed that such an adduct is formed with hemoglobin in alcoholics. This study describes the verification of this observation in alcoholic patients. In addition, the adduct has been isolated and purified by cation exchange and affinity chromatography for the purpose of characterization. If this adduct proves to be a unique marker, it may serve to identify alcoholics or even to indicate alcohol use after conventional blood alcohol concentrations are not detectable.

CAN WE LEGISLATE A DECLINE IN MVA'S?

J. Beno Ph.D., D. Diegert, H.E. Riley, S. Kriewall B.S., P. Schantz B.S.
Monroe County Medical Examiners Office
Rochester, New York

Monroe County, N.Y. has seen numerous laws adopted over the past several years increasing the severity of the penalties for D.W.I. and increasing the drinking age from 18 to 21. Implicit in the adoption of these laws is the belief that they will save lives! In 1985, in the wake of a mandatory seatbelt law and an increase in the NYS drinking age to 21, this appeared to be well founded. MVA's dropped 38% to their lowest level in county history. ETOH involvement in the 13-20 year old age group dropped from 68% to 35%. MVA's have rebounded however, up 116% in the first 8 months of 1986. This paper will focus on the impact of D.W.I. and safety legislation on the occurrence, age distribution and ETOH involvement in Motor Vehicle fatalities. The impact of media attention and police enforcement on MVA's will also be assessed.

FENTANYL RELATED OVERDOSE A CASE REPORT

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Madison, WI 53705

Fentanyl is a potent narcotic analgesic widely used as a clinical anesthetic. The potency of fentanyl is 100 times that of morphine. An accidental fentanyl overdose of a hospital employee is reported. Fentanyl was extracted from autopsy specimens by a modified benzene extraction procedure. Quantitation was performed by GC/MS using selected ion monitoring. Analytical methodology and fentanyl levels determined in autopsy specimens will be presented

DETECTION OF COCAINE USE AT THE 50 NG/ML LEVEL USING AN EMIT SCREEN AND GC/MS CONFIRMATION.

William A. Joern, St. Anthony's Med. Ctr., St. Louis, MO 63128.

The EMIT Cocaine test cut-off calibrator is set at 300 ng/ml of benzoyl ecgonine. However, while urine concentrations of benzoyl ecgonine between 50 and 300 ng/ml give an EMIT readout distinctly above that of the average negative urine, they must be called "negative" according to the EMIT protocol. A published GC/MS method for detecting benzoyl ecgonine in urine by an extractive alkylation procedure was extended to very low concentrations. The modified procedure uses five ions for reliable identification, employs an easily-prepared internal standard, and has a sensitivity of 30-50 ng/ml. Patient urines with EMIT readouts 50-80 units below the low calibrator were tested for the presence of benzoyl ecgonine by the modified GC/MS procedure, and more than 80% of these urines were positive. Thus, the detection time for cocaine identification can be extended to 2-3 times that found with the usual EMIT protocol.

DETECTION OF PAST AND RECURRENT MARIJUANA USE BY A MODIFIED GC/MS PROCEDURE. William A. Joern, St. Anthony's Med. Ctr., St. Louis, MO 63128.

A published GC/MS procedure for detecting the THC "acid metabolite" in urine was modified. Five ions of the PFPA-PFPOH derivative were used for improved reliability; two ions of the trideuterated internal standard were used for excellent quantitative precision; a methanolic KOH extraction was used to produce a cleaner extract; and the conditions were adjusted so that no silylation of glassware was necessary. The sensitivity of the modified procedure was 1.8 ng/ml using the MSD mass spectrometer. Patient urines were analyzed by both the new procedure and the "EMIT" method. For 32 specimens, the average and range of EMIT/GC-MS concentration ratios were 2.8 and 0.9 - 7.2, respectively. Concentrations of the "acid metabolite" measured by the GC/MS procedure may be more indicative of recent marijuana use than the EMIT semi-quantitative concentration values.

SCREENING & CONFIRMATION OF 3,4-METHYLENEDIOXY-METHAMPHETAMINE(MDMA) IN URINE:EVALUATION OF 1000 SPECIMENS. Brian Sedgwick,Peter Lo & Mike Yee,PharmChem Labs Inc,3925 Bohannon Drive, Menlo Park,CA 94025.

Methods were developed for mass screening & confirmation of MDMA in urine. Screening was by solvent extraction/TLC. MDMA had essentially the same Rf(0.27) & gave the same color reactions with ninhydrin & iodoplatinate as methamphetamine(MA). Sensitvity was approx.0.5ug/ml. Preliminary confirmation was by GC(DB-17 fused silica col.200 iso.)with NP detection using N-propylamphetamine as IS. Retention times were 1.0,1.2 & 3.9min.for MA,IS & MDMA respectively. Potential interference was noted at 3.9 min in several specimens which had screened positive at the Rf of MA/MDMA. Therefore, GC-positive specimens were subsequently confirmed by GC/MS (HP-1 cap. column,130 -160 @5 /min.) Retention time for MDMA was 4.8 min., and ion masses 58, 135 & 136 were monitored for identification & quantitation.

Two potentially "at risk" groups of 500 specimens were analysed using the above procedures. QA samples (1.0ug MDMA/ml) were interspersed with the unknowns. The first group gave 131 TLC pos.,19 GC pos. and no GC/MS pos. The second group gave 11,2 and zero pos.respectivly. All QA samples were correctly identified by TLC, GC & GC/MS.

In summary, no MDMA positives were detected in urine specimens from 1000 potentially at-risk persons.

BLOOD METHAMPHETAMINE: GC/MS QUANTITATION AND A CORRELATION OF LEVELS WITH AGENCY FOR POSITIVES RECEIVED IN 1986.
Susan A. Rasmussen, Criminalist II
San Diego Sheriff's Crime Lab, San Diego, CA

Because the RIA screen is designed for Amphetamine, it can be used only as a general screening tool. In order to leave enough blood for further screening of the negatives, a GC/MS Quant procedure using only one ml of blood has been developed.

The procedure begins with a TCA protein precipitation of the prepared sample-I.S.. The decanted supernatant is then extracted from the base into methylene chloride. An acetic anhydride derivative is formed in the methylene chloride as the solvent evaporates to low volume.

The samples and standards are injected into a Finnigan 1020 GC/MS megabore SPB 1 30 m column at 165°C with a flow rate of 22 ml/min He. The MS is set in a MI mode to scan for masses 58, 86, 100, 118, 128.

The method is sensitive to 5 ng/ml of Methamphetamine, will separate Amphetamine from B-phenethylamine and Methamphetamine from Ephedrine/Pseudoephedrine.

The positive blood Methamphetamine cases received from January to the end of July, 1986, those above 50 ng/ml, have been about equally split between traffic and non-traffic cases. A study is in progress to correlate the Methamphetamine levels with driving patterns and FST performance.