

The 1998 “Research on Drug Evidence” Report
[From the 12th ICPO / INTERPOL Forensic Science Symposium]

Robert F. X. Klein* and Patrick A. Hays

U.S. Department of Justice

Drug Enforcement Administration

Special Testing and Research Laboratory

22624 Dulles Summit Court

Dulles, VA 20166

[dea-microgram@usdoj.gov]

ABSTRACT: A reprint of the 1998 “Research on Drug Evidence” Report (a review) is provided.

KEYWORDS: INTERPOL, Illicit Drugs, Controlled Substances, Forensic Chemistry.

Important Information:

Presented at the 12th ICPO / INTERPOL Forensic Science Symposium, Lyon, France, October 20 - 23, 1998.

Authorized Reprint. Copyright INTERPOL. All rights reserved. May not be reprinted without express permission from INTERPOL.

For pertinent background, see: Klein RFX. ICPO / INTERPOL Forensic Science Symposia, 1995 - 2016. “Research on Drug Evidence”. Prefacing Remarks (and a Request for Information). *Microgram Journal* 2016;13(1-4):1-3.

Blank pages in the original document are not duplicated in this reprint.

Page numbering in the bottom center of the first two pages and the upper corners of all subsequent pages are those in the original document, while those in the footers of each page represent the *Microgram Journal* numbering.

Citations in this report from the *Journal of the Clandestine Laboratory Investigating Chemists Association* and *Microgram* were (and remain) Law Enforcement Restricted.

This is an image of the best available hard copy. Blank pages in the original document are not duplicated in this reprint.

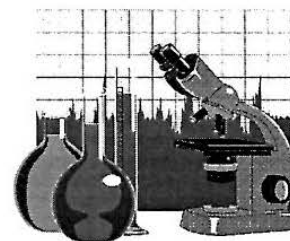
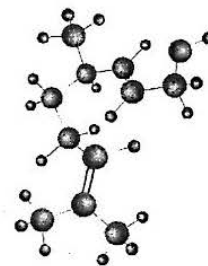
Research on Drug Evidence

Compiled by the
Drug Enforcement Administration
Office of Forensic Sciences
Special Testing and Research Laboratory

Twelfth ICPO-INTERPOL Forensic Science Symposium



*October 1998
Lyon, France*



Research On Drug Evidence

July 1, 1995 - June 30, 1998

Presented by: Robert P. Bianchi

Prepared by: Robert F.X. Klein and Patrick A. Hays

**U.S. Department of Justice
Drug Enforcement Administration
Office of Forensic Sciences
Special Testing and Research Laboratory
7704 Old Springhouse Road
McLean, VA 22102-3494 USA**

(Coordinating Laboratory)

**Twelfth ICPO - INTERPOL
Forensic Sciences Symposium
October, 1998**

Lyon, France

i

Table of Contents

I) Routine and Improved Analysis of Drug Substances	1
II) Novel Syntheses of Illicit Drugs, Precursors and Essential Chemicals	22
III) Clandestine Laboratory Appraisals and Safety Issues	26
IV) Reference Drug Standards	29
V) Comparative Analyses	31
VI) Source Determination of Drugs (Impurity Profiling)	34
VII) Analysis of Adulterants and Diluents	41
VIII) Analytical Artifacts	44
IX) New and/or Improved Instrumental Techniques	46
X) Portable Detection and Analytical Instrumentation	54
XI) Miscellaneous	56

D) Routine and Improved Analysis of Drug Substances**Problem/Issue:**

Improved methods of analysis, i.e., faster, more discriminatory, more sensitive, less costly, etc., are needed for all drugs of abuse. Additionally, standard analytical data are required for previously unknown drugs of abuse and new analog (i.e., "designer"-type) drugs.

Solution:

Illicit drug seizures and clandestine laboratory operations are continuously monitored to provide a comprehensive overview of new developments. Ongoing research in the forensic community, as well as the general analytical field, constantly provide new and/or improved methods of analysis for routine analysis of seized drugs. Case reports providing standard analytical data for new drugs and/or improved analytical protocols for known drugs are generated for the forensic and enforcement communities.

Recent Developments:

In the United States, use of methamphetamine has dramatically increased over the past three years, with concurrent increases in use of amphetamine and other related phenethylamines. Recent increases in both heroin and LSD have leveled off, while use of cocaine has somewhat decreased. Designer drugs - notably the methylenedioxy-amphetamines and 4-bromo-2,5-dimethoxyphenethylamine (aka: NEXUS or 2-CB) - are still widely used. Use of anabolic steroids is steady, with use of human growth hormones or steroid natural production-stimulating drugs (e.g., gamma-hydroxybutyrolactone or GHB) continuing to increase. Abuse of flunitrazepam (Rohypnol) as a so-called "date-rape" drug continues. A wide variety of commercial products derived from hemp (cannabis) have been marketed and represent a challenging problem for trace-level analyses of cannabinoids.

In Europe, use of amphetamines, methylenedioxyamphetamines, and heroin

remains widespread, while use of cocaine and LSD continue to grow; "crack" cocaine use is now widespread.

In the Far East, Australia and New Zealand report general across-the-board increases in drug abuse, while methamphetamine use remains ubiquitous in Japan. Cocaine use is growing throughout the Far East.

Use of cocaine, heroin, Mandrax (methaqualone), amphetamine, LSD and methylenedioxyamphetamines all continue to increase in South Africa.

Summary:

Since 1995, routine and/or new/improved methods of analysis have been reported for amphetamines, mono-substituted amphetamines, Amanita Muscaria, Ayahuasca, barbiturates, benzodiazepines, 4-bromo-2,5-dimethoxyphenethylamine (NEXUS) and related compounds, bufotenine, clenbuterol, cocaine, coca tea, dimethpramide, Dragon's Blood Incense, fenethylamine, fenfluramine, fentanyls, flunitrazepam (Rohypnol), heroin, hydrocodone, gamma-hydroxybutyric acid (or lactone) (GHB), N-(2-hydroxyethyl)-amphetamine, imazalil, inhalants, khat (Catha Edulis) and cathinone, LSD and related ergot alkaloids, marijuana and related cannabinoids, mescaline, methamphetamines, methcathinone, methylenedioxyamphetamines and related compounds, morphine and codeine, opium, opium, morphine and heroin (combined studies), 2-phenylethylamine (phenethylamine) and related compounds, poppy tea, psilocybin and psilocin, steroids, and theophylline.

References:

Amphetamines (see also methamphetamines, methylenedioxyamphetamines):

Pastor-Navarro, M.D.; Porrás-Serrano, R.; Herraéz-Hernández, R.; Campins-Falco, P., "Automated determination of amphetamine enantiomers using a two-dimensional column-switching chromatographic system for derivatization and separation," *Analyst*, **1998**, *123*

(2), 319.

Dasgupta, A.; Hart, A.P., "Distinguishing amphetamine, methamphetamine and 3,4-methylenedioxymethamphetamine from other sympathomimetic amines after rapid derivatization with propyl chloroformate and analysis by gas chromatography-chemical ionization mass spectrometry," J. Forensic Sci., **1997**, 42 (1), 106.

Esseiva, P.; Lock, E.; Gueniat, O.; Cole, M.D., "Identification and quantification of amphetamine and analogs by capillary zone electrophoresis," Science Justice, **1997**, 37 (2), 113.

Sadeghipour, F.; Varesio, E.; Gilroud, C.; Rivier, L.; Veuthey, J.L., "Analysis of amphetamine by capillary electrophoresis and liquid chromatography: application to drug seizures and cross-validation," Forensic Sci. Intl., **1997**, 86 (1,2), 1.

Sadeghipour, F.; Giroud, C.; Rivier, L.; Veuthey, J.L., "Rapid determination of amphetamines by high-performance liquid chromatography with UV detection," J. Chromatogr. A, **1997**, 761 (1-2), 71.

Van Bocxlaer, J.F.; Lambert, W.E.; Thienpont, L.; De Leenheer, A.P., "Quantitative determination of amphetamine and alpha-phenethylamine enantiomers in judicial samples using capillary gas chromatography," J. Anal. Toxicol., **1997**, 21 (1), 5.

Kaufman, M.S.; Hatzis, A.C.; Stuart, J.G., "Negative-ion chemical ionization of amphetamine derivatives," J. Mass Spec., **1996**, 31 (8), 913.

Makino, Y.; Ohta, S.; Hirobe, M., "Enantiomeric separation of amphetamine by high-performance liquid chromatography using chiral crown ether coated reversed-phase packing: application to forensic analysis," Forensic Sci. Intl., **1996**, 78, 65.

Shin, H.S.; Donike, M., "Stereospecific derivatization of amphetamines, phenol alkylamines, and hydroxyamines and quantification of the enantiomers by capillary GLC/MS," Anal. Chem., **1996**, 68 (17), 3015.

Wang, Z.; Sun, Y.L.; Sun, Z.P., "Enantiomeric separation of amphetamine and phenylephrine by cyclodextrin-mediated capillary zone electrophoresis," J. Chromatogr. A, **1996**, 735 (1-2), 295.

Cladrowarunge, S.; Hirz, R.; Kenndler, E.; Rizzi, A., "Enantiomeric separation of amphetamine related drugs by capillary zone electrophoresis using native and derivatized beta-cyclodextrin as chiral additives," J. Chromatogr. A, **1995**, 710 (2), 339.

Trenerly, V.C.; Robertson, J.; Wells, R.J., "Analysis of Illicit Amphetamine Seizures by Capillary Electrophoresis," J. Chromatogr. A, **1995**, 708, 169.

Valtier, S.; Cody, J.T., "Evaluation of Internal Standards for the Analysis of Amphetamine and Methamphetamine," J. Anal. Toxicol., **1995**, 19, 375.

Varesio, E.; Veuthey, J.L., "Chiral separation of amphetamines by high-performance capillary electrophoresis," J. Chromatogr. A, **1995**, 717 (1-2), 219.

Wang, S.M.; Ling, Y.C.; Tsai, L.C.; Giang, Y.S., "Headspace sampling and gas chromatographic mass spectrometric determination of amphetamine and methamphetamine in betel," J. Chromatogr. A, **1995**, 715 (2), 325.

Mono-Substituted Amphetamines:

Felgate, H.E.; Felgate, P.D.; James, R.A.; Sims, D.N.; Vozzo, D.C., "Recent para-methoxyamphetamine deaths," J. Anal. Toxicol., **1998**, 22 (2), 169.

Groombridge, C.R., "The Identification of 4-Methylthioamphetamine in a Drug Seizure," Microgram, 1998, 31 (5), 150.

Poortman-van der Meer, A.J., "The Identification of 4-methylthioamphetamine," Microgram, 1998, 31 (6), 174.

Amanita Muscaria:

Gennaro, M.C.; Giacosa, D.; Gioannini, E.; Angelino, S., "Hallucinogenic species in Amanita muscaria. Determination of muscimol and ibotenic acid by ion-interaction HPLC," J. Liq. Chromatogr. & Rel. Tech., 1997, 20 (3), 413.

Ayahuasca:

Casale, J.F.; Koles, J.E., "Analysis of Ayahuasca (Santo Daime)," Microgram, 1995, 28 (9), 296.

Barbiturates:

Hall, B.J.; Brodbelt, J.S., "Determination of barbiturates by solid-phase microextraction (SPME) and ion trap gas chromatography mass spectrometry," J. Chromatogr. A, 1997, 777 (2), 275.

Hassan, S.S.M.; Elnemma, E.M.; El Naby, E.H., "Mercurimetric potentiometric determination of barbiturates using a solid-state iodide ion-selective electrode," Anal. Lett., 1997, 30 (6), 1081.

Li, S.; Weber, S.G., "Determination of barbiturates by solid phase microextraction and capillary electrophoresis," Anal. Chem., 1997, 69 (6), 1217.

Morley, J.A.; Elrod, L., "Determination of pentobarbital and pentobarbital sodium in bulk drug substance and dosage forms by high performance liquid chromatography," J. Pharm. Biomed.

Anal., 1997, 16, 119.

Rukhadze, M.D.; Bezarashvili, G.S.; Sebiskveradze, M.V.; Meyer, V.R., "Separation of barbiturates with micellar liquid chromatography and optimization by a second order mathematical design," J. Chromatogr. A, 1997, 805 (1-2), 45.

Kuroda, N.; Inoue, K.; Mayahara, K.; Nakashima, K.; Akiyama, S., "Application of 3-(1,8-naphthalimido)propyl-modified silyl silica gel as a stationary phase in high-performance liquid chromatography of barbiturates and diastomeric compounds," J. Liq. Chromatogr. Relat. Technol., 1996, 19 (17,18), 2867.

Fater, Z.; Szabady, B.; Nyiredy, S., "Two-dimensional Overpressured Layer Chromatographic Separation of Barbiturated Derivatives," J. Planar Chromatogr., 1995, 8, 145.

Benzodiazepines (see also flunitrazepam):

Boonkerd, S.; Detaevernier, M.R.; Vindevogel, J.; Michotte, Y., "Migration behaviour of benzodiazepines in micellar electrokinetic chromatography," J. Chromatogr. A, 1996, 756 (1-2), 279.

Kleinschnitz, M.; Herderich, M.; Schreier, P., "Determination of 1,4-benzodiazepines by high-performance liquid chromatography electrospray tandem mass spectrometry," J. Chromatogr. B, 1996, 676 (1), 61.

Renougonnord, M.F.; David, X., "Optimized micellar electrokinetic chromatographic separation of benzodiazepines," J. Chromatogr. A, 1996, 735 (1-2), 249.

Tomita, M.; Okuyama, T., "Application of capillary electrophoresis to the simultaneous screening and quantitation of benzodiazepines," J. Chromatogr. B, 1996, 678 (2), 331.

4-Bromo-2,5-dimethoxyphenethylamine (NEXUS) and related compounds:

DeRuiter, J.; Holston, P.; Clark, C.R.; Noggle, F.T., "Liquid chromatographic and mass spectral methods of identification for regioisomeric dimethoxyamphetamines and brominated dimethoxyamphetamines," J. Chromatogr. Sci., **1998**, 36 (2), 73.

DeRuiter, J.; Clark, C.R.; Noggle, F.T., "Analysis of The Bromination Products of The Isomeric Dimethoxyphenethylamines: Differentiation of "Nexus" From Five Positional Isomers," Microgram, **1997**, 30 (5), 96.

DeRuiter, J.; Clark, C.R.; Noggle, F.T., "LC and GC-MS analysis of 4-bromo-2,5-dimethoxyphenethylamine (Nexus) and 2-propanamine and 2-butanamine analogs," J. Chromatogr. Sci., **1995**, 33 (10), 583.

Bufotenine:

Chamakura, R.P., "'Love Stone' - a Hallucinogen, an Aphrodisiac, and a Deadly Poison," Microgram, **1998**, 31 (5), 127.

Barry, T.L.; Petzinger, G.; Zito, S.W., "GC/MS Comparison of the West Indian Aphrodisiac 'Love Stone' to the Chinese Medication 'Chan Su': Bufotenine and Related Bufadienolides," J. Forensic Sci., **1996**, 41 (6), 1068.

Clenbuterol:

Krawczeniuk, A.S., "Identification of Clenbuterol: GC-Derivatization Techniques and Electro spray Mass Spectrometry," Microgram, **1996**, 29 (8), 210.

Cocaine:

Elsherbini, S.H., "Cocaine base identification and quantification," Forensic Sci. Rev., **1998**, 10, 1.

Krawczeniuk, A.S.; Bravenec, V.A., "Quantitative determination of cocaine in illicit powders by free-zone capillary electrophoresis," J. Forensic Sci., **1998**, 43 (4), 738.

Smith, R.M., "The mass spectrum of cocaine," J. Forensic Sci., **1997**, 42 (3), 475.

Campanella, L.; Colapicchioni, C.; Tomassetti, M.; Dezzi, S., "Comparison of three analytical methods for cocaine analysis of illicit powders," J. Pharm. Biomed. Anal., **1996**, 14 (8-10), 1047.

Fernandez-Abedul, M.T.; Costa-Garcia, A., "Flow injection analysis with amperometric detection of cocaine in confiscated samples," Anal. Chim. Acta, **1996**, 328 (1), 67.

Saha, U.; Mazurndar, K.K.; Sanyal, M.; Chakraborty, N.N., "Determination of Cocaine in Suspected Narcotic Substances by FTIR and HPTLC Methods," Microgram, **1996**, 29 (3), 64.

Bogusz, M.; Althoff, H.; Erkens, M.; Maier, R.; Hofmann, R., "Internally Concealed Cocaine: Analytical and Diagnostic Aspects," J. Forensic Sci., **1995**, 40 (5), 811.

Coca Tea:

Jenkins, A.J.; Llosa, T.; Montoya, I.; Cone, E.J., "Identification and quantitation of alkaloids in coca tea," Forensic Sci. Intl., **1996**, 77, 179.

Dimethpramide:

Dawson, B.A.; Black, D.B.; Cyr, T.D.; Neville, G.A.; Shurvell, H.F., "Spectroscopic

Characterization and Proof of Structure for Dimethpramide," Can. J. App. Spect., 1995, 40 (5), 131.

Dragon's Blood Incense:

Steiner, R.S., "Dragon's Blood Incense," Microgram, 1997, 30 (11), 258.

Fenethylline:

Al-Hussaini, S.R., "Counterfeit Captagon: an analytical study," Science Justice, 1996, 36, 139.

Fenfluramine:

Ferretti, R.; Gallinella, B.; Latorre, F.; Lusi, A., "Direct high-performance liquid chromatography resolution on a chiral column of dexfenfluramine and its impurities, in bulk raw drug and pharmaceutical formulations," J. Chromatogr. A, 1996, 731 (1-2), 340.

Fentanyls:

Micovic, I.V.; Roglic, G.M.; Ivanovic, M.D.; Dosenmicovic, L.; Kiricojevic, V.D.; Popovic, J.B., "The Synthesis of Lactam Analogues of Fentanyl," J. Chem. Soc. - Perkin Trans., 1996, 1 (16), 2041.

Poklis, A., "Fentanyl: A Review for Clinical and Analytical Toxicologists," Clinical Toxicology, 1995, 33 (5), 439.

Flunitrazepam (Rohypnol) (see also benzodiazepines):

McKibben, T., "Simple and Rapid Color Screening Tests for Flunitrazepam (Rohypnol)," J.

Forensic Sci., **1998** (in press).

Rucker, C., "Chemical Screening and Identification Techniques for Flunitrazepam," Microgram, **1998**, 31 (7), 198.

Churchill, K.T., "Roofies," Microgram, **1995**, 28 (10), 329.

Heroin:

Naess, O.; Rasmussen, K.E., "Micellar electrokinetic chromatography of charged and neutral drugs in acidic running buffers containing a zwitterionic surfactant, sulfonic acids or sodium dodecyl sulphate - Separation of heroin, basic by-products and adulterants," J. Chromatogr. A, **1997**, 760 (2), 245.

Holt, P.; Bruce, N.C.; Lowe, C.R., "Bioluminescent Assay for Heroin and Its Metabolites," Anal. Chem., **1996**, 68 (11), 1877.

Burla, R.; Avraham, S.; Glattstein, B.; Levy, S., "Separation of Heroin from Several Adulterants in Illicit Powders," Microgram, **1995**, 28 (9), 300.

Hydrocodone:

Hadzija, B.W.; Shrewsbury, R.P.; Cody, J.T., "Determination of hydrocodone in Tussionex extended-release suspension by high-performance liquid chromatography (HPLC)," J. Forensic Sci., **1996**, 41 (5), 878.

gamma-Hydroxybutyric Acid (or lactone) (GHB):

Wolnik, K.A.; Keitkemper, D.T.; Crowe, J.B.; Barnes, B.S.; Brueggemeyer, T.W., "Application

of Inductively Coupled Plasma Atomic Emission and Mass Spectrometry to Forensic Analysis of Sodium Gamma Hydroxy Butyrate and Ephedrine Hydrochloride," J. Anal. Atomic Spect., **1995**, 10, 177.

N-(2-Hydroxyethyl)amphetamine:

Cyr, T.; Dawson, B.; By, A.; Neville, G.; Shurvell, H., "Structural Elucidation of Unusual police Exhibits. II. Identification and Spectral Characterization of N-(2-Hydroxyethyl)amphetamine Hydrochloride," J. Forensic Sci., **1996**, 41 (4), 608.

Imazalil:

Hays, P.A.; Koles, J.E.; Morello, D.R., "Imazalil Sulfate," Microgram, **1995**, 28 (12), 389.

Inhalants:

Hindmarsh, K.W.; Taylor, A.; Fandrey, S., "Solvent Abuse - Changes in Attitudes and Knowledge?," Can. Soc. Forensic Sci., **1997**, 30 (1), 17.

Khat (Catha Edulis) and Cathinone:

Al Obaid, A.M.; Al Tamrah, S.A.; Aly, F.A.; Alwarthan, A.A., "Determination of (S)-(-)-cathinone by spectrophotometric detection," J. Pharm. Biomed. Anal., **1998**, 17 (2), 321.

Kalix, P., "Catha edulis, a plant that has amphetamine effects," Pharmacy World and Science, **1996**, 18 (2), 69.

Ripani, L.; Schiavone, S.; Garafano, L., "GC/MS identification of Catha edulis stimulant-active principles," Forensic Sci. Intl., **1996**, 78, 39.

LSD and Related Ergot Alkaloids:

Blackwell, T.M., "Identification of LSD on Single Blotter Paper via GC/MS Using Electronic Pressure Controls and Pulsed Split Injection," Microgram, **1998**, 31 (2), 51.

Frost, M.; Kohler H., "Analysis of lysergic acid diethylamide: comparison of capillary electrophoresis with laser-induced fluorescence (CE-LIF) with conventional techniques," Forensic Sci. Intl., **1998**, 92, 213.

Flieger, M.; Wurst, M.; Shelby, R., "Ergot Alkaloids - Sources, Structures and Analytical Methods," Folia Microbiologica, **1997**, 42 (1), 3.

Salamone, S.; Li, Z.; McNally, A.J.; Vitone, S.; Wu, R.S., "Epimerization Studies of LSD Using 1H Nuclear Magnetic Resonance (NMR) Spectroscopy," J. Anal. Toxicol., **1997**, 21, 492.

Webb, K.S.; Baker, P.B.; Cassells, N.P.; Francis, J.M.; Johnston, D.E.; Lancaster, S.L.; Minty, P.S.; Reed, G.D.; White, S.A., "The analysis of lysergide (LSD): The development of novel enzyme immunoassay and immunoaffinity extraction procedures together with an HPLC-MS confirmation procedure," J. Forensic Sci., **1996**, 41 (6), 938.

Marijuana and related cannabinoids:

Linacre, A.; Thorpe, J., "Detection and identification of cannabis by DNA," Forensic Sci. Intl., **1998**, 91, 71.

Ndjoko, K.; Wolfender, J.L.; Hostettmann, K., "Analysis of cannabinoids by liquid chromatography - Thermospray mass spectrometry and liquid chromatography-Tandem mass spectrometry," Chromatographia, **1998**, 47 (1-2), 72.

Backstrom, B.; Cole M.D.; Carrott, M.J.; Jones, D.C.; Davidson, G.; Coleman, K., "A preliminary study of the analysis of Cannabis by supercritical fluid chromatography with atmospheric pressure chemical ionization mass spectrometric detection," Science Justice, 1997, 37 (2), 91.

Matsunaga, T.; Ano, M.; Watanabe, K.; Yamamoto, I.; Yoshimura, H., "Analysis of nitrogen containing compounds, p-coumaroyltyramine and feruloyltyramine, for discrimination of cannabis seeds by HPLC," Jpn. J. Toxicol. Environ. Health, 1997, 43 (4), 215.

Sochocki, R.E., "Purifying Charred Marihuana Residue for the Duquenois-Levine Test," Microgram, 1997, 30 (8), 181.

Jagadish, V.; Robertson, J.; Gibbs, A., "RAPD analysis distinguishes Cannabis sativa samples from different sources," Forensic Sci. Intl., 1996, 79 (2), 113.

Mescaline:

Gennaro, M.C.; Gioannini, E.; Giacosa, D.; Siccardi, D., "Determination of mescaline in hallucinogenic Cactaccae by ion-interaction HPLC," Anal. Lett., 1996, 29 (13), 2399.

Methamphetamines (see also amphetamines and methylenedioxyamphetamines):

Bokor, I.; Trenerry, V.C.; Scheelings, P., "Separation and quantitation of optical isomers of methylamphetamine samples by capillary electrophoresis," Forensic Sci. Intl., 1997, 85 (3), 177.

Chappell, J. S., "Infrared discrimination of enantiomerically enriched and racemic samples of methamphetamine salts," Analyst, 1997, 122 (8), 755.

Kuroda, N.; Nomura, R.; Al Dirbashi, O.; Akiyama, S.; Nakashima, K., "Determination of

methamphetamine and related compounds by capillary electrophoresis with UV and laser induced fluorescence detection," J. Chromatogr. A, **1997**, 798 (1-2), 325.

Oulton, S.R., "Separation and Identification of Ephedrine, Pseudoephedrine and Methamphetamine Mixtures," Microgram, **1997**, 30 (12), 289.

Oulton, S.R., "Separation and Identification of Ephedrine, Pseudoephedrine and Methamphetamine Mixtures," J. Clan. Lab. Invest. Chem. Assoc., **1997**, 7 (4), 19.

Gavcik, J.; Stransky, Z.; Ingelse, B.A.; Lemr, K., "Capillary electrophoretic enantioseparation of selegiline, methamphetamine and ephedrine using a neutral beta-cyclodextrin epichlorhydrin polymer," J. Pharm. Biomed. Anal., **1996**, 14 (8-10), 1089.

Kaufman, M.S.; Hatzis, A.C., "Electron Impact Mass Spectrometry of N-substituted Amphetamines," Microgram, **1996**, 29 (7), 179.

Clark, C.R.; DeRuiter, J.; Valaer, A.K.; Noggle, F.T., "GC-MS analysis of acylated derivatives of methamphetamine and regioisomeric phenethylamines," J. Chromatogr. Sci., **1995**, 33 (9), 485.

Masseti, J., "Amphetamine in Suspected Methamphetamine Samples," J. Clan. Lab. Invest. Chem. Assoc., **1995**, 5 (4), 9.

Wang, S.M.; Ling, Y.C.; Tsai, L.C.; Giang, Y.S., "Headspace sampling and gas chromatographic mass spectrometric determination of amphetamine and methamphetamine in betel," J. Chromatogr. A, **1995**, 715 (2), 325.

Methylenedioxyamphetamines and related compounds:

DeRuiter, J.; Holston, P.L.; Clark, C.R.; Noggle, F.T., "Liquid chromatographic and mass

spectral methods of identification for the regioisomeric 2,3- and 3,4-methylenedioxyphenalkylamines," J. Chromatogr. Sci., **1998**, 36 (3), 131.

Furnari, C.; Ottaviano, V.; Rosati, F.; Tondi, V., "Identification of 3,4-methylenedioxyamphetamine analogs encountered in clandestine tablets," Forensic Sci. Intl., **1998**, 92, 49.

Parker, M.A.; Maronalewicka, D.; Kurrasch, D.; Shulgin, A.T.; Nichols, D.E., "Synthesis and Pharmacological Evaluation of Ring-methylated Derivatives of 3,4-(Methylenedioxy)amphetamine (MDA)," J. Med. Chem., **1998**, 41 (6), 1001.

Pearson, J.R.; Rowe, J.E., "Explorations with Ecstasy and Amphetamine derivatives," J. Clan. Lab. Invest. Chem. Assoc., **1998**, 8 (1), 29.

Sadeghipour, F.; Veuthey, J.L., "Enantiomeric separation of four methylenedioxyylated amphetamines on beta-cyclodextrin chiral stationary phases," Chromatographia, **1998**, 47 (5-6), 285.

Bovolenta, A.; Morselli, O., "Italian Clandestine Drug Market: MDEA, MDMMA, and MBDB in Street Tablets," Microgram, **1997**, 30 (1), 14.

Burns, D.T.; Lewis, R.J.; Stevenson, P., "Determination of 3,4-methylenedioxyamphetamine analogues ("Ecstasy") by proton nuclear magnetic resonance spectroscopy," Anal. Chim. Acta, **1997**, 339 (3), 259.

Dal Cason, T.A.; Young, K.; Glennon, R.A., "Cathinone: An Investigation of Several n-Alkyl and Methylenedioxy-Substituted Analogs," Pharmacol. Biochem. Behav., **1997**, 58 (4), 1109.

Dal Cason, T.A., "The characterization of some 3,4-methylenedioxycathinone (MDCATH) homologs," Forensic Sci. Intl., **1997**, 87 (1), 9.

Dal Cason, T.A.; Meyers, J.A.; Lankin, D.C., "Proton and carbon-13 NMR assignments of 3,4-methylenedioxyamphetamine (MDA) and some analogues of MDA," Forensic Sci. Intl., **1997**, 86, 15.

Dal Cason, T.A.; Meyers, J.A.; Lankin, D.C., "Erratum to Proton and Carbon-13 NMR assignments of 3,4-methylenedioxyamphetamine (MDA) and some analogues of MDA," Forensic Sci. Intl., **1997**, 87, 175.

Sadeghipour, F.; Veuthey, J.L., "Sensitive and selective determination of methylenedioxyylated amphetamines by high-performance liquid chromatography with fluorimetric detection," J. Chromatogr. A, **1997**, 787 (1-2), 137.

Baudot, P.; Dresch, M.; Dzierzynski, M.; Vicherat, A., "Identification of 3,4-methylenedioxyamphetamine derivatives by capillary gas chromatography-mass spectrometry with an ion trap detector," Ann. Falsif. Expert. Chim. Toxicol., **1996**, 89 (937), 255.

Callahan, S.A.; Latham, D.J.; Dawson, B.A.; Black, D.B.; Cyr, T.D.; Ethier, J.-C.; By, A.W.; Neville, G.A., "Identification of 3,4-Methylenedioxyethylamphetamine Hydrochloride in Street Tablets," Microgram, **1996**, 29 (5), 119.

Clark, C.R.; DeRuiter, J.; Noggle, F.T., "Chromatographic and mass spectrometric methods for the differentiation of N-methyl-1-(3,4-methylenedioxyphenyl)-2-butanamine from regioisomeric derivatives," J. Chromatogr. Sci., **1996**, 34 (5), 230.

Clark, C.R.; DeRuiter, J.; Noggle, F.T., "Analysis of 1- (3-methoxy-4,5-methylenedioxy-phenyl)-2-propanamine (MMDA) derivatives synthesized from nutmeg oil and 3-methoxy-4,5-methylenedioxybenzaldehyde," J. Chromatogr. Sci., **1996**, 34 (1), 34.

Gerhards, P.; Szigan, J., "Determination of designer drugs and ecstasy," Labor Medizin, **1996**, 19

(5), 212.

Noggle, F.T.; Clark, C.R.; DeRuiter, J.; Cain, P., "Analytical Properties of N-hydroxy-3,4-methylenedioxyamphetamine ("Flea"), a Potential New Street Drug," Microgram, **1996**, 29 (1), 10.

Pisternick, W.; Guendisch, D.; Kovar, K.-A., "HPTLC discrimination of 3,4-methylenedioxy-amphetamines of the ecstasy group using 0-benzenesulfonamido-p-benzoquinone as detection reagent," J. Planar Chromatogr.-Mod. TLC, **1996**, 9 (4), 286.

Verweij, A.M.A.; Lipman, P.J.L., "Comparison of mass spectrometric ionization techniques for the analysis of phenethylamines," J. Chromatogr. Sci., **1996**, 34 (8), 379.

Clark, C.R.; DeRuiter, J.; Valaer, A.; Noggle, F.T., "Gas chromatographic-mass spectrometric and liquid chromatographic analysis of designer butanamines related to MDMA," J. Chromatogr. Sci., **1995**, 33 (6), 328.

Noggle, F.T.; Clark, C.R.; DeRuiter, J., "Chromatographic and Mass Spectrometric Analysis of N-methyl-1-(3,4-Methylenedioxyphenyl)-2-butanamine and Regioisomeric Derivatives," Microgram, **1995**, 28 (10), 321.

Morphine and Codeine:

Hassan, S.S.; El-Naby, E.H.; Elnemma, E.M., "Kinetic determination of morphine in illicit powders using a fluoride-selective electrode based on the reaction with 1-fluoro-2,4-dinitrobenzene," Mikrochim. Acta, **1996**, 124 (1-2), 55.

Kalasz, H.; Hosztafi, S.; Csermely, T.; Gotz, H.; Szabo, M.G., "Displacement thin layer chromatography of morphine and its semi-synthetic derivatives," J. Liq. Chromatogr. & Rel.

Tech., **1996**, 19 (1), 23.

Christie, T.J.; Hanway, R.H.; Paulls, D.A.; Townsend, A., "Chemiluminescence determination of codeine by permanganate oxidation," Anal. Proc., **1995**, 32 (3), 91.

Opium:

Krenn, L.; Glantschnig, S.; Sorgner, U., "Determination of the five major opium alkaloids by reversed-phase high-performance liquid chromatography on a base-deactivated stationary phase," Chromatographia, **1998**, 47 (1-2), 21.

Budvari Barany, Z.; Szasz, G.; Gyimesi Forras, K., "Optimized and validated HPLC methods for compendial quality assessment. II. Opium alkaloids," J. Liq. Chromatogr. & Rel. Tech., **1997**, 20, 3257.

Paul, B.D.; Dreka, C.; Knight, E.S.; Smith, M.L., "Gas Chromatographic/Mass Spectrometric Detection of Narcotine, Papaverine, and Thebaine in Seeds of *Papaver somniferum*," Planta Medica, **1996**, 62, 544.

Trenerry, V.C.; Wells, R.J.; Robertson, J., "Determination of morphine and related alkaloids in crude morphine, poppy straw and opium preparations by micellar electrokinetic capillary chromatography," J. Chromatogr. A, **1996**, 718 (1), 217.

Zenk, M.H.; Tabata, M., "Opium. Its history, merits, and demerits," Nat. Med., **1996**, 50 (2), 86.

Baudot, P.; Bourbonneux, C.; Viriot, M.L.; Carre, M.C.; Andre, J.C., "Synchronous excitation spectrofluorimetry of morphine derivatives and opium. II: Papaverine, noscapine, and codeine," Ann. Falsif. Expert. Chim. Toxicol., **1995**, 88 (930), 19.

Bjornsdottir, I.; Hansen, S.H., "Determination of opium alkaloids in crude opium using non-aqueous capillary electrophoresis," J. Pharm. Biomed. Anal., **1995**, 13 (12), 1473.

Mitsui, T.; Hida, M.; Fujimura, Y., "Determination of the total amount of morphine alkaloids in opium by pyrolysis-gas chromatography using principal component analysis," J. Anal. Appl. Pyrolysis, **1995**, 32, 205.

Tetenyi, P., "Biodiversity of *Papaver somniferum* L (Opium poppy)," International Symposium on Medicinal and Aromatic Plants (Series: Acta Horticulturae), **1995**, 390, 191.

Opium, Morphine and Heroin (Combined Studies):

United Nations International Drug Control Programme, Vienna, "Recommended Methods for Testing Opium, Morphine and Heroin, Manual for Use by National Drug Testing Laboratories, Laboratory Section United Nations, New York (1998)."

2-Phenylethylamine (Phenethylamine) and related compounds:

Serennes, G.; Chabrilat, M.; Deyris, I.; Bettocchi, A., "N-methyl-1-phenylethylamine in Tablets," Microgram, **1998**, 31 (2), 44.

Groombridge, C.J., "NMR Spectroscopy in Forensic Science," Annual Reports on NMR Spectroscopy, **1996**, 32, 215.

Groombridge, C.J.; Hooker, R.H., "The Identification of 1- (4-Methylphenyl)ethylamine in a Drug Seizure," Microgram, **1996**, 29 (2), 38.

King, L.A.; Poortman-van der Meer, A.J.; Huizer, H., "1-Phenylethylamines: a new series of illicit drugs," Forensic Sci. Intl., **1996**, 77 (3), 141.

Meyer, E.; Van Bocxlaer, J.; Lambert, W.; Thienpont, L.; De Leenheer, A., " α -Phenethylamine Identified in Judicial Samples," Forensic Sci. Intl., 1995, 76 (2), 159.

Poppy Tea:

King, M.A.; McDonough, M.A.; Drummer, O.H.; Berkovic, S.F., "Poppy tea and the baker's first seizure," Lancet, 1997, 350, 716.

Psilocybin and Psilocin:

Pedersen Bjergaard, S.; Rasmussen, K.E.; Sannes, E., "Strategies for the capillary electrophoretic separation of indole alkaloids in *Psilocybe semilanceata*," Electrophoresis, 1998, 19 (1), 27.

Steroids:

Mesmer, M.Z.; Satzger, R.D., "Determination of anabolic steroids by HPLC with UV Vis-particle beam mass spectrometry," J. Chromatogr. Sci., 1997, 35 (1), 38.

Musshoff, F.; Daldrup, T.; Ritsch, M., "Black market in anabolic steroids - analysis of illegally distributed products," J. Forensic Sci., 1997, 42 (6), 1119.

Baiocchi, C.; Giacosa, D.; Roggero, M.A.; Marengo, E., "Analysis of steroids by capillary supercritical fluid chromatography with flame-ionization and electron-capture detectors," J. Chromatogr. Sci., 1996, 34 (9), 399.

Bowers, L.D.; Borts, D.J., "Separation and confirmation of anabolic steroids with quadrupole ion trap tandem mass spectrometry," J. Chromatogr. B, 1996, 687 (1), 69.

Caerhati, T.; Forgacs, E., "Effect of beta-cyclodextrin derivatives on the retention of steroidal

drugs," J. Chromatogr. B, **1996**, 681 (1), 205.

Nascimento, E.S.; Salvadori, M.C.; Ribeiro-Neto, L.M., "Determination of synthetic estrogens in illegal veterinary formulations by HPTLC and HPLC," J. Chromatogr. Sci., **1996**, 34 (7), 330.

Guiney, L.B., "Quantitation of Testosterone Propionate and Boldenone by ¹³C - NMR," Microgram, **1995**, 28 (9), 285.

Theophylline:

Perez Martinez, I.; Sagrado, S.; Medina Hernandez, M.J., "Determination of theophylline in pharmaceuticals by micellar liquid chromatography and spectrophotometric detection," J. Liq. Chromatogr. & Rel. Tech., **1996**, 19 (12), 1957.

II) Novel Syntheses of Illicit Drugs, Precursors and Essential Chemicals

Problem/Issue:

Forensic chemists must maintain familiarity with existing and new clandestine syntheses of illicit drugs in order to assist enforcement activities, to ensure safety and effectiveness during enforcement operations, and to provide expert testimony in legal proceedings.

Solution:

Illicit drug seizures and clandestine laboratory operations are continuously monitored to maintain a comprehensive overview of the field. In cases where new drugs are synthesized, or new methodologies are utilized, case reports are generated for the forensic and enforcement communities.

Recent Developments:

Expanding use of the InterNet has spread a wide variety of both new and old synthetic procedures for all drugs throughout the world. In the United States, most new syntheses have concentrated on reduction of ephedrine or pseudoephedrine to methamphetamine. Use of active metal reductions (i.e., with lithium or sodium metals in ammonia) continues to increase throughout the midwest, while a wide variety of red phosphorus/hydrionic acid or red phosphorus/iodine based reductions have been seen in the west. New reductions based on hypophosphorous acid have appeared, and some older methods, including reductive aminations of phenylacetone, have reappeared. Use of unusual sources of ephedrine (notably ground ephedra) and pseudoephedrine (primarily commercial tablets) have increased. Use of unusual solvents for salting out procedures, including new refrigerants (freons) and camping stove fuels, has dramatically increased. Counterfeit flunitrazepam (Rohypnol) tablets have appeared. Hydroponics-based marijuana operations continue to spread throughout North America, with hash oil use remaining popular in Canada.

In Europe, most new syntheses have concentrated on production of variants of methylenedioxyamphetamine; however, it remains unclear whether these new analogs are by design or rather unintended errors. In Europe and Southeast Asia, amphetamines and methylenedioxyamphetamines are commonly produced on industrial scales.

In South America, cocaine production continues to be simplified, and a large variety of commercially available farming and industrial products have been used as effective substitutes for "classic" reagents, especially in Colombia. Production of heroin continues to increase.

Summary:

Since 1995, a variety of alternate precursors, unusual substitutes for essential chemicals, and new or modified synthetic methods have been reported.

References:

Clandestine Laboratory Updates:

Cutler, R., "White Phosphorus Replacing Red Phosphorus in Idaho," J. Clan. Lab. Invest. Chem. Assoc., 1998, 8 (1), 3.

Cain, P.; Yip, S., "Notification Of A New Presentation Of Cannabis Seized In The United Kingdom," Microgram, 1997, 30 (10), 230.

Dal Cason, T., "Perspectives on "Nazi Dope" and the Mythical "Nazi Patent", " J. Clan. Lab. Invest. Chem. Assoc., 1997, 7 (2), 13.

Massetti, J., "Hypophosphorous Acid Use Increases at California Clandestine Methamphetamine Labs," J. Clan. Lab. Invest. Chem. Assoc., 1997, 7 (3), 6.

Clacher, F.; Clark, K., "Illicit steroid laboratories," Science and Justice: 1995 Annual General Meeting of the Forensic Science Society (summary), 1996, 36 (2), 123.

Fifka, P., "Pervitin (Methamphetamine) Production in Slovak Republic," J. Clan. Lab. Invest. Chem. Assoc., 1996, 6 (2), 13.

Oulton, S., "Dichlorofluoroethane in the Clandestine Manufacture of Methamphetamine," Microgram, 1996, 29 (10), 261.

Oulton, S., "Dichlorofluoroethane In the Clandestine Manufacture of Methamphetamine," J. Clan. Lab. Invest. Chem. Assoc., 1996, 6 (4), 16.

Reader, B., "Concealment and trafficking of illicit drugs," Science and Justice: 1995 Annual General Meeting of the Forensic Science Society (summary), 1996, 36 (2), 123.

Dawson, N., "The Sodium-Ammonia 'Nazi' Method of Methamphetamine Synthesis: An Historical Overview, Methodology and Case Reviews," J. Clan. Lab. Invest. Chem. Assoc., 1995, 5 (3), 12.

King, L.A.; Clarke, K.; Scott, R.J., "Unusual Defense to charge of MDMA manufacture," J. Clan. Lab. Invest. Chem. Assoc., 1995, 5 (3), 6.

Popovich, G.L., "Instant Methamphetamine," J. Clan. Lab. Invest. Chem. Assoc., 1995, 5 (3), 7.

New or Unusual Drugs and/or Precursors:

Hugel, J., "The Planned Manufacture of LSD from the Fungus *Claviceps Paspali*," J. Clan. Lab. Invest. Chem. Assoc., 1998, 8 (1), 27.

Poortman, A., "Unusual Manufacturing of MDMA in the Netherlands," J. Clan. Lab. Invest. Chem. Assoc., **1998**, 8 (1), 25.

Callahan, S.A.; Latham, D.J.; Dawson, B.A.; Black, D.B.; Cyr, T.D.; Ethier, J.-C.; By, A.W.; Neville, G.A., "Identification of an Unusual Police Exhibit as Cyclofenil, a Gonad-Stimulating Substance," Microgram, **1997**, 30 (7), 145.

Cody, J.T., "Enantiomeric composition of amphetamine and methamphetamine derived from the precursor compound famprofazone," Forensic Sci. Intl., **1996**, 80, 189.

Sorokin, V.I.; Orlova, O.S., "The Method of Acetylated Opium Manufacturing," J. Clan. Lab. Invest. Chem. Assoc., **1996**, 6 (1), 14.

Stein, D., "Use of Helicopter Oil as a Precursor Source for Piperonal," J. Clan. Lab. Invest. Chem. Assoc., **1996**, 6 (3), 17.

Valentine, M.D., "delta-9-Tetrahydrocannabinol Acetate from Acetylation of Cannabis Oil," Science Justice, **1996**, 36 (3), 195.

Clark, C.R.; Noggle, F.T.; DeRuiter, J., "Analysis of Methoxy MDA Derivatives Synthesized From Nutmeg Oil and 3-methoxy-4,5-methylenedioxybenzaldehyde," Microgram, **1995**, 28 (11), 358.

Skinner, H.F.; Oulton, S.R., "Identification and Quantitation of Hydriodic Acid Manufactured from Iodine, Red Phosphorus and Water," J. Clan. Lab. Invest. Chem. Assoc., **1995**, 5 (4), 12.

III) **Clandestine Laboratory Appraisals and Safety**

Problem/Issue:

Forensic chemists must maintain familiarity with clandestine laboratory procedures, setups and techniques in order to assist enforcement activities, to ensure safety and effectiveness during enforcement operations, and in order to provide expert testimony in Court proceedings.

Solution:

Clandestine laboratory operations are continuously reviewed to provide a comprehensive overview of the field. In cases where new methodologies are noted, or unusual safety concerns are salient, case reports are generated for the forensic and enforcement communities.

Recent Developments:

Expanding use of the InterNet has spread a wide variety of clandestine laboratory methodologies throughout the world, including basic set-up procedures, adaptations of standard consumer products as substitutes for laboratory glassware, equipment, and essential chemicals, concealment techniques, covert surveillance and countersurveillance techniques, and booby trapping.

In the United States, widespread use of active metal reductions of ephedrine (i.e., with lithium or sodium metals in ammonia) represents a serious threat to forensic, law enforcement and fire department personnel. Continuing increases in the numbers of "confined space" clandestine laboratories (e.g., buried vehicles, caves, underground chambers or hidden internal compartments in residences, etc.) has necessitated new guidelines and training for enforcement and forensic personnel entry and disassembly/cleanup. Use of booby-traps in clandestine laboratories and storage sites continues.

Summary:

Since 1995, a number of reports concerning safety in confined space laboratories or unusual hazards associated with certain methamphetamine syntheses have been reported.

References:**Safety Issues - Case Reports:**

Anjaria, M.B., "Cook' Fails Chem 101; Hydrogen Sulfide Fatality," J. Clan. Lab. Invest. Chem. Assoc., 1997, 7 (3), 5.

Kummerlowe, D., "Field Tested Methods to Render Safe 5-gallon Pressurized Tanks of Ammonia Gas Associated With Clandestine Drug Labs," J. Clan. Lab. Invest. Chem. Assoc., 1997, 7 (2), 14.

Kummerlowe, D., "Initial considerations for handling 5-gallon pressurized tanks of ammonia gas associated with clandestine drug labs," J. Clan. Lab. Invest. Chem. Assoc., 1996, 6 (4), 23.

Confined Space Laboratories:

Conibear, S.A., "What NIOSH's New Respirator Certification Regulation Means for You," J. Clan. Lab. Invest. Chem. Assoc., 1997, 7 (1), 21.

Counts, J.W., "When is a confined space not a confined space?," J. Clan. Lab. Invest. Chem. Assoc., 1997, 7 (1), 19.

Johnson, L.F., "Confined Spaces as Training Grounds," J. Clan. Lab. Invest. Chem. Assoc., 1997, 7 (2), 4.

Von Ruden, D., "Using Ventilation Blowers in Confined Spaces," J. Clan. Lab. Invest. Chem. Assoc., 1997, 7 (2), 3.

Boyd, V., "Dealing with Heat Stress Basic Precautions Can Prevent Workers in Hot Environments from Becoming Victims of Serious Heat-related Illnesses," J. Clan. Lab. Invest. Chem. Assoc., 1996, 6 (4), 18.

IV) Reference Drug Standards

Problem/Issue:

Many reference drug standards or structurally related internal standards are either commercially unavailable, or if available are extremely expensive.

Solution:

Controlled substances and their structural or isotopically labelled analogs are synthesized as needed. Internal standards are also prepared as needed. Case reports are published for new or unusual standards or improved synthetic approaches.

Recent Developments:

Increasing use of single ion-monitoring techniques for identification and quantitation of controlled substances and/or precursor compounds and essential chemicals has necessitated the development and use of isotopically labelled analogues or closely related structural isomers.

Summary:

Since 1995, several reports detailing "total syntheses" of various controlled substances have been reported.

References:

Lin, R.H.; Castells, I.; Rapoport, H., "Enantiospecific Synthesis of Natural (-)-cocaine and Unnatural (+)-cocaine from D- and L-glutamic Acid," *J. Org. Chem.*, **1998**, *63* (12), 4069.

Kachensky, D.F., "Preparation of Racemic, (-) and (+)-11-nor-delta (9)-Tetrahydrocannabinol-9-carboxylic Acid," *J. Org. Chem.*, **1997**, *62* (20), 7065.

Li, Z.Y.; Goczkutnicka, K.; McNally, A.J.; Pilcher, I.; Polakowski, S.; Vitone, S.; Wu, R.S.; Salamone, S.J., "New Synthesis and Characterization of (+)-lysergic Acid Diethylamide (LSD) Derivatives and the Development of a Microparticle-based Immunoassay for the Detection of LSD and its Metabolites," Bioconjugate Chemistry, **1997**, 8 (6), 896.

White, J.D.; Hrcniar, P.; Stappenbeck, F., "Asymmetric Synthesis of (+)-morphine. The Phenanthrene Route Revisited," J. Org. Chem., **1997**, 62 (16), 5250.

V) Comparative Analyses**Problem/Issue:**

Comparative analysis (i.e., the systematic application of impurity profiling for determination of commonality of origin) is complicated due to both the high complexity of the data and the large numbers of exhibits. Improved analytical and data handling techniques are needed.

Solution:

In-depth analysis via improved instrumental methodologies help identify discriminatory components in impurity profiles. Computer databases, sorting programs and pattern recognition/neural networks provide enhanced data handling and analysis. Case reports of new methodologies are generated for the forensic and enforcement communities.

Recent Developments:

In conjunction with impurity profiling, a number of comparative analysis protocols were reported.

Summary:

Since 1995, comparative analyses have been conducted on amphetamines, heroin, LSD blotter papers, marijuana, opium, and tablet and capsule logos. Comparative analysis has also been addressed in general terms.

References:**Amphetamines:**

Pikkarainen, A.L., "Systematic approach to the profiling analysis of illicit amphetamine," Forensic

Sci. Intl., 1996, 82, 141.

Heroin:

Stromberg, L.; Lundberg, L.; Neumann, H.; Bobon, B.; Huizer, H.; van der Stelt, N.W., "Heroin Impurity Profiling: A harmonization study for retrospective comparisons," Statens Kriminaltekniska Laboratorium (Sweden), Bundeskriminalamt (Germany), Gerechtelijk Laboratorium (The Netherlands), **December, 1997**.

Besacier, F.; Chaudron-Thozet, H.; Rousseau-Tsangaris, M.; Girard, J.; Lamotte, A., "Comparative chemical analyses of drug samples: general approach and application to heroin," Forensic Sci. Intl., 1997, 85, 113.

Levy, R.; Ravreby, M.; Meirovich, L.; Shapira-Heiman, O., "A Survey and Comparison of Heroin Seizures in Israel During 1992 by Fourier Transform Infrared Spectrometry," J. Forensic Sci., 1996, 41 (1), 6.

LSD:

Franzosa, E.S., "Current LSD Blotter Paper Designs," Microgram, 1997, 30 (8), 182.

Marijuana:

Gillan, R.; Cole, M.D.; Linacre, A.; Thorpe, J.W.; Watson, N.D., "Comparison of Cannabis Sativa by Random Amplification of Polymorphic DNA (RAPD) and HPLC of Cannabinoids: a Preliminary Study," Science Justice, 1995, 35 (3), 169.

Opium:

Shoyama, Y.; Kawachi, F.; Tanaka, H.; Nakai, R.; Shibata, T.; Nishi, K., "Genetic and alkaloid analysis of Papaver species and their F1 hybrid by RAPD, HPLC and ELISA," Forensic Sci. Intl., 1998, 91, 207.

Source Determination (Ballistics/Toolmarks):

"EUROPOL: Working-group Precursors and Synthetic Drugs, Logo Project, Synthetic Drugs Catalogue," EUROPOL Drugs Unit, 1997, File No. 2521-15r2.

Van Zyl, E.F.; Louw, M., "The Differentiation of Illicit Methaqualone Tablet Formulations Using Principal Component and Soft Independent Modeling of Class Analogy Analysis of Their Near-infrared Reflectance Spectra," J. Forensic Sci., 1995, 40 (6), 1072.

General Discussions:

Welsh, W.J.; Lin, W.; Tersigni, S.H.; Collantes, E.; Duta, R.; Carey, M.S.; Zielinski, W.L.; Brower, J.; Spencer, J.A.; Layloff, T.P., "Pharmaceutical fingerprinting: evaluation of neural networks and chemometric techniques for distinguishing among same-product Manufacturers," Anal. Chem., 1996, 68 (19), 3473.

VII) Source Determination of Drugs (Impurity Profiling)**Problem/Issue:**

Impurity profiling of drugs is important for comparative analysis protocols, geosourcing and synthetic route determinations. However, although certain drugs have been well characterized with respect to their impurity profiles, most have not been properly investigated.

Solution:

High sensitivity analytical techniques (primarily chromatographic) provide detailed profiles of trace-level impurities, ions, trace metals and stable isotopes. Identification of individual impurities enhance origin identification and comparative analyses and also aid in development of internal standards for improved accuracy and precision of analysis. Case reports are generated for the forensic and enforcement communities.

Recent Developments:

Since 1995, the ongoing and systematic effort to identify impurities and establish signature profiles via in-house syntheses has continued and expanded. Heroin impurity profiling continues in the United States, Germany and Australia. Cocaine impurity profiling continues in the United States and Europe, and has expanded in South America. Analysis of occluded solvents in finished products (notably cocaine, heroin and methamphetamine) continues, and stable isotope analyses have expanded.

Summary:

Since 1995, impurity profiling has been conducted on amphetamine, cocaine, heroin, marijuana, methamphetamine, methylenedioxyamphetamines, and precursors.

References:**Amphetamine:**

Jonson, C.S.L.; Artizzu, N., "Factors influencing the extraction of impurities from Leuckart amphetamine," Forensic Sci. Intl., **1998**, 93 (1-2), 99.

Cocaine:

Casale, J.F.; Moore, J.M.; Odeneal, N.G., "Comparative Determination of 2-Carbomethoxy-3-Alkyloxy- and Heteroaroyloxy-Substituted Tropanes in Illicit South American Cocaine Using Capillary Gas Chromatography- Single Ion Monitoring," J. Forensic Sci., **1998**, 43 (1), 125.

Lurie, I.S.; Hays, P.A.; Casale, J.F.; Moore, J.M.; Castell, D.M.; Chan, K.C.; Issaq, H.J., "Capillary electrophoresis analysis of isomeric truxillines and other high molecular weight impurities in illicit cocaine," Electrophoresis, **1998**, 19, 51.

Moore J.M.; Casale, J.F., "Cocaine profiling methodology - Recent advances," Forensic Sci. Rev., **1998**, 10, 13.

Chen, Z.M.; Meltzer, P.C., "Synthesis of 6- or 7- hydroxy and 6- or 7- methoxy tropanes," Tetrahedron Letters, **1997**, 38 (7), 1121.

Glass, R.L., "Analysis of hygrine and cuscohygrine in Coca leaves using gas chromatography and high-performance liquid chromatography," J. Agric. and Food Chem., **1997**, 45 (8), 3114.

Moore, J.M.; Casale, J.F., "Lesser alkaloids of cocaine-bearing plants. Part 1: nicotinoyl-, 2'-pyrroloyl, and 2'- and 3'-furanylecgonine methyl ester - Isolation and mass spectral

- characterization of four new alkaloids of South American *Erythroxylum coca* Var. *coca*," J. Forensic Sci., **1997**, 42 (2), 246.
- Teerhuis, N.M.; Hiemstra, H.; Speckamp, W.N., "Synthesis of Enantiopure Aza-analogues of Cocaine," Tetrahedron Letters, **1997**, 38 (1), 159.
- Bermejo-Barrera, P.; Moreda-Pineiro, A.; Moreda-Pineiro, J.; Bermejo-Barrera, A., "Determination of traces of chromium in cocaine and heroin by flameless atomic absorption spectrometry," Talanta, **1996**, 43 (1), 77.
- Casale, J.F.; Moore, J.M., "Lesser alkaloids of cocaine-bearing plants. Part III: 2-carbomethoxy-3-oxo substituted tropane esters: Detection and gas chromatographic-mass spectrometric characterization of new minor alkaloids found in South American *Erythroxylum coca* var *coca*," J. Chromatogr. A, **1996**, 756 (1-2), 185.
- Casale, J.F.; Moore, J.M., "Lesser alkaloids of cocaine bearing plants Part II. 3-Oxo-substituted tropane esters: detection and mass spectral characterization of minor alkaloids found in South American *Erythroxylum coca* var. *coca*," J. Chromatogr. A, **1996**, 749, 173.
- Glass, R.L.; Johnson, M.B., "Analysis of Cuscohygrine in Coca Leaves by High Performance Liquid Chromatography," J. Liq. Chromatogr. & Rel. Tech., **1996**, 19 (11), 1777.
- Kozikowski, A.P.; Simoni, D.; Manfredini, S.; Roberti, M.; Stoelwinder, J., "Synthesis of the 6- and 7-Hydroxylated Cocaines and Pseudococaines," Tetrahedron Letters, **1996**, 37 (30), 5333.
- Moore, J.M.; Casale, J.F.; Cooper, D.A., "Comparative determination of total isomeric truxillines in illicit, refined, South American cocaine hydrochloride using capillary gas chromatography electron capture detection," J. Chromatogr. A, **1996**, 756 (1-2), 193.

Paul, B.D.; Dreka, C.; Summers, J.L.; Smith, M.L., "One-step Esterification of Benzoylcegonine with Dimethylformamide-dipropylacetal or Dimethylformamide-disopropylacetal in the Presence of Pyridine," J. Anal. Toxicol., **1996**, 20 (6), 506.

Moore, J.M.; Casale, J.F.; Hays, P.A.; Klein, R.F.X.; Cooper, D.A., "Hygrine, bona fide alkaloid or artifact: its chemical reduction, novel di-heptafluorobutyrylation and sensitive detection in South American coca leaves using capillary gas chromatography-electron capture detection," J. Chromatogr. A, **1995**, 704, 483.

Moore, J.M.; Casale, J.F.; Fodor, G.; Jones, A.B., "Detection and Characterization of Cocaine and Related Tropane Alkaloids in Coca Leaf, Cocaine, and Biological Specimens," Forensic Sci. Rev., **1995**, 7 (2), 78.

Heroin:

Besacier, F.; Guilluy, R.; Brazier, J.L.; Chaudron-Thozet, H.; Girard, J.; Larnoue, A., "Isotopic Analysis of ¹³C as a Tool for Comparison and Origin Assignment of Seized Heroin Samples," J. Forensic Sci., **1997**, 42 (3), 429.

Sibley, J.A., "Formation of O-6-acetylmorphine in the 'Homebake' Preparation of Heroin," Forensic Sci. Intl., **1996**, 77, 159.

Wells, R.J.; Skopec, S.V.; Iavetz, R.; Robertson, J., "Trace Element Analysis of Heroin by ICP-MS," Chemistry in Australia, **1995**, 62 (7), 14.

Marijuana:

Smith, R.M., "Identification of butyl cannabinoids in marijuana," J. Forensic Sci., **1997**, 42 (4), 610.

Ross, S.A.; ElSohly, M.A., "Constituents of Cannabis sativa L. XXVIII. A review of the natural constituents 1980-1994," J. Nat. Prod., **1996**, 59 (1), 49.

Rustichelli, C.; Ferioli, V.; Vezzalini, F.; Rossi, M.C.; Ganberini, G., "Simultaneous separation and identification of hashish constituents by coupled liquid chromatography mass spectrometry (HPLC-MS)," Chromatographia, **1996**, 43 (3/4), 129.

Hida, M.; Mitsui, T.; Minami, Y.; Fujimura, Y., "Classification of hashish by pyrolysis-gas chromatography," J. Anal. Appl. Pyrolysis, **1995**, 32, 197.

Methamphetamine:

Conn, C.; Dawson, M.; Baker, A.; Keegan, J.; Fryirs, B., "Identification of N-Acetyl-methamphetamine in a Sample of Illicitly Synthesized Methamphetamine," J. Forensic Sci., **1996**, 41 (4), 645.

Noggle, F.T.; Clark, C.R.; DeRuiter, J., "Gas Chromatographic and Mass Spectral Analysis of Methamphetamine Synthesized from Allylbenzene," J. Chromatogr. Sci., **1995**, 33, 153.

Noggle, F.T.; Clark, C.R.; DeRuiter, J., "GC-MS and LC of Addition Products Formed from the Reaction of Allylbenzene and Related Arylpropenes with Acetonitrile and Sulfuric Acid," J. Chromatogr. Sci., **1995**, 33 (5), 256.

Windahl, K.L.; McTigue, M.J.; Pearson, J.R.; Pratt, S.J.; Rowe, J.E.; Sear, E.M., "Investigation of the Impurities Found in Methamphetamine Synthesized from Pseudoephedrine by Reduction with Hydriodic Acid and Red Phosphorus," Forensic Sci. Intl., **1995**, 76 (2), 97.

Precursors:

Laing, R.R.; Dawson, B., "Identification of the Major Product from The Ritter Reaction Using Safrole," J. Clan. Lab. Invest. Chem. Assoc., **1997**, 7 (2), 22.

General Discussions:

Goosens, E.C.; Stegman, K.H.; Dejong, D.; Dejong, G.J.; Brinkman, U.A.T., "Investigation of on-line reversed-phase liquid chromatography gas chromatography mass spectrometry as a tool for the identification of impurities in drug substances," Analyst, **1996**, 121 (1), 61.

Harakuwe, A.H.; Haddad, P.R., "Manipulation of separation selectivity for inorganic anions in capillary zone electrophoresis using control of electrolyte pH," J. Chromatogr. A, **1996**, 734 (2), 416.

Berridge, J.C., "Impurities in drug substances and drug products: New Approaches to quantification and qualification," J. Pharm. Biomed. Anal., **1995**, 14 (1-2), 7.

McKibben, T., "Analyses of Inorganic Components Found in Clandestine Drug Laboratory Evidence," J. Clan. Lab. Invest. Chem. Assoc., **1995**, 5 (4), 19.

Occluded Solvent Analyses:

Cartier, J.; Gueniat, O.; Cole, M.D., "Headspace analysis of solvents in cocaine and heroin samples," Science Justice, **1997**, 37 (3), 175.

Dejarne, L.E.; Lawhon, S.J.; Ray, P.; Kuhlman, M.R., "Analysis of the volatile organic compounds in seized cocaine," Proc. SPIE-Int. Soc. Opt. Eng., **1997**, 2937 (Chemistry- and Biology-Based Technologies for Contraband Detection), 2.

Mulligan, K.J.; Brueggemeyer, T.W.; Crockett, D.F.; Schepman, J.B., "Review: Analysis of organic volatile impurities as a forensic tool for the examination of bulk pharmaceuticals," J. Chromatogr. B, **1996**, 686, 85.

Schuberth, J., "Volatile organic compounds determined in pharmaceutical products by full evaporation technique and capillary gas chromatography ion-trap detection," Anal. Chem., **1996**, 68 (8), 1317.

Morello, D.R.; Meyers, R.P., "Qualitative and Quantitative Determination of Residual Solvents in Illicit Cocaine HCl and Heroin HCl," J. Forensic Sci., **1995**, 40 (6), 957.

VII) Analysis of Adulterants and Diluents**Problem/Issue:**

Most "street-level" drugs are "cut" with various adulterants and diluents. Separation and identification of these extraneous materials can be tedious. In addition, new or unusual adulterants and/or diluents are occasionally identified in drug exhibits, and standard analytical data are required for these substances. Finally, improved methods of analysis, i.e., faster, more discriminatory, less costly, etc., are needed for all cutting agents.

Solution:

Illicit drug seizures are continuously monitored to provide a comprehensive overview of adulterants and diluents. Case reports providing standard analytical data for new and/or unusual cutting agents are generated for the forensic and enforcement communities. Ongoing research in forensic community provides new and/or improved methods of analysis for routine identification of all adulterants and diluents.

Recent Developments:

In the United States dimethylsulfone and dimethylphthalate (or its isomers) were commonly identified in methamphetamine and cocaine, respectively. Additional programs for simultaneous identification of moderate quantities (i.e., 5 - 20 %) of certain cutting agents in cocaine or heroin by FT-IR spectroscopy were reported. Several unusual cutting agents were identified. Adulterants and diluents were the focus of several general surveys.

Summary:

Since 1995, several reports detailing common cutting agents were published. In addition, a number of simultaneous determinations of controlled substances and cutting agents were reported.

References:**Adulterants/Diluents:**

Blackwell, T.M., "The Differentiation of Dimethylterephthalate from Dimethylisophthalate via GC/FTIR," Microgram, 1998, 31 (2), 62.

Chew, S.L., "1,4-Butanediol in Liquid Exhibit," Microgram, 1997, 30 (7), 154.

Ely, R.A., Ed., "Dimethyl Sulfone Identified In Amphetamine and Methamphetamine Samples," J. Clan. Lab. Invest. Chem. Assoc., 1996, 6 (1), 12.

Cooper, S.D.; Toske, S.G., "Identification and differentiation of dimethyl terephthalate and its geometrical isomer dimethyl phthalate," Microgram, 1996, 29 (12), 307.

Matkovich, C.E., "Identification of Dimethyl Terephthalate in Cocaine Samples," Microgram, 1996, 29 (12), 316.

Pestaner, J.P.; Mullick, F.G.; Centeno, J.A., "Characterization of acetaminophen: Molecular microanalysis with Raman microprobe spectroscopy," J. Forensic Sci., 1996, 41 (6), 1060.

Simultaneous Analyses of Drugs and Adulterants/Diluents:

Atay, O.; Oztop, F., "Quantitative determination by using HPLC and GLC methods for cocaine HCl in synthetic binary mixtures with procaine HCl, lidocaine HCl and caffeine," Anal. Lett., 1997, 30 (3), 565.

Bautista, R.D.; Jimenez, A.I.; Jimenez, F.; Arias, J.J., "Resolution of ternary and quaternary mixtures of drugs in pharmaceutical preparations by use of spectrophotometric data in

conjunction with PLS-1 and PLS-2 data processing methods," Anal. Lett., **1996**, 29 (15), 2645.

Dautraix, S.; Guilluy, R.; Chaudron-Thozet, H.; Brazier, J.L.; Lamotte, A., "C-13 isotopic analysis of an acetaminophen and diacetylmorphine mixture," J. Chromatogr. A, **1996**, 756 (1-2), 203.

Deorsi, D.; Gagliardi, L.; Bolasco, A.; Tonelli, D., "Simultaneous determination of triprolidine, pseudoephedrine, paracetamol and dextromethorphan by HPLC," Chromatographia, **1996**, 43 (9-10), 496.

Pantea, A.; Pop, A.; Dogaru, S., "Identification of cocaine in samples also containing other local anesthetics," Rev. Chim. (Bucharest), **1996**, 47 (6), 580.

Cruz, A.; Lopez-Rivadulla, M.; Bermejo, A.M.; Sanchez, I.; Fernandez, P., "Sequential second-derivative spectroscopy of cocaine and adulterants in street drug samples. Part I: cocaine, procaine, and lidocaine," Anal. Lett., **1994**, 27 (14), 2663.

VIII)

Analytical Artifacts**Problem/Issue:**

Gas chromatographic and tandem gas chromatographic techniques are increasingly the method of choice for routine screening and/or identification of illicit drugs. However, use of high-temperature injectors with GC's occasionally results in formation of artifacts due to unimolecular rearrangements of the drug substance(s) (or adulterants or diluents) or reaction(s) of the various components in the exhibit with the injection solvent(s). Such artifacts can severely complicate drug analyses, especially when they involve the controlled substance.

Solution:

Case reports providing information of the appearance and reduction/elimination of analytical artifacts are generated for the forensic community.

Recent Developments:

Nitrites have been recognized to be problematic for analyses of cannabinoids.

Summary:

Since 1995, several case reports on artifact appearance in GC and/or GC/MS analyses of various controlled substances were reported.

References:

ElSohly, M.A.; Feng, S.; Murphy, T.P., "Improved procedure for overcoming nitrite interferences in GC-MS procedures for cannabinoids - The authors' reply," *J. Anal. Toxicol.*, **1998**, *22* (3), 256.

Frederick, D.L., "Improved procedure for overcoming nitrite interferences in GC-MS procedures for cannabinoids," *J. Anal. Toxicol.*, **1998**, *22* (3), 255.

Bogusz, M.J., "Large Amounts of Drugs May Considerably Influence the Peak Areas of Their Coinjected Deuterated Analogues Measured with APCI-LC-MS," J. Anal. Toxicol., **1997**, 21, 246.

El Sohly, M.A.; Feng, S.; Kopycki, W.J.; Murphy, T.P.; Jones, A.B.; Davis, A.; Carr, D., "A procedure to overcome interferences caused by the adulterant "Klear" in the GC-MS analysis of 11-nor-Delta (9)-THC-9-COOH," J. Anal. Toxicol., **1997**, 21 (3), 240.

Neudorfl, P.; Hupe, M.; Pilon, P.; Lawrence, A.H., "Determination of Ecgonidine Methyl Ester Vapor Pressure Using a Dynamic Gas Blending System and Gas Chromatographic Analysis," Anal. Chem., **1997**, 69 (20), 4283.

Poortman-van der Meer, A.J., "Artifacts in the GC Analysis of Amphetamine and MDA," Microgram, **1996**, 29 (4), 91.

Waggoner, R.J., "A Simple Procedure that Allows Software to Determine if a GC/MS Blank Injection is Contaminated," J. Forensic Sci., **1996**, 41 (4), 681.

Chappell, J.S., "Matrix Effects in the Infrared Examination of Methamphetamine Salts," Forensic Sci. Intl., **1995**, 75 (1), 1.

IX) New and/or Improved Instrumental Techniques**Problem/Issue:**

Forensic Chemists must maintain familiarity with updates in current instrumental techniques and become versant in new, improved methods of analysis.

Solution:

Improved/existing and new technologies are reviewed and applied to both routine and specialized analyses of drugs. In cases where improved performance is observed, case reports are generated for the forensic community.

Recent Developments:

Capillary electrophoresis and related techniques (electrokinetic chromatography, capillary electrochromatography, and micellar electrokinetic capillary chromatography) have moved to the forefront in liquid chromatographic analyses of controlled substances. Applications have included direct chiral discrimination of optical isomers without derivatization or specialized columns. Specialized injection techniques have enhanced detection limits for a variety of liquid chromatographic techniques. Raman spectroscopy has been investigated for identification of controlled substances. Laser-induced fluorescence has been utilized for ultra-trace level detection of both controlled substances and their impurity profiles.

Summary:

Since 1995, a variety of new and/or improved/existing instrumental methods have been utilized for drug analysis; most have been based on capillary electrophoretic techniques.

References:**Capillary Electrophoresis (and related CE techniques):**

Deyl, Z.; Miksik, I.; Tagliaro, F., "Advances in capillary electrophoresis," Forensic Sci. Intl., **1998**, 92, 89.

Fanali, S.; Aturki, Z.; Desiderio, C., "New strategies for chiral analysis of drugs by capillary electrophoresis," Forensic Sci. Intl., **1998**, 92, 137.

Frost, M.; Kohler H., "Analysis of lysergic acid diethylamide: comparison of capillary electrophoresis with laser-induced fluorescence (CE-LIF) with conventional techniques," Forensic Sci. Intl., **1998**, 92, 213.

Lurie, I.S., "Capillary electrophoresis of illicit drug seizures," Forensic Sci. Intl., **1998**, 92, 125.

Tagliaro, F.; Manetto, G.; Crivellente, F.; Smith, F.P., "A brief introduction to capillary electrophoresis," Forensic Sci. Intl., **1998**, 92, 75.

Jinno, K.; Han, Y.H.; Sawada, H., "Analysis of toxic drugs by capillary electrophoresis using polyacrylamide-coated columns," Electrophoresis, **1997**, 18 (2), 284.

Jinno, K.; Han, Y.; Sawada, H.; Taniguchi, M., "Capillary electrophoretic separation of toxic drugs using a polyacrylamide-coated capillary," Chromatographia, **1997**, 46 (5-6), 309.

Lurie, I.S., "Separation selectivity in chiral and achiral capillary electrophoresis with mixed cyclodextrins," J. Chromatogr. A, **1997**, 792, 297.

Altria, K.D., "Determination of drug-related impurities by capillary electrophoresis," J.

Chromatogr. A, 1996, 735 (1-2), 43.

Belder, D.; Stockigt, D., "Analysis of basic pharmaceuticals by capillary electrophoresis in coated capillaries and on-line mass spectrometric detection," J. Chromatogr. A, 1996, 752 (1-2), 271.

Dhulst, A.; Verbeke, N., "Chiral analysis of basic drugs by oligosaccharide-mediated capillary electrophoresis," J. Chromatogr. A, 1996, 735 (1-2), 283.

Dittmann, M.A.; Rozing, G.P., "Capillary electrochromatography - a high-efficiency micro-separation technique," J. Chromatogr. A, 1996, 744, 63.

Fanali, S., "Identification of chiral drug isomers by capillary electrophoresis," J. Chromatogr. A, 1996, 735 (1-2), 77.

Gaus, H.J.; Gogus, Z.Z.; Schmeer, K.; Behnke, B.; Kovar, K.A.; Bayer, E., "Separation and identification of designer drugs with capillary electrophoresis and on-line connection with ionspray mass spectrometry," J. Chromatogr. A, 1996, 735 (1-2), 221.

Janini, G.M.; Muschik, G.M.; Issaq, H.J., "Electrokinetic Chromatography in suppressed electroosmotic flow environment: Use of a charged cyclodextrin for the separation of enantiomers and geometric isomers," Electrophoresis, 1996, 17 (10), 1575.

Koppenhoefer, B.; Epperlein, U.; Christian, B.; Lin, B.; Ji, Y.; Chen, Y., "Separation of enantiomers of drugs by capillary electrophoresis. 3. Beta-cyclodextrin as chiral solvating agent," J. Chromatogr. A, 1996, 735 (1-2), 333.

Kuffner Jr., C.A.; Marchi, E.; Morgado, J.M.; Rubio, C.R., "Capillary Electrophoresis and Daubert: Time for Admission," Anal. Chem. News and Features, 1996, April 1, 241 A.

Leung, G.N.W.; Tang, H.P.O.; Tso, T.S.C.; Wan, T.S.M., "Separation of basic drugs with non-aqueous capillary electrophoresis," J. Chromatogr. A, **1996**, 738 (1), 141.

Lurie, I., "Application of capillary electrophoresis to the analysis of seized drugs," American Laboratory, **1996** (January) 26.

McGrath, G.; Smyth, W.F., "Large-volume sample stacking of selected drugs of forensic significance by capillary electrophoresis," J. Chromatogr. B, **1996**, 681 (1), 125.

McGrath, G.; McClean, S.; Okane, E.; Smyth, W.F.; Tagliaro, F., "Study of the capillary zone electrophoretic behaviour of selected drugs, and its comparison with other analytical techniques for their formulation assay," J. Chromatogr. A, **1996**, 735 (1-2), 237.

Nishi, H., "Enantiomer separation of basic drugs by capillary electrophoresis using ionic and neutral polysaccharides as chiral selectors," J. Chromatogr. A, **1996**, 735 (1-2), 345.

Nishi, H., "Enantiomer separation of drugs by electrokinetic chromatography," J. Chromatogr. A, **1996**, 735 (1-2), 57.

Tagliaro, F.; Turrina, S.; Smith, F.P., "Capillary electrophoresis: principles and applications in illicit drug analysis," Forensic Sci. Intl., **1996**, 77, 211.

Tagliaro, F.; Smith, F.R., "Capillary Electrophoresis: A New Analytical Tool," Bulletin of the International Association of Forensic Toxicologists, **1996**, 26 (2), 25.

Tanaka, Y.; Yanagawa, M.; Terabe, S., "Separation of neutral and basic enantiomers by cyclodextrin electrokinetic chromatography using anionic cyclodextrin derivatives as chiral pseudo-stationary phases," J. High Resol. Chromatogr., **1996**, 19, 421.

Walker, J.A.; Marche, H.L.; Newby, N.; Bechtold, E.J., "A free zone capillary electrophoresis method for the quantitation of common illicit drug samples," J. Forensic Sci., **1996**, 41 (5), 824.

Aumatell, A.; Guttman, A., "Ultra-fast chiral separation of basic drugs by capillary electrophoresis," J. Chromatogr. A, **1995**, 717 (1-2), 229.

Bjornsdottir, I.; Hansen, S.H., "Evaluation of the use of cyclodextrins in chiral separation of basic drug substances by capillary electrophoresis," Chirality, **1995**, 7 (4), 219.

Curcuruto, O.; Zaramella, A.; Hamdan, M., "Capillary Zone Electrophoresis/Electrospray Ionization Mass Spectrometry for the Characterization of Drugs of Forensic Interest," Rapid Communication in Mass Spectrometry, **1995**, 9, 1487.

Gas Chromatography (and GC/MS):

Galipo, R.C.; Morgan, S.L.; Brewer, W.E., "A sample concentrator for sensitivity enhancement in chromatographic analyses," Anal. Chem., **1998**, 70 (10), 2191.

Urry, F.M.; Kushnir, M.; Nelson, G.; McDowell, M.; Jennison, T., "Improving ion mass ratio performance at low concentrations in methamphetamine GC-MS assay through internal standard selection," J. Anal. Toxicol., **1996**, 20 (7), 592.

Liu, R.H.; Foster, G.; Cone, E.J.; Kumar, S.D., "Selecting an Appropriate Isotopic Internal Standard for Gas Chromatography/Mass Spectrometry Analysis of Drugs of Abuse - Pentobarbital Example," J. Forensic Sci., **1995**, 40 (6), 983.

Malcolm, M.J.; Hudson, J.C.; Proulx, J.G.F.; Sharp, M.E.; Whiting, C., "Internal Quality Control of a General GC Drug Screen in Forensic Toxicology: Experience, Questions, Proposals," Can. Soc. Forensic Sci., **1995**, 28 (3), 215.

High-Performance Liquid Chromatography (and tandem HPLC techniques):

Achilli, G.; Cellerino, G.P.; Deril, G.V.M.; Tagliaro, F., "Determination of illicit drugs and related substances by high-performance liquid chromatography with an electrochemical coulometric-array detector," *J. Chromatogr. A*, **1996**, *729* (1-2), 273.

Duenas, E.V.; Forero, M.E., "Standardized Methods to Separate and Identify Cocaine, Morphine, Heroin, Codeine, Papaverine, Benzocaine, Procaine, Lidocaine by High-Efficiency, Liquid Chromatography with Diode Array Detector (HPLC-DAD)," *Microgram*, **1996**, *29* (8), 207.

Sellers, J.K.; Duffitt, G.L.; Gaines, M.L.; Liu, R.H., "High performance liquid chromatographic analysis of enantiomeric composition of abused drugs," *Forensic Sci. Rev.*, **1996**, *8*, 91.

Wu, M.T.; Aderjan, R., "The effect of chromatographic conditions on the retention indices of forensically relevant substances in reversed-phase HPLC," *J. Liq. Chromatogr. & Rel. Tech.*, **1996**, *19* (12), 1967.

Theodondis, G.; Papadoyarlnis, I.; Vasilikiotis, G.; Tsoukali-Papadopoulou, H., "Reversed Phase High-Performance Liquid Chromatography-Photodiode-Array Analysis of Alkaloid Drugs of Forensic Interest," *J. Chromatogr.*, **1995**, *668*, 253.

Valtier, S.; Cody, J.T., "Evaluation of Internal Standards for the Analysis of Amphetamine and Methamphetamine," *J. Anal. Toxicol.*, **1995**, *19*, 375.

Micellar Electrokinetic Capillary Chromatography:

Lurie, I.S., "Application of micellar electrokinetic capillary chromatography to the analysis of illicit drug seizures," *J. Chromatogr. A*, **1997**, *780* (1-2), 265.

Naess, O.; Rasmussen, K.E., "Micellar electrokinetic chromatography of charged and neutral drugs in acidic running buffers containing a zwitterionic surfactant, sulfonic acids or sodium dodecyl sulphate - Separation of heroin, basic by-products and adulterants," J. Chromatogr. A, 1997, 760 (2), 245.

Nishi, H.; Terabe, S., "Micellar electrokinetic chromatography - perspectives in drug analysis," J. Chromatogr. A, 1996, 735 (1-2), 3.

Tagliaro, F.; Smith, F.P.; Turrina, S.; Equisetto, V.; Marigo, M., "Complementary use of capillary zone electrophoresis and micellar electrokinetic capillary chromatography for mutual confirmation of results in forensic drug analysis," J. Chromatogr. A, 1996, 735 (1-2), 227.

Trenerry, V.C.; Wells, R.J.; Robertson, J., "Determination of morphine and related alkaloids in crude morphine, poppy straw and opium preparations by micellar electrokinetic capillary chromatography," J. Chromatogr. A, 1995, 718, 217.

Other General Analytical Methods:

Sands, H.S.; Hayward, I.P.; Kirkbride, T.E.; Bennett, R.; Lacey, R.J.; Batchelder, D.N., "UV-excited Resonance Raman Spectroscopy of Narcotics and Explosives," J. Forensic Sci., 1998, 43 (3), 509.

Fitzgerald, R.L.; O'Neal, C.L.; Hart, B.J.; Poklis, A.; Herold, D.A., "Comparison of an ion-trap and a quadrupole mass spectrometer using diazepam as a model compound," J. Anal. Toxicol., 1997, 21 (6), 445.

Gal, T.; Veress, T.; Ambrus, I., "Sample preparation of illicit drugs for FT-IR microspectrophotometry," Mikrochim Acta, Suppl., 1997, 14 (Progress in Fourier Transform Spectroscopy), 377.

Horvath, E.; Mink, J.; Kristof, J., "Surface-enhanced Raman spectroscopy as a technique for drug analysis," Mikrochim Acta, Suppl., **1997**, *14* (Progress in Fourier Transform Spectroscopy), 745.

Peterson, K.L.; Logan, B.K.; Christian, G.D.; Ruzicka, J., "Sequential-injection extraction for sample preparation," Anal. Chim. Acta, **1997**, *337* (1), 99.

Brazier J.L., "Use of Isotope Ratios in Forensic Analysis," Ynon, Jehuda, Ed., Forensic Applications of Mass Spectrometry, Boca Raton, FL: CRC Press Inc., **1995**, 259.

Pfeifer, A.M.; Kovar, K.A., "Identification of LSD, MBDB, and Atropine in Real Samples with On-Line HPTLC-FTIR Coupling," J. Planar Chromatogr., **1995**, *8*, 388.

X) Portable Detection and Analytical Instrumentation**Problem/Issue:**

New trade agreements and the easing of formally restrictive national and international borders have resulted in dramatic increases in cargo transshipments and personal travel, thereby complicating drug inspection and interdiction efforts at POE's. Discovery and confirmational analysis of suspected drugs in cargo or on individuals is severely hampered by the lack of on-site detection and/or analytical equipment.

Solution:

Development of portable and highly sensitive detectors for drug detection and analyses allows law enforcement personnel and/or forensic chemists to perform screening type analyses on-site. In those cases where new methodologies have proven effective, case reports are generated for the forensic and enforcement communities.

Recent Developments:

Use of ion mobility spectrometers has become routine in the United States, and has resulted in numerous seizures of controlled substances (primarily cocaine) at POE's, highway monitoring stations, on board marine vessels (both in port and on the high seas), and at individual buildings (both residential and commercial). Other ongoing efforts involve further miniaturization of various GC, GC/MS, and ion mobility-type instruments and development of new technologies based on surface-acoustic-wave (SAW), pulsed neutron or biosensor technologies. This field continues to expand very rapidly.

Summary:

Since 1995, a variety of new, portable vapor and/or particle detectors have been reported for drug analyses. Several instruments based on fast neutron analyses have also been reported.

References:

Brown, P.A., "Field applications of ion mobility spectrometry," Proc. SPIE-Int. Soc. Opt. Eng., 1997, 2937 (Chemistry- and Biology-Based Technologies for Contraband Detection), 154.

Goubran, R.A.; Lawrence, A.H., "DSP techniques for narcotic detection using ion mobility spectrometry," IEEE Instrum. Meas. Technol. Conf., 1997, 1, 404.

Orzechowska, G.E.; Poziomek, E.J.; Tersol, V., "Use of solid phase microextraction (SPME) with ion mobility spectrometry," Anal. Lett., 1997, 30 (7), 1437.

Holt, P.A., "Particle size analysis of six illicit heroin preparations seized in the U.K.," Forensic Sci. Intl., 1996, 81, 17.

Pilon, P.; Hupe, M.; Chauhan, M.; Lawrence, A., "Development of Test Material for Narcotics Detection Equipment; Sand/Drug Mixtures," J. Forensic Sci., 1996, 4 (3), 371.

Brown, P.A.; Comparin, J.H., "Application of the Ionscan for the detection of methamphetamine and ephedrine in abandoned clandestine laboratories," NASA Conf. Publ (Third International Workshop on Ion Mobility Spectrometry, 1994), 1995, 3301, 245.

Fink, C.L.; Micklich, B.J.; Yule, T.J.; Humm, P.; Sagalovsky, L.; Martin, M.M., "Evaluation of neutron techniques for illicit substance detection," Nucl. Instrum. Methods Phys. Res., Sect. B, 1995, 99 (1-4), 748.

Womble, P.C.; Schultz, F.J.; Vourvopoulos, G., "Nondestructive characterization using pulsed fast-thermal neutrons," Nucl. Instrum. Methods Phys. Res., Sect. B, 1995, 99 (1-4), 757.

XI

Miscellaneous

References:

Khadka, S., "Useful Internet Websites For Safety and Health Professionals," Microgram, 1998, 31 (3), 90.

O'Donnell, J.F.; Paulson, J.D., "Solubility of Cocaine Free Base and Cocaine Hydrochloride in Gasoline," Microgram, 1998, 31 (2), 67.

Azoury, M.; Meirovich, L.; Refae, E., "Computerized Management of a Forensic Analytical Laboratory," Microgram, 1997, 30 (12), 297.

Chamakura, R.P., "Forensic Science and the Internet - Current Utilization and Future Potential," Forensic Sci. Rev., 1997, 9, 97.

Christian, D., "Ammonium Molybdate Crystal Test for Phosphorus," J. Clan. Lab. Invest. Chem. Assoc., 1997, 7, 28.

Hatcher, A.P.; Ryan, C.R., "Response to Claim of Error by Drug Enforcement Administration (DEA) Chemist in Calculating Quantity of Methadone Synthesized from Precursor Chemicals," J. Forensic Sci., 1997, 42 (5), 963.

Liu, C.L.; Bowers, L.D., "Mass spectrometric characterization of the beta-subunit of human chorionic gonadotropin," J. Mass Spec., 1997, 32 (1), 33.

McKibben, T., "Protecting Group Chemistry," J. Clan. Lab. Invest. Chem. Assoc., 1997, 7 (4), 30.

- Nichols, R.G., "Drug proficiency test false positives: a lack of critical thought," Science Justice, 1997, 37 (3), 191.
- Skalican, Z.; Koblíha, Z.; Halamek, E., "Ionic associates of phencyclidine with sulfophthaleins and azo dyes," Anal. Lett., 1997, 30 (7), 1349.
- Thornton, J.I., "Visual color comparisons in forensic science," Forensic Sci. Rev., 1997, 9, 37.
- Zedeck, M., "Drug Enforcement Administration (DEA) Chemists Erred in Calculating Quantity of Methadone that could be synthesized from precursor chemicals," J. Forensic Sci., 1997, 42 (2), 349.
- Casale, J.F.; Meyers, R.P., "The Stability of Cocaine in Agua Rica/Agua Madre," Microgram, 1996, 29 (7), 175.
- Evet, I.W., "Expert evidence and forensic misconceptions of the nature of exact science," Science Justice, 1996, 26 (2), 118.
- Hutchinson, K., "The Manufacture of Cannabis Sativa For Legitimate Applications," J. Clan. Lab. Invest. Chem. Assoc., 1996, 6 (4), 20.
- Johnson, E.L., "Alkaloid content in Erythroxylum, coca tissue during reproductive development," Phytochemistry, 1996, 42 (1), 35.
- Johnson, E.L.; Foy, C.D., "Biomass Accumulation and Alkaloid Content in Leaves of Erythroxylum coca and Erythroxylum novogranatense var. novogranatense Grown in Soil with Varying pH," J. Plant Physiol., 1996, 149, 444.
- Malcolm, M.; Gutfriend, M., "Chemistry against crime," Can. Chem. News, 1996, 48 (9), 13.

Malmusi, L.; Dukat, M.; Young, R.; Teitler, M.; Darmani, N.A.; Ahmad, B.; Smith, C.; Glennon, R.A., "1,2,3,4-Tetrahydroisoquinoline analogs of phenylalkylamine stimulants and hallucinogens," Med. Chem. Res., **1996**, 6 (6), 400.

Malmusi, L.; Dukat, M.; Young, R.; Teitler, M.; Darmani, N.A.; Ahmad, B.; Smith, C.; Glennon, R.A., "1,2,3,4-Tetrahydroisoquinoline and related analogs of the phenylalkylamine designer drug MDMA," Med. Chem. Res., **1996**, 6 (6), 412.

Monte, A.P.; Maronalewicka, D.; Parker, M.A.; Wainucott, D.B.; Nelson, D.L.; Nichols, D.E., "Dihydrobenzofuran analogues of hallucinogens. 3. Models of 4-substituted (2,5-dimethoxy-phenyl)alkylamine derivatives with rigidified methoxy groups," J. Med. Chem., **1996**, 39 (15), 2953.

Rothchild, R., "Some Considerations for Planning and Site Preparation for Modern Laboratory Instrumentation," J. Clan. Lab. Invest. Chem. Assoc., **1996**, 6 (1), 15.

Rothchild, R., "Some Considerations for Planning and Site Preparation for Modern Laboratory Instrumentation," Microgram, **1996**, 29 (3), 69.

Tessarolo, A.A.; Marignani, A., "Forensic Science and the Internet," Can. Soc. Forensic Sci., **1996**, 29 (2), 87.

Sharp, M.E.; Voll, L.J., "Modification of an extraction procedure for acidic and neutral drugs," Can. Soc. Forensic Sci., **1995**, 28, 171.

General Surveys:

Barrio, G.; Saavedra, P.; de la Fuente, L.; Royuela, L., "Purity of cocaine seized in Spain, 1985-1993: variations by weight, province and year of seizure," Forensic Sci. Intl., **1997**, 85, 15.

Henderson, G. L., "Designer drugs," Anal. Toxicol. Clin., Forensic Pharm. Chem., 1997, 685.

Edited by: Brandenberger, Hans; Maes, Robert A. A. de Gruyter: Berlin, Germany.

King, L.A., "Drug content of powders and other illicit preparations in the UK," Forensic Sci. Intl., 1997, 85, 135.

Gough, T., "Illicit drug trafficking," Science and Justice: 1995 Annual General Meeting of the Forensic Science Society (summary), 1996, 36 (2), 123.

King, L.A., "Designer Drugs Related to Amphetamine (1990-1996)," J. Clan. Lab. Invest. Chem. Assoc., 1996, 6 (3), 15.

Levy, R.; Ravreby, M.; Meirovich, L.; Shapira-Heiman, O., "A Survey and Comparison of Heroin Seizures in Israel During 1992 by Fourier Transform Infrared Spectrometry," J. Forensic Sci., 1996, 41 (1), 6.

Lodge, B.A., "Canadian designer drugs," Science and Justice: 1995 Annual General Meeting of the Forensic Science Society (summary), 1996, 36 (2), 123.

McGregor, A., "Global amphetamine abuse causes concern," Lancet, 1996, 348 (9041), 1579.

King, L.A., "Estimating the Proportion of UK Drug Consumption Which is Imported on the Basis of Customs and Police Seizures for Particular Drugs," Forensic Sci. Intl., 1995, 76 (3), 217.

Peterson, J.L.; Markham, P.N., "Crime Laboratory Proficiency Testing Results, 1978-1991, I: Identification and Classification of Physical Evidence," J. Forensic Sci., 1995, 40 (6), 994.