The 1995 "Research on Drug Evidence" Report
[From the 11th ICPO / INTERPOL Forensic Science Symposium]

Robert F. X. Klein
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 1995 "Research on Drug Evidence" Report (a review) is provided.

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

Important Information:

Presented at the 11th ICPO / INTERPOL Forensic Science Symposium, Lyon, France, November 21 - 24, 1995.

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.

Citations in this report from *Microgram* were (and remain) Law Enforcement Restricted. Most citations listed as "Personal Communications" were unpublished reports from "assisting laboratories" provided to the Laboratory Director of the Special Testing and Research Laboratory upon his request; note that this was the last Symposium where such reports were requested for the "Research on Drug Evidence" review.

Page numbering in the bottom center of the first three 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.

Although not shown in the document, this review was prepared by the author listed above (Klein), and the summary presentation at the Symposium was provided by Richard S. Frank.

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

June, 1992 - June, 1995

Prepared by: U.S. Department of Justice
Drug Enforcement Administration
(Coordinating Laboratory)

Assisted by: Bundeskriminalamt
Kriminaltechnisches Institut
Wiesbaden, Germany

Australian Government Analytical Laboratories
Canberra, Australia

Victoria Forensic Science Center
Victoria, Australia

National Research Institute of Police Science
Tokyo, Japan

Crime Laboratory
National Bureau of Investigation
Helsinki, Finland

Laboratorio de Criminalistica
Policia de Investigaciones de Chile
Santiago, Chile

Forensic Sciences Service
Stadtpolizei Zurich
Zurich, Switzerland

Forensic Science Laboratory
South African Police Service
Pretoria, Republic of South Africa

Institute of Forensic Science
Sofia, Bulgaria

Eleventh ICPO - INTERPOL
Forensic Sciences Symposium
November, 1995

Lyon, France

Table of Contents

I) Routine and Improved Analysis of Drug Substances

Problem/Issue:

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

Solution:

Illicit drug seizures are constantly monitored to provide a comprehensive overview of new developments. Case reports providing standard analytical data for new drugs are generated for the forensic and enforcement communities. 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.

Recent Developments:

In the United States, use of both heroin and LSD are increasing, while use of cocaine has levelled off and shows some signs of decreasing. "Smoking" heroin has increased, due to both increased purity levels and fear of contracting illnesses (AIDS and Hepatitis B) from use of intraveneous needles. Designer drugs are sporadically noted; the most prevalent of the new designer drugs is 4-bromo-2,5-dimethoxyphenethylamine (aka: NEXUS or 2-CB). Use of anabolic steroids is steady, with use of human growth hormones or steroid natural production-stimulating drugs (e.g., gamma-hydroxybutyric acid) showing some increases. Among the amphetamines, use of "ice" methamphetamine continues to be only a regional problem, while use of methcathinone (aka: "CAT") has spread slowly. Abuse of flunitrazepam (Rohypnol) has unexpectedly and dramatically increased and throughout the American southwest.

In Europe, use of amphetamines and heroin remains widespread, while use of cocaine and LSD are both growing; "crack" cocaine use is now evident in many of the larger cities. "Record-level" seizures of bulk cocaine shipments (up to metric ton

quantities) have been reported by several nations. Among the "designer drugs," use of the methylenedioxyamphetamines has rapidly expanded, with several analogs (notably N-ethyl) and the corresponding methylenedioxyphenyl-2-butanamines also appearing.

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 slowly growing throughout the Far East.

Summary:

Since 1992, routine and/or new/improved methods of analysis have been reported for amphetamines (1-11), Angel Trumpet (12), barbiturates (13-14), benzodiazepines (15-19) 4-bromo-2,5-dimethoxyphenethylamine (NEXUS) (20), bufotenine (21-24), cocaine (25-29), dimethpramide (30-31), dimethylaminorex (32), fenethylline (33), fentanyls (34-35), 4-fluoroamphetamine and 4-fluoromethamphetamine (36), heroin (37-42), gamma-hydroxybutyric acid (GHB) (43), N-(2-hydroxyethyl)amphetamine (44), N-hydroxy-3,4-methylenedioxyamphetamine (45), inhalants (45-47), Jimson Weed (48), Khat (49-53), LSD (54-60), marijuana (61-63), methylenedioxyamphetamines and methylenedioxyphenylbutanamines (64-68), methcathinone and cathinone (69-75), methylmethaqualone (76), N-methyl-1-phenethylamine (77), opium (78-79), PCP and PCP analogs (80-82), and steroids (83-91).

References:

Amphetamines:

(1) LeBelle, M.J.; Savard, C.; Dawson, B.A.; Black, D.B.; Katyal, L.K.; Zrcek, F.; By, A.W. "Chiral Identification and Determination of Ephedrine, Pseudoephedrine, Methamphetamine and Methcathinone by Gas Chromatography and Nuclear Magnetic Resonance," Forensic Sci. Intnl. 1995, 71, 215.

(2) Munro, C.H.; White, P.C. "Evaluation of Diazonium Salts as Visualization Reagents for the Thin Layer Chromatographic Characterization of Amphetamines," Science Justice 1995, 35, 37.

(3) Chen, Y.-P.; Hsu, M.-C.; Chien, C.S. "Analysis of Forensic Samples Using Precolumn Derivatization with (+)-1-(9-Fluorenyl)ethylchloroformate and Liquid Chromatography with Fluorimetric Detection," J. Chromatogr., A 1994, 672, 135.

(4) Longo, M.; Martines, C.; Rolandi, L.; Cavallaro, A. "Simple and Fast Determination of Some Phenethylamines in Illicit Tablets by Base-Deactivated Reversed Phase HPLC," J. Liq. Chromatogr. 1994, 17, 649.

(5) Rizzi, A.M.; Hirz, R.; Cladrowa-Runge, S.; Jonsson, H. "Enantiomeric Separation of Amphetamine, Methamphetamine, and Ring Substituted Amphetamines by Means of a beta-Cyclodextrin-Chiral Stationary Phase," Chromatographia 1994, 39, 131.

(6) Melgar, R.; Kelly, R.C. "A Novel GC/MS Derivatization Method for Amphetamines," J. Anal. Toxicol. 1993, 17, 399.

(7) Bailey, V.A.; Buer, L.O. "The Stability of Dimethylamphetamine Upon Exposure to Heat, Air and Moisture," Microgram 1993, 26, 96.

(8) Madden, J.E.; Pearson, J.R.; Rowe, J.E. "Differentiation of Side Chain Isomers of Methamphetamine Using Gas Chromatography, High-Performance Liquid Chromatography and Mass Spectrometry," Forensic Sci. Intnl. 1993, 61, 169.

(9) Hideg, Z.; Dinya, Z. "A Simple and Rapid Method for the Identification of Amphetamine and Methamphetamine Hydrochlorides by Mass Spectrometry," Anal. Lett. 1993, 26, 2637.

(10) Lurie, I.S. "Micellar Electrokinetic Capillary Chromatography of the Enantiomers of Amphetamines, Methamphetamine and their Hydroxyphenethylamine Precursors," J. Chromatogr. 1992, 605, 269.

(11) Kram, T.C.; Lurie, I.S. "The Determination of Enantiomeric Composition of Methamphetamine by ^{1}H-NMR Spectroscopy," Forensic Sci. Intnl. 1992, 55, 131.

Angel Trumpet:

(12) Churchill, K.T. "Angel Trumpet," Microgram 1995, 28, 250.

Barbiturates:

(13) Roy, M. "Analysis of Barbiturates Using Ion Trap and Quadrupole Mass Spectrometers: A Comparison," J. Can. Soc. Forens. Sci. 1994, 27, 19.

(14) Ryan, T.W. "Resolution of the Non-Specific Spectra of Barbiturates by UV-Photodiode Array Detection. II. Effects of Sample Concentration on Spectral Matching Accuracy," J. Liq. Chromatogr. 1993, 16, 33.

Benzodiazepines:

(15) Rochholz, G.; Ahrens, B.; Schuetz, H. "Modified Screening Procedure with Fluorescence Detection for Flunitrazepam," Arzneimittel Forschung 1995 (?), 44, 469.

(16) Ripani, L.; Lovera, P.; Muzi, F.; Schiavone, S. "GC/MS Analysis of a Fruit Juice Extract that was Suspected to be a Narcotic Beverage," Microgram 1994, 27, 149.

(17) Neville, G.A.; Beckstead, H.D.; Shurvell, H.F. "A Fourier Transform-Raman and Infrared Vibrational Study of Delorazepam, Fludiazepam, Flurazepam and Tetrazepam," J. Pharm. Sci. 1994, 83, 143.

(18) Shimamine, M.; Masunari, T.; Nakahara, Y. "Identification of Drugs of Abuse by Diode-Array Detection. I. Screening Test and Identification of Benzodiazepines by HPLC-DAD with ICOS Software System," Eisei Shikensho Hokoku 1993, 111, 47.

(19) Lurie, I.S.; Cooper, D.A.; Klein, R.F.X. "High-Performance Liquid Chromatographic Analysis of Benzodiazepines Using Diode-Array, Electrochemical and Thermospray Mass Spectrometric Detection," J. Chromatogr., A 1992, 598, 59.

4-Bromo-2,5-dimethoxyphenethylamine (NEXUS):

(20) Noggle, F.T.; DeRuiter, J.; Clark, C.R. "Analytical Profiles of 4-Bromo-2,5-Dimethoxyphenethylamine (NEXUS) and Related Precursor Chemicals," Microgram 1994, 27, 343.

Bufotenine:

(21) Chamakura, R.P. "Bufotenine - A Hallucinogen in Ancient Snuff Powders of South America and a Drug of Abuse on the Streets of New York," Forensic Sci. Rev. 1994, 6, 1.

(22) Chamakura, R.P. "Tryptamines," Microgram 1994, 27, 316.

(23) Vohlken, B.A. "Bufotenine and Psilocin, Mass Spectral Distinctions," Microgram 1993, 26, 233.

(24) Chamakura, R.P. "Bufotenine," Microgram 1993, 26, 185.

Cocaine:

(25) Krueger, S.T. "New Application for the Quantitation of Cocaine Base by Gas Chromatography," J. Forensic Sci. 1994, 39, 177.

(26) Trenerry, V.C.; Robertson, J.; Wells, R.J. "The Determination of Cocaine and Related Substances by Micellar Electrokinetic Capillary Chromatography," Electrophoresis 1994, 15, 103.

(27) Nishikawa, M.; Nakajima, K.; Tatsuno, M.; Kasuya, F.; Igarashi, K.; Fukui, M.; Tsuchihashi, H. "The Analysis of Cocaine and its Metabolites by Liquid Chromatography/Atmospheric Pressure Chemical Ionization-Mass Spectrometry (LC/APCI-MS)," Forensic Sci. Intnl. 1994, 66, 149.

(28) Glass, R.L.; Johnson, E.L. "Comparison of High Performance Liquid Chromatographic Analyses of Cocaine in Coca Leaves," J. Liq. Chromatogr. 1993, 16, 3543.

(29) MacGregor, R.R.; Fowler, J.S; Wolf, A.P. "Determination of the Enantiomeric Composition of Samples of Cocaine by Normal-Phase High Performance Liquid Chromatography with UV Detection," J. Chromatogr. 1992, 590, 354.

Dimethpramide:

(30) Dawson, B.A.; Black, D.B. et al. "Structural Elucidation of Unusual Police Exhibits. I. Dimethpramide (Dimetcarb)," Forensic Sci. Intnl. 1995, 71, 169.

(31) Latham, D.; Callahan, S. et al. "Identification of an Unusual Police Exhibit as Dimethpramide," Microgram 1994, 27, 227.

Dimethylaminorex:

(32) Noggle, F.T.; Clark, C.R.; DeRuiter, J. "Liquid Chromatographic and Spectral Analysis of the Stereoisomers of Dimethylaminorex," JAOAC Intnl. 1992, 75, 423.

Fenethylline:

(33) Al-Gharably, N.; Al-Obaid, A.R. "The Characterization of Counterfeit Captagon Tablets," J. Forensic Sci. Soc. 1994, 34, 165.

Fentanyls:

(34) Sorokin, V.I.; Semkin, E.P.; Savilov, A.P. "Expert Examination of 3-Methylfentanyl," Microgram 1994, 27, 221.

(35) Noggle, F.T.; Andurkar, S.V.; Clark, C.R.; DeRuiter, J. "GC-MS Analysis of Fentanyl Synthesized from 1-Phenethyl-4-piperidone," Microgram 1993, 26, 285.

4-Fluoroamphetamine/4-Fluoromethamphetamine:

(36) Alexander, G.L. "Characterization of Aromatic Fluoro-Analogs of Phenylacetone, Amphetamine and Methamphetamine," Microgram 1994, 27, 268.

Heroin:

(37) Walker, J.A.; Krueger, S.T.; Lurie, I.S.; Marche, H.L.; Newby, N. "Analysis of Heroin Drug Seizures by Micellar Electrokinetic Capillary Chromatography (MECC)," J. Forensic Sci. 1995, 40, 6.

(38) Trenerry, V.C.; Wells, R.J. "The Analysis of Illicit Heroin Seizures by Capillary Zone Electrophoresis," J. Chromatogr. Sci. 1994, 32, 1.

(39) Aizenman, N.; Ravreby, M. "Detecting Heroin in Heroin Citrate Residues," Microgram 1994, 27, 182.

(40) Wijesekera, A.R.L.; Abeysinghe, D.M.U.J.; Pathirana, K.C. "Studies of the Degradation of Heroin," Forensic Sci. Intnl. 1994, 67, 147.

(41) Chow, S.T.; Lee, T.K.; Saw, C.G.; Soon, T.W.; Ng, T.L. "The Homogenization of Illicit Heroin Samples: An Empirical and Statistical Approach," J. Forensic Sci. 1993, 38, 885.

(42) Kalia, A.; Sharm, R.M.; Singh, G.; Kaur, S.; Verma, R.S. "Analysis of Heroin and its Adulterants by Thin Layer Chromatography," Indian J. Forensic Sci. 1992, 6, 95.

gamma-Hydroxybutyric Acid (GHB):

(43) Krawczeniuk, A. "The Occurrence of gamma-Hydroxybutyric Acid (GHB) in a Steroid Seizure," Microgram 1993, 26, 160.

N-(2-Hydroxyethyl)amphetamine:

(44) Carpenter, J.; Hugel, J.; Weaver, K. "The Identification of N-(2-Hydroxyethyl)-amphetamine," J. Can. Soc. Forens. Sci. 1993, 26, 143.

N-Hydroxy-3,4-methylenedioxyamphetamine:

(45) Smith, R.M.; Koch, M.G. "Thermal Decomposition of N-Hydroxy-3,4-methylene-dioxyamphetamine During GC and GC/MS Analysis," Microgram 1995, 28, 199.

Inhalants:

(46) Flanagan, R.J.; Ives, R.J. "Volatile Substance Abuse," Bull. Narcotics 1994, 46, 49.

(47) Kurz, M.E.; Witherspoon, J.R.; Savage, S.; Johns, S. "Analysis of Alkyl Nitrites by Gas Chromatography-Infrared Spectroscopy," J. Forensic Sci. 1992, 37, 1662.

Jimson Weed:

(48) Koverman, G. "Identification of Scopolamine and Hyoscyamine in Jimson Weed (Datura Stramonium)," Microgram 1993, 26, 122.

Khat (Catha Edulis):

(49) Lee, M.M. "The Identification of Cathinone in Khat (Catha Edulis): A Time Study," J. Forensic Sci. 1995, 40, 116.

(50) Dawson, B.A.; Black, D.B.; Lavoie, A.; LeBelle, M.J. "Nuclear Magnetic Resonance Identification of the Phenethylamine Alkaloids of Khat Using a Chiral Solvating Agent," J. Forensic Sci. 1994, 39, 1026.

(51) LeBelle, M.J.; Lauriault, G.; Lavoie, A. "Gas Chromatographic-Mass Spectrometric Identification of Chiral Derivatives of the Alkaloids of Khat," Forensic Sci. Intnl. 1993, 61, 53.

(52) Mathys, K.; Brenneisen, R. "HPLC and TLC Profiles of Phenylalkylamines of Khat (Catha edulis Forsk.) Confiscated in Switzerland," Pharm. Acta Helv. 1993, 5, 53.

(53) Morselli, O.; Bovolenta, A.; Ripani, L.; Garofano, L. "Gas-Chromatography/Mass Spectrometry Determination of the Active Principles of "Catha Edulis" African Vegetable," Microgram 1992, 25, 290.

LSD:

(54) Heagy, J.A.; Moriwaki, W.M.; Chan; K.T. "LSD Extraction From "Blotter" Paper," Microgram 1995, 28, 85.

(55) Kilmer, S.D. "The Isolation and Identification of Lysergic Acid Diethylamide (LSD) from Sugar Cubes and a Liquid Substrate," J. Forensic Sci. 1994, 39, 860.

(56) Ripani, L.; Schiavone, S.; Garofano, L. "GC Quantitative Determination of Illicit LSD," J. Forensic Sci. 1994, 39, 512.

(57) Veress, T. "Study of the Extraction of LSD from Illicit Blotters for HPLC Determination," J. Forensic Sci. 1993, 38, 1105.

(58) Morales, R. "Quantitative Analysis of Lysergic Acid Diethylamide by Gas Liquid

Chromatography," Microgram 1993, 26, 129.

(59) Pulte, L.; Mayberry, J. "Fingerprint Processing of Blotter Paper and Its Effect on LSD Analysis," Microgram 1993, 26, 246.

(60) Neville, G.A.; Beckstead, H.D.; Black, D.B.; Dawson, B.A.; Ethier, J.C. "USP Lysergic Acid Diethylamide Tartrate (Lot 1) Authentic Substance Recharacterized for Authentication of a House Supply of Lysergide (LSD) Tartrate," Can. J. Appl. Spectrosc. 1992, 37, 149.

Marijuana:

(61) Taylor, R.; Lydon, J.; Anderson, J.D. "Anatomy and Viability of Cannabis Sativa Stem Cuttings with and without Adventitious Roots," J. Forensic Sci. 1994, 39, 769.

(62) Veress, T. "Sample Preparation by Supercritical Fluid Extraction for Quantitation. A Model Based on the Diffusion Layer Theory for Determination of Extraction Time," J. Chromatogr., A 1994, 668, 285.

(63) Hauber, D.J. "Marijuana Analysis with Recording of Botanical Features Present and Without the Environmental Pollutants of the Duquenois-Levine Test," J. Forensic Sci. 1992, 37, 1656.

Methylenedioxyamphetamines and Methylenedioxyphenylbutanamines:

(64) Casale, J.F.; Hays, P.A.; Klein, R.F.X. "Synthesis and Characterization of the 2,3-Methylenedioxyamphetamines," J. Forensic Sci. 1995, 40, 391.

(65) Clark, C.R.; DeRuiter, J.; Noggle, Jr., F.T.; Valaer, A. "Identification of 1-(3,4-Methylenedioxyphenyl)-2-butanamines Related to MDMA," Microgram 1995, 28, 154.

(66) Morselli, O.; Bovolenta, A.; Ripani, L.; Garofano, L.; Schiavone, S. ""Designer Drugs" in the Italian Clandestine Market: Situation and New Analytical Problems," Microgram 1994, 27, 28.

(67) Veress, T.; Gal, T.; Nagy, G.; Nagy, J.; Korosi, A. "Analytical Study on Illegally Produced 3,4-Methylenedioxy-N-ethylamphetamine," Microgram 1994, 27, 48.

(68) Rosner, P.; Junge, T. "N-Methyl-1-(3,4-methylenedioxyphenyl)-2-butanamine, a Representative of a New Class of Street Drugs," Microgram 1994, 27, 411.

Methcathinone and Cathinone:

(69) Glennon, R.A.; Young, R.; Martin, B.R.; Dal Cason, T.A. "Methcathinone ("Cat"): An Enantiomeric Potency Comparison," Pharm. Biochem. Behav. 1995, 50, 601.

(70) [Duplicate Citation] LeBelle, M.J.; Savard, C.; Dawson, B.A.; Black, D.B.; Katyal, L.K.; Zrcek, F.; By, A.W. "Chiral Identification and Determination of Ephedrine, Pseudoephedrine, Methamphetamine and Methcathinone by Gas Chromatography and Nuclear Magnetic Resonance," Forensic Sci. Intnl. 1995, 71, 215.

(71) Noggle, F.T.; DeRuiter, J.; Hayes, L.; Clark, C.R. "Stereochemical Analysis of Methcathinone Prepared by Oxidation of Ephedrines and Pseudoephedrines," Microgram 1994, 27, 119.

(72) Noggle, F.T.; DeRuiter, J.; Valaer, A.; Clark, C.R. "GC-MS Analysis of Methcathinone and its Major Decomposition Product," Microgram 1994, 27, 106.

(73) Semkin, E.P.; Sorokin, V.I.; Savenko, V.G. "Examination of Ephedrone," Microgram 1993, 26, 11.

(74) Dal Cason, T.A. "The Identification of Cathinone and Methcathinone," Microgram 1992, 25, 313.

(75) Kalix, P. "Cathinone, a Natural Amphetamine," Pharm. Toxicol. 1992, 70, 77.

Methylmethaqualone:

(76) Angelos, S.A.; Lankin, D.C.; Meyers, J.A.; Raney, J.K. "The Structural Identification of a Methyl Analog of Methaqualone via 2-Dimensional NMR Techniques," J. Forensic Sci. 1993, 38, 455.

N-Methyl-1-phenylethylamine:

(77) Clark, C.C. "The Identification of N-Methyl-1-phenylethylamine," Microgram

1993, 26, 90.

Opium:

(78) Zhanpin, W. "A Simple and Rapid Method for the Extraction of Five Major Alkaloids from Opium," Forensic Sci. Intnl. 1994, 64, 103.

(79) Remberg, B.; Nikiforov, A.; Buchbauer, G. "Fifty Years of Development of Opium Characterization Methods," Bull. Narcotics 1994, 46, 99.

PCP (Phencyclidine) and PCP Analogs:

(80) Haerer, M.; Kovar, K. "Designer-Drugs of the Phencyclidine Series," Pharmazie in Unserer Zeit 1994, 23, 52.

(81) Allen, A.C.; Robles, J.; Dovenski, W.; Calderon, S. "PCP: A Review of Synthetic Methods for Forensic Clandestine Investigation," Forensic Sci. Intnl. 1993, 61, 85.

(82) Lodge, B.A.; Duhaime, R.; Zamecnik, J.; MacMurray, P.; Brousseau, R. "New Street Analogs of Phencyclidine," Forensic Sci. Intnl. 1992, 55, 13.

Steroids:

(83) Dwyer, J.; Chapman, A.E.; Liu, X. "Analysis of Steroids by Combined Chromatography-Infrared Spectroscopy," LC-GC 1995, 13, 240.

(84) Shepherd, III, E.J. "Analysis of Nandralone-p-Hexyloxyphenylpropionate," Microgram 1995, 28, 111.

(85) Lurie, I.S.; Sperling, A.R.; Meyers, R.P. "The Determination of Anabolic Steroids by MECC, Gradient HPLC and Capillary GC," J. Forensic Sci. 1994, 39, 74.

(86) Maroge, W. "Use of Acetone in GC Quantitation of Some Anabolic Steroids in Vegetable Oils," Microgram 1994, 27, 356.

(87) Cairns, T.; Siegmund, E.G.; Rader, B. "Analysis of Testosterone Esters by Tandem Mass Spectrometry," JAOAC Intnl. 1993, 76, 306.

(88) Koverman, G. "Analysis of Anabolic Steroids," Microgram 1993, 26, 248.

(89) Morley, M.; Matkovich, C. "Screening of Steroids by Thin Layer

Chromatography," Microgram 1993, 26, 214.

(90) Clark, C.C. "The GLC Quantitation of Some Anabolic Steroids in Vegetable Oil Preparations," Microgram 1992, 25, 255.

(91) Somsen, G.W.; Gooijer, C.; Brinkman, U.A.T.; Velthorst, N.H.; Visser, T. "Coupling of LC and FT-IR: Impurity Profiling of Testosterone Undecanoate," Appl. Spectrosc. 1992, 46, 1514.

II) Novel Syntheses of Illicit Drugs

Problem/Issue:

Forensic chemists must maintain familiarity with current and potential clandestine syntheses of illicit drugs in order to assist enforcement activities, for enhanced safety and effectiveness during enforcement operations and in order to provide expert testimony in Court proceedings.

Solution:

Clandestine laboratory operations are constantly reviewed to provide a comprehensive overview of the field. In cases where new methodologies are in use, case reports are generated for the forensic and enforcement communities.

Recent Developments:

Enforcement efforts and precursor control/monitoring laws have had a fairly dramatic impact on illicit syntheses of amphetamines in the United States, notably on the hydriodic acid/red phosphorus reduction of ephedrine to methamphetamine. Clandestine laboratory operators have responded with new sources of ephedrine and red phosphorus, clandestine syntheses of hydriodic acid and use of alternate phosphorus/iodine reagents. Use of highly reactive active metal reductions of ephedrine (i.e., with lithium or sodium metals in ammonia) is increasing. Use of mobile labs (i.e., motor homes or tractor-trailers) or hotels/short-term rental properties has complicated enforcement efforts; similarly, leaving operating laboratories unattended has become common practice and has increased hazards associated with clandestine laboratory entry. Increasing numbers of "confined space" clandestine laboratories (e.g., buried vehicles, caves, underground chambers or hidden internal compartments in residences, etc.) has necessitated new guidelines for enforcement and forensic personnel entry and disassembly/cleanup. Use of booby-traps in clandestine laboratories and storage sites is increasing.

Summary:

Since 1992, a variety of new methods for synthesis of amphetamine and methamphetamine and their precursors have been reported (1 - 7).

References:

(1) Andrews, K.M. "Ephedra's Role as a Precursor in the Clandestine Manufacture of Methamphetamine," J. Forensic Sci. 1995, 40, 551.

(2) Hutchinson, K.; Andrews, K.M. "The Use and Availability of Ephedra Products in the United States," Microgram 1995, 28, 256.

(3) Popovich, G.L. "A New Reducing Agent for Ephedrine," Microgram 1995, 28, 79.

(4) Henderson, B.A.; Comparin, J.H. "Clandestine Manufacture of Hydriodic Acid via Iodine, Red Phosphorus and Hydrochloric Acid," Microgram 1994, 27, 382.

(5) Skinner, H.F. "Methamphetamine Synthesis via Reductive Alkylation Hydrogenolysis of Phenyl-2-Propanone with N-Benzylmethylamine," Forensic Sci. Intnl. 1993, 60, 155.

(6) Guiney, L.B. "Synthesis of Amphetamine via Isopropyl Nitrite," Microgram 1992, 25, 295.

(7) Hanel, H.F. "Substitute Bases for Sodium Acetate in the Clandestine Synthesis of Phenyl-2-propanone (P2P)," Microgram 1992, 25, 236.

III) Reference Drug Standards

Problem/Issue:

Reference drug standards are either commercially unavailable or if available are extremely expensive. No reported procedures existed for cleanup and or authentication of reference standards from either synthesized or seized materials.

Solution/Summary:

A systematic procedure for authentication of reference drug standards was developed (1); several reports (2-3) detailed syntheses and/or authentication of specific drug standards.

References:

(1) Hays, P.A. "Authentication Procedures for Reference Drug Standards," Microgram 1994, 27, 14.

(2) Shimamine, M.; Takahashi, K.; Nakahara, Y. "Identification of Psychotropic Substances. IX. Preparation and Various Analytical Data of Reference Standards of New Psychotropic Substances, N-Ethylmethylenedioxyamphetamine, N-Hydroxymethylenedioxyamphetamine, Mecloqualone, 4-Methylaminorex, Phendimetrazine and Phenmetrazine," Eisei Shikensho Hokoku 1993, 111, 66.

(3) [Duplicate Citation] Neville, G.A.; Beckstead, H.D.; Black, D.B.; Dawson, B.A.; Ethier, J.C. "USP Lysergic Acid Diethylamide Tartrate (Lot 1) Authentic Substance Recharacterized for Authentication of a House Supply of Lysergide (LSD) Tartrate," Can. J. Appl. Spectrosc. 1992, 37, 149.

IV) 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 extremely large numbers of exhibits. Improved methods are needed for data handling and analysis.

Solution:

In-depth analysis helps 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, significant advances in comparative analyses were reported for the amphetamines/methylenedioxyamphetamines and cocaine, especially with respect to data handling. Advances in computer speed and capabilities have allowed direct downloading of data from chromatographic systems and rapid analyses. Improved instrumentation, notably capillary electrophoresis, isotope/ratio-mass spectrometry and deuterium nuclear magnetic resonance spectroscopy, offer new methods for more definitive impurity profiling and comparative analysis. New and dramatic improvements in pattern recognition/neural network programs have immensely improved data handling in cocaine comparative analysis and is expected to find similar application for other comparative analysis problems, especially heroin.

Summary:

Since 1991, impurity profiling has been conducted on amphetamines (5-9), cocaine (10-15), heroin (16-18), marijuana (19-22), methamphetamine (23-25), methylene-dioxyamphetamines (26), opium (27), and Source Determination (Ballistics) (28-29).

Comparative Analyses has also been addressed in general terms (1-4).

References:

General Discussions:

(1) Wells, R.J.; Skopec, J.V.; Lewis, T.; Robertson, J. (Australian Government Analytical Laboratories and Australian Federal Police, Pymble and Weston, Australia, and Kingston, Tasmania) "The Profiling of Illicit Drugs Using Data from Anion and Cation Analysis," Personal Communication, 1995.

(2) Perillo, B.A.; Klein, R.F.X.; Franzosa, E.S. "Recent Advances by the U.S. Drug Enforcement Administration in Drug Signature and Comparative Analysis," Forensic Sci. Intnl. 1994, 69, 1.

(3) Huizer, H. "A Contribution to Comparison," Forensic Sci. Intnl. 1994, 69, 17.

(4) Flurer, C.L.; Wolnik, K.A. "Chemical Profiling of Pharmaceuticals by Capillary Electrophoresis in the Determination of Drug Origin," J. Chromatogr., A 1994, 674, 153.

Amphetamines:

(5) Jonson, C.S.L.; Stromberg, L. "Two-Level Classification of Leuckart Amphetamine," Forensic Sci. Intnl. 1994, 69, 31.

(6) Jonson, C.S.L. "Amphetamine Profiling - Improvements in Data Processing," Forensic Sci. Intnl. 1994, 69, 45.

(7) Karkkainen, M.; Sippola, E.; Pikkarainen, A.-L.; Rautio, T.; Himberg, K. "Automated Gas Chromatographic Amphetamine Profiling," Forensic Sci. Intnl. 1994, 69, 55.

(8) King, L.A.; Clarke, K.; Orpet, A.J. "Amphetamine Profiling in the UK," Forensic Sci. Intnl. 1994, 69, 65.

(9) Jonson, C.S.L.; Stromberg, L. "Computer Aided Retrieval of Common-Batch Members in Leuckart Amphetamine Profiling," J. Forensic Sci. 1993, 38, 1472.

Cocaine:

(10) Neumann, H. (Bundeskriminalamt, Wiesbaden, Germany) "Comparison of Cocaine," Personal Communication, 1995.

(11) Hohmann, R.R.; Correa, S.L. (Laboratorio de Criminalistica, Seccion Quimica y Fisica, Policia de Investigaciones de Chile, Santiago, Chile) "Analysis of Traces of Cocaine in Paper," Personal Communication, 1995.

(12) Janzen, K.E.; Fernando, A.R.; Walter, L. "A Database for Comparison of Illicit Cocaine Samples," Forensic Sci. Intnl. 1994, 69, 23.

(13) Moore, J.M.; Cooper, D.A. "The Application of Capillary Gas Chromatography-Electron Capture Detection in the Comparative Analyses of Illicit Cocaine Samples," J. Forensic Sci. 1993, 38, 1286.

(14) Moore, J.M.; Meyers, R.P.; Jimenez, M.D. "The Anatomy of a Cocaine Comparison Case: A Prosecutorial and Chemistry Perspective," J. Forensic Sci. 1993, 38, 1305.

(15) Casale, J.F.; Watterson, J.W. "A Computerized Neural Network Method for Pattern Recognition of Cocaine Signatures," J. Forensic Sci. 1993, 38, 292.

Heroin:

(16) Robertson, J. (Australian Federal Police, Canberra, Australia) "An Evaluation of Multi-Element Analysis in the Profiling of Illicit Drug Seizures," Personal Communication, 1995.

(17) Neumann, H. "Comparison of Heroin by Capillary Gas Chromatography in Germany," Forensic Sci. Intnl. 1994, 69, 7.

(18) See also: Desage, M.; Guilluy, R.; Brazier, J.L.; Chaudron, H.; Girard, J.; Cherpin, H.; J. Jumeau, J. "Gas Chromatography with Mass Spectrometry or Isotope-Ratio Mass Spectrometry in Studying the Geographical Origin of Heroin," Anal. Chim. Acta 1991, 247, 249.

Marijuana:

(19) Robertson, J. (Australian Federal Police, Canberra, Australia); Jagadish, V.;

Gibbs, A. (Australian National University, Canberra, Australia) "RAPD Analysis Detects Genetic Variation of Cannabis Sativa," Personal Communication, 1995.

(20) Court, T.M. (Department of Botany, University of Queensland, St. Lucia, Brisbane, Australia) "Characters in Cannabis Indicative of Provenance," Personal Communication, 1995.

(21) Lehmann, T.; Brenneisen, R. "High Performance Liquid Chromatographic Profiling of Cannabis Products," J. Liq. Chromatogr. 1995, 18, 689.

(22) Neuninger, H.; Saukel, J.; Witzmann, J. "The Tetrahydrocannabinol (THC) Content of Cannabis Plants Cultivated in Austria. Influence of Harvest Time and Weather," Sci. Pharm. 1992, 60, 105.

Methamphetamine:

(23) Inoue, T. (National Research Institute of Police Science, Sanban-cho, Chiyoda-ku, Tokyo, Japan) "Impurity Profiling Analysis of Methamphetamine in Japan," Personal Communication, 1995.

(24) Perkal, M.; Ng, Y.L.; Pearson, J.R. "Impurity Profiling of Methylamphetamine in Australia and the Development of a National Drug Database," Forensic Sci. Intnl. 1994, 69, 77.

(25) Inoue, T.; Tanaka, K.; Ohmori, T.; Togawa, Y.; Seta, S. "Impurity Profiling Analysis of Methamphetamine Seized in Japan," Forensic Sci. Intnl. 1994, 69, 97.

Methylenedioxyamphetamines:

(26) Mas, F.; Beemsterboer, B.; Veltkamp, A.C.; Verweij, A.M.A. "Determination of 'Common Batch' Members in a Set of Confiscated 3,4-(Methylenedioxy)-methylamphetamine Samples by Measuring the Natural Isotope Abundances: A Preliminary Study," Forensic Sci. Intnl. 1995, 71, 225.

Opium:

(27) Remberg, B.; Krenn, L.; Kopp, B.; Buchbauer, G.; Nikiforov, A. "Principal Component Analysis (PCA) of Opium Alkaloid Contents for Origin Determina-

tion," Pharmazie 1994, 49, 766.

Source Determination (Ballistics):

(28) Franzosa, E.S.; Harper, C.W (U.S. Drug Enforcement Administration, Special Testing and Research Laboratory, McLean, VA, USA) "The Logo Index for Tablets and Capsules," 1995, U.S. Department of Justice, Drug Enforcement Administration (Arlington, VA).

(29) Perkal, M. (Victoria Forensic Science Centre, Victoria, Australia) "Illicit Drug Logos," Personal Communication, 1995.

V) Source Determination of Drugs (Impurity Profiling)

Problem/Issue:

Impurity profiling of drugs is important for comparative analysis protocols. 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 impurity profiles. 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:

In contrast to most case reports prior to 1992 (which commonly only reported specific impurities noted in individual seized exhibits), there has been a much more systematic effort to identify impurities and establish signature profiles via in-house syntheses, notably with the amphetamines and methylenedioxyamphetamines. Heroin impurity profiling continues in the United States and Germany, with Australia a recent and fast-growing new research investigator in the field. The United States has made significant advances in cocaine impurity profiling, and several European groups have recently initiated new cocaine signature studies. Impurities in precursor chemicals and occluded trace solvents in finished products (notably cocaine and heroin) were both recognized as being increasingly important in impurity profiling.

Summary:

Since 1992, impurity profiling has been conducted on amphetamine (1-4), cocaine (5-17), heroin (18-20), methamphetamine (21-25), methylenedioxyamphetamines (26-32), occluded solvents in drugs (33-34) and drug precursors (35).

References:

Amphetamine:

(1) Noggle, F.T.; Clark, C.R.; DeRuiter, J. "GC-MS and Liquid Chromatographic Analysis of Amphetamine and Amphetamine-type Products Formed in the Reaction of Arylpropenes with Acetonitrile and Sulfuric Acid," Microgram 1995, 28, 12.

(2) Noggle, F.T.; DeRuiter, J.; Clark, C.R. "GC-MS Analysis of Products, By-Products and Impurities in the Synthesis of Amphetamine from 1-Phenyl-2-Nitropropene," Microgram 1994, 27, 153.

(3) DeRuiter, J.; Clark, C.R.; Noggle, F.T. "Gas Chromatographic and Mass Spectral Analysis of Amphetamine Products Synthesized from 1-Phenyl-2-nitropropene," J. Chromatogr. Sci. 1994, 32, 511.

(4) Forbes, I.J.; Kirkbride, K.P. "The Origin of Alkenes in Illicit Amphetamine: An Examination of the Illicit Synthesis of Phenyl-2-propanone," J. Forensic Sci. 1992, 37, 1311.

Cocaine:

(5) 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-Heptafluoro-butyrylation and Sensitive Detection in South American Coca Leaves Using Capillary Gas Chromatography-Electron Capture Detection," J. Chromatogr., A 1995, 704, 483.

(6) Casale, J.F.; Moore, J.M.; Cooper, D.A. "Novel Chlorinated Tropanes Derived from the Treatment of Cocaine with Sodium Hypochlorite," J. Forensic Sci. 1995, 40, 815.

(7) Kumar, A.; Kiser, W.O. "Identification and Quantitation of Norcocaine in Illicit Cocaine Samples," J. Forensic Sci. 1995, 40, 464.

(8) Casale, J.F.; Moore, J.M. "3',4',5'-Trimethoxy-Substituted Analogs of Cocaine, cis/trans-Cinnamoylcocaine and Tropacocaine: Characterization and

Quantitation of New Alkaloids in Coca Leaf, Coca Paste and Refined Illicit Cocaine," J. Forensic Sci. 1994, 39, 462.

(9) Casale, J.F.; Moore, J.M. "An In-Depth Analysis of Pharmaceutical Cocaine: Cocaethylene and Other Impurities," J. Pharm. Sci. 1994, 83, 1186.

(10) Moore, J.M.; Hays, P.A.; Cooper, D.A.; Casale, J.F.; Lydon, J. "1-Hydroxy-tropacocaine: An Abundant New Alkaloid of Greenhouse Cultivated Erythroxylum Novogranatense var. Novogranatense," Phytochem. 1994, 36, 357.

(11) Carpenter, A.; Laing, R.R. "Cocaine in Bleach: Destroying the Evidence (Identification of Degradation Products)," Microgram 1994, 27, 249.

(12) Casale, J.F.; Moore, J.M. "Detection and Determination of Pseudococaine in Coca Leaves and Illicit Cocaine Samples," J. Forensic Sci. 1994, 39, 1537.

(13) Moore, J.M.; Casale, J.F. "In-Depth Chromatographic Analyses of Illicit Cocaine and its Precursor, Coca Leaves," J. Chromatogr., A 1994, 674, 165.

(14) Moore, J.M.; Casale, J.F.; Klein, R.F.X.; Cooper, D.A.; Lydon, J. "Determination and In-Depth Chromatographic Analyses of Alkaloids in South American and Greenhouse-Cultivated Coca Leaves," J. Chromatogr. 1994, 659, 163.

(15) Casale, J.F. "Methyl Esters of Ecgonine: Injection-Port Produced Artifacts from Cocaine Base (Crack) Exhibits," J. Forensic Sci. 1992, 37, 1295.

(16) Dang, V.B.; Levillain, P.; Galliot, M.; Fabiani, P.; Lich, N.P. "Analysis of Cocaine in Drug Seizures," Ann. Falsif. Expert. Chim. Toxicol. 1992, 85, 149.

(17) Violante, N.; Quaglia, M.G.; Lopez, A.; Caroli, S. "Characterization of Cocaine and Heroin samples as a function of their trace element content: An Analytical Pilot Study," J. Microchem. 1992, 45, 79.

Heroin:

(18) Lurie, I.S.; Chan, K.C.; Spratley, T.K; Casale, J.F.; Issaq, H.J. "Separation and Detection of Acid/Neutral Impurities in Illicit Heroin via Capillary Electrophoresis," J. Chromatogr., A 1995, 669, 3.

(19) Kaa, E. "Impurities, Adulterants and Diluents of Illicit Heroin. Changes During a 12 Year Period," Forensic Sci. Intnl. 1994, 64, 171.

(20) [Duplicate Citation] Violante, N.; Quaglia, M.G.; Lopez, A.; Caroli, S. "Characterization of Cocaine and Heroin samples as a function of their trace element content: An Analytical Pilot Study," J. Microchem. 1992, 45, 79.

Methamphetamine:

(21) Pearson, J.R., et al. "Investigation of Impurities in Methylamphetamine Synthesized from Ephedrine by Reduction with HI and Red Phosphorus," Forensic Sci. Intnl. 1995 (In Press).

(22) Noggle, F.T.; Clark, C.R.; DeRuiter, J. "Evaluation of Allylbenzene as a Precursor for the Synthesis of Methamphetamine," Microgram 1994, 27, 302.

(23) Noggle, F.T.; Clark, C.R.; DeRuiter, J. "Characterization of Methamphetamine and Synthetic By-Products in Clandestine Samples: A Case Report," Microgram 1994, 27, 253.

(24) Tanaka, K.; Ohmori, T.; Inoue, T.; Seta, S. "Impurity Profile Analysis of Illicit Methamphetamine by Capillary Gas Chromatography," J. Forensic Sci. 1994, 39, 500.

(25) Tanaka, K.; Ohmori, T.; Inoue, T.; Seta, S. "Analysis of Impurities in Illicit Methamphetamine," Forensic Sci. Intnl. 1992, 56, 157.

Methylenedioxyamphetamines:

(26) Robak, D.R. "Identification of Clandestinely Produced Methylenedioxyisoquinolines," Microgram 1995, 28, 46.

(27) Clark, C.R.; DeRuiter, J.; Andurkar, S.; Noggle, F.T. "Analysis of 3,4-Methylenedioxyphenyl-2-propanone and 3,4-Methylenedioxyamphetamine Prepared From Isosafrole," J. Chromatogr. Sci. 1994, 32, 393.

(28) Bohn, M.; Bohn, G.; Blaschke, G. "Synthesis Markers in Illegally Manufactured 3,4-Methylenedioxyamphetamine and 3,4-Methylenedioxymethamphetamine," Intnl. J. Legal Med. 1993, 106, 19.

(29) Clark, C.R.; Noggle, F.T. "GC-MS Analysis of Products, Intermediates and By-Products in the Synthesis of MDA from Isosafrole," Microgram 1994, 27, 188.

(30) Verweij, A.M.A.; Sprong, A.G.A. "A Note About Some Impurities in Commercially Available Piperonylmethylketone," Microgram 1993, 26, 209.

(31) Renton, R.J.; Cowie, J.S.; Oon, M.C.H. "A Study of the Precursors, Intermediates and Reaction By-Products in the Synthesis of 3,4-Methylenedioxymethylamphetamine and its Application to Forensic Drug Analysis," Forensic Sci. Intnl. 1993, 60, 189.

(32) Verweij, A.M.A. "Impurities in Illicit Drug Preparations: 3,4-(Methylenedioxy)-amphetamine and 3,4-(Methylenedioxy)methylamphetamine," Forensic Sci. Rev. 1992, 4, 137.

Occluded Solvents:

(33) Morello, D.R.; Meyers, R.P. "Qualitative and Quantitative Determination of Residual Solvents in Illicit Cocaine Hcl and Heroin Hcl," J. Forensic Sci. 1995 (In Press).

(34) Mulligan, K.; McCauley, H. "Factors that Influence the Determination of Residual Solvents in Pharmaceuticals by Automated Static Headspace Sampling Coupled to Capillary GC-MS," J. Chromatogr. Sci. 1995, 33, 49.

Precursors:

(35) Verweij, A.M.A. "Impurities in Illicit Drug Preparations (XXIV). Spectroscopic Properties of Some Compounds Present in Essential Oils Used as Starting Compounds in the Synthesis of "Designer Drugs" of the Phenethylamine Type," Microgram 1995, 28, 224.

VI) 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; 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 constantly monitored to provide a comprehensive overview of adulterants and diluents. Case reports providing standard analytical data for new 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:

Increased computer speed and enhanced search routines enable simultaneous identification of moderate quantities (i.e., 5 - 20 %) of certain cutting agents in cocaine or heroin by FT-IR spectroscopy. The techniques are based on identification of marker peaks for the adulterant or diluent in available "windows" in the infrared spectra of the controlled substance.

Summary:

Since 1992, four new cutting agents were identified and case reports generated (1-4); in addition, several new methodologies for simultaneous identification of common adulterants and diluents via FT-IR techniques were reported (5-6).

References:

Unusual Adulterants/Diluents:

(1) Hays, P.A.; Koles, J.E.; Morello, D.R. (U.S. Drug Enforcement Administration, Special Testing and Research Laboratory, McLean, VA, USA) "Imazalil Sulfate," Personal Communication, 1995.

(2) Knox, M.E.; Fukayama, B.; Haas, J.S.; Alcaraz, A.; Andresen, B.D. "Identification of Diethylaminoethylaniline in a Clandestine Laboratory Reaction Mixture," Microgram 1993, 26, 28.

(3) Carr, S.; Cooper, D. "3-Hydroxy-N-phenyl-2-naphthalene Carboxamide," Microgram 1992, 25, 182.

(4) MacLaren, F.; Cooper, D. "4-(N-Methylacetamido)-antipyrine," Microgram 1992, 25, 164.

Simultaneous Analyses of Drugs and Adulterants/Diluents:

(5) Lopez-Artiguez, M.; Camean, A.; Repetto, M. "Unequivocal Identification of Several Common Adulterants and Diluents in Street Samples of Cocaine by Infrared Spectroscopy," J. Forensic Sci. 1995, 40, 602.

(6) Wielbo, D.; Tebbett, I.R. "The Use of Micro-Fourier Transform Infrared Spectroscopy for the Rapid Identification of Street Drugs: Determination of Interference by Common Diluents," J. Forensic Sci. Soc. 1993, 33, 25.

VII) 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:

Anabolic Steroids and N-hydroxylated amphetamine/methylenedioxyamphetamine compounds (all fairly recent arrivals in the forensic arena) are all quite prone to artifact formation in heated injection ports. Use of certain solvents, notably chloroform, methanol or ethanol, have been recognized to be problematic for analyses of certain amine drugs at higher injection port temperatures.

Summary:

Since 1992, four case reports on artifact appearance in GC and/or GC/MS analyses of various controlled substances were reported (1-4).

References:

(1) Clark, J.D. "Identification of a Heroin/Chloroform-Impurity Reaction Product," Microgram 1994, 27, 385.

(2) Verweij, A.M.A.; Poortman-van der Meer, A.J. "A Note About an Artifact in GC Analysis of Piperonal," Microgram 1992, 25, 160.

(3) Clark, C.R.; DeRuiter, J.; Noggle, F.T. "GC-MS Identification of Amine-Solvent Condensation Products Formed During Analysis of Drugs of Abuse," J. Chromatogr. Sci. 1992, 30, 399.

(4) Clark, C.R.; Noggle, F.T.; DeRuiter, J. "Alcohol-Amine Condensation Products Formed During the GC-MS Analysis of Drugs of Abuse," Microgram 1992, 25, 330.

VIII) 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 routine analysis of drugs. In cases where improved performance is observed, case reports are generated for the forensic community.

Recent Developments:

Capillary electrophoresis has moved to the forefront in liquid chromatographic analyses of controlled substances. In essence, the technique combines the resolution mechanisms of HPLC and electrophoresis, i.e., a flow system under an electric field, and gives (in many cases) dramatically enhanced resolution and speed of analysis. In particular, use of any of a variety of chiral additives to the run buffer results in chiral differentiation of substrates (a common forensic problem) without use of derivatization or expensive columns. Cleanup time between runs is very fast, and total reagent use and waste materials are far less than in HPLC. Approximately a dozen "routine use" instruments are now commercially offered.

Summary:

Since 1992, a variety of new and/or improved/existing instrumental methods have been utilized for drug analysis, including capillary electrophoresis (and related CE techniques) (1-12), gas chromatography (and tandem GC techniques) (13-18), high performance liquid chromatography (and tandem HPLC techniques) (19-31), thin-layer chromatography (37-39) and several other techniques (32-36).

References:

Capillary Electrophoresis (and Related CE Techniques):

(1) Lurie, I.S. "Analysis of Seized Drugs by Capillary Electrophoresis," Anal. Addict. Misused Drugs 1995, 151.

(2) Trenerry, V.C.; Wells, R.J. (Australian Government Analytical Laboratories, Pymble, Australia) "Analysis of Drugs of Abuse by Micellar Electrokinetic Capillary Chromatography," Personal Communication, 1995.

(3) Northrup, D.M.; McCord, B.R.; Butler, J.M. "Forensic Applications of Capillary Electrophoresis," J. Capillary Electrophor. 1994, 1, 158.

(4) Lurie, I.S.; Klein, R.F.X.; Dal Cason, T.; LeBelle, M.; Brenneisen, R.; Weinberger, R. "Chiral Resolution of Cationic Drugs of Forensic Interest by Capillary Electrophoresis with Mixtures of Neutral and Anionic Cyclodextrins," Anal. Chem. 1994, 66, 4019.

(5) Aumatell, A.; Wells, R. "Enantiomeric Differentiation of a Wide Range of Pharmacologically Active Substances by Cyclodextrin-Modified Micellar Electrokinetic Capillary Chromatography Using a Bile Salt," J. Chromatogr., A 1994, 688, 329.

(6) Krough, M.; Brekke, S.; Tonnesen, F.; Rasmussen, K.E. "Analysis of Drug Seizures of Heroin and Amphetamine by Capillary Electrophoresis," J. Chromatogr., A 1994, 674, 235.

(7) [Duplicate Citation] Flurer, C.L.; Wolnik, K.A. "Chemical Profiling of Pharmaceuticals by Capillary Electrophoresis in the Determination of Drug Origin," J. Chromatogr., A 1994, 674, 153.

(8) Nielen, M.W.F. "Chiral Separation of Basic Drugs Using Cyclodextrin-Modified Capillary Zone Electrophoresis," Anal. Chem. 1993, 65, 885.

(9) Peterson, T.E. "Separation of Drug Stereoisomers by Capillary Electrophoresis with Cyclodextrins," J. Chromatogr. 1993, 630, 353.

(10) Altria, K.D. "Quantitative Aspects of the Application of Capillary Electrophoresis to the Analysis of Pharmaceuticals and Drug Related Impurities," J. Chromatogr. 1993, 646, 245.

(11) Quang, C.; Khaledi, M.G. "Improved Chiral Separation of Basic Compounds in

Capillary Electrophoresis Using beta-Cyclodextrin and Tetraalkylammonium Reagents," Anal. Chem. 1993, 65, 3354.

(12) Tomita, M.; Okuyama, T. "Analysis of Drugs by Capillary Electrophoresis: Effect of Methanol in Sample Solution on the Electropherogram," Hochudoku 1993, 11, 140.

Gas Chromatography (and Tandem GC Techniques):

(13) Lillsunde, P.; Korte, T. "Thin-Layer Chromatographic Screening and Gas Chromatographic/Mass Spectrometric Confirmation in the Analysis of Abused Drugs," Anal. Addict. Misused Drugs 1995, 221.

(14) Clark, C.R.; Valaer, A.K.; Noggle, F.T.; DeRuiter, J. "GC-MS Differentiation of Acylated Derivatives of Methamphetamine and Regioisomeric Phenethylamines," Microgram 1995, 28, 118.

(15) Echevarria, P.A. "A Dual Internal Standard Method for Screening by Gas-Liquid Chromatography at a One Percent Level," Microgram 1995, 28, 54.

(16) Hattori, H.; Yamada, T; Suzuki, O. "Gas Chromatography with Surface Ionization Detection in Forensic Analysis," J. Chromatogr., A 1994, 674, 15.

(17) Dent, B.R.; Vannoort, R.W. "GC-MS Analysis of Drugs, with Deuterated Internal Standards - the New Zealand Perspective," Forensic Toxicol., Proc. Int. Meet. Assoc. Forensic Toxicol., 26th 1992, 57.

(18) Terada, M.; Shinozuka, T.; Yasuda, M.; Yanagida, J.; Wakasugi, C. "Simultaneous Determination of Acidic and Neutral Drugs by Wide-Bore Column Gas Chromatography with Nitrogen-Phosphorus Detection," Hochudoku 1992, 10, 142.

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

(19) Lurie, I.S. "Reversed-Phase High-Performance Liquid Chromatography Analysis of Drugs of Forensic Interest," Anal. Addict. Misused Drugs 1995, 51.

(20) Binder, S.R. "High-Performance Liquid Chromatography Using UnModified Silica with Polar Solvents," Anal. Addict. Misused Drugs 1995, 133.

(21) Sato, K.; Kumazawa, T.; Katsumata, Y. "On-Line High-Performance Liquid Chromatography-Fast Atom Bombardment Mass Spectrometry in Forensic Analysis," J. Chromatogr., A 1994, 674, 127.

(22) Golkiewicz, W.; Kuczynski, J.; Markowski, W.; Jusniak, L. "High-Performance Liquid Chromatography of Some Alkaloids on Unmodified Silica Gel with Aqueous-Organic Solvent Mixtures," J. Chromatogr., A 1994, 686, 85.

(23) Hermansson, J.; Grahn, A. "Resolution of Racemic Drugs on a New Chiral Column Based on Silica-Immobilized Cellobiohydrolase. Characterization of the Basic Properties of the Column," J. Chromatogr., A 1994, 687, 45.

(24) Logan, B.K. "Liquid Chromatography with Photodiode Array Spectrophotometric Detection in the Forensic Sciences," Anal. Chim. Acta 1994, 288, 111.

(25) Nakahara, Y. "Recent Progress in HPLC Analysis for Drugs of Abuse," Jpn. J. Toxicol. Environ. Health 1993, 39, 369.

(26) Lurie, I.S.; Cooper, D.A.; Krull, I.S. "High-Performance Liquid Chromatography Using Continuous On-Line Post-Elution Photoirradiation with Subsequent Diode-Array UV or Thermospray Mass Spectrometric Detection," J. Chromatogr. 1993, 629, 143.

(27) Lin, L.A. "Detection of Alkaloids in Foods with a Multi-Detector High-Performance Liquid Chromatographic System," J. Chromatogr. 1993, 632, 69.

(28) Hernandez, A.F.; Pla, A.; Moliz, J.; Gil, F.; Gonzalvo, M.C.; Villanueva, E. "Application of the Combined Use of HPLC/Diode Array Detection and Capillary GC/Nitrogen Phosphorus Detection for the Rapid Analysis of Illicit Heroin and Cocaine Samples," J. Forensic Sci. 1992, 37, 1276.

HPLC Retention Indices:

(29) Hill, D.W.; Kind, A.J. "Reversed-Phase Solvent-Gradient HPLC Retention Indexes of Drugs," Anal. Toxicol. 1994, 18, 233.

(30) Bogusz, M.; Erkens, M. "Reversed-Phase High-Performance Liquid Chromatographic Database of Retention Indices and UV Spectra of Toxicologically Relevant Substances and its Interlaboratory Use," J. Chromatogr., A 1994, 674, 97.

(31) Bogusz, M.; Erkens, M.; Franke, J.P.; De Zeeuw, R.A. "Interlaboratory Applicability of a Retention Index Library of Drugs for Screening by Reversed Phase HPLC in Systematic Toxicological Analysis," J. Liq. Chromatogr. 1993, 16, 1341.

Other General Analytical Methods:

(32) Schlatter, K.; Schneider, R.; Widmer, R.; Steiger, R.; Grummisch, U. (Forensic Science Service of the City Police of Zurich, Zurich, Switzerland) "Rapid Semi-Quantitative Method For Determination of Heroin and Cocaine Using NIR Spectroscopy," Personal Communication, 1995.

(33) Kalasinsky, K.S.; Levine, B.; Smith, M.L.; Platoff, Jr., G.E. "Comparison of Infrared and Mass Spectroscopies for Drug Analysis," Crit. Rev. Anal. Chem. 1993, 23, 441.

(34) Hueske, E.E.; Weaver, T.W. "The Application of Videomicroscopy to the Analysis of Controlled Substances," Microgram 1992, 25, 189.

(35) Wielbo, D.; Tebbett, I.R. "The Use of Microcrystal Tests in Conjunction with Fourier Transform Infrared Spectroscopy for the Rapid Identification of Street Drugs," J. Forensic Sci. 1992, 37, 1134.

(36) Japp, M.; Vinall, M.; Osselton, M.D. "Assessment of a Programmable Temperature Vaporizing (PTV) Injector and Cold On-Column Injector for Analysis of Drugs in Forensic Samples," Forensic Toxicol., Proc. Int. Meet. Assoc. Forensic Toxicol., 26th 1992, 222.

Thin-Layer Chromatography:

(37) Jeger, A.N.; Briellmann, T.A. "Quantitative Applications of TLC in Forensic Science," J. Planar Chromatogr. - Mod. TLC 1994, 7, 157.

(38) Pothier, J.; Galand, N.; Viel, C. "Determination of Some Narcotic and Toxic Alkaloidal Compounds by Overpressured Thin-Layer Chromatography with Ethyl Acetate as Eluant," J. Chromatogr. 1993, 634, 356.

(39) Tandon, R. "Separation of Cocaine, Pramocaine, Fentanyl and Diphenhydramine; Some Common Drugs by Thin Layer Chromatography," Microgram 1992, 25, 168.

IX) Robotics and Computer Programs

Problem/Issue:

Repetitive multi-step analyses and/or data entry is tedious, time wasting and can lead to errors due to fatigue and/or boredom.

Solution:

Repetitive, multi-step analyses have been automated via use of robotics systems. Repetitive data entry/analyses have been improved via development of enhanced computer programs, macros, search routines, etc. In cases where improved performance is observed, case reports are generated for the forensic community.

Recent Developments:

Auto-injectors are now routinely available for virtually all high-quality GC and GC/MS systems, with software already included in the operating systems. Overall capabilities of robotics systems have been greatly enhanced with improved computer support, better "tracking" hardware and software, and new sample-handling protocols (including solid-phase extraction). At least a half-dozen new companies have entered the robotics field.

Summary:

Since 1992, several robotics procedures (1-2) and computer programs (3-7) have been reported.

References:

(1) Pakulniewicz, K.J.; Town, J. "A Robotic Procedure for the Capillary Gas Chromatographic Quantitation of Heroin," LC-GC 1995, 13, 116.

(2) de Kanel, J.; Korbar, T. "Robotics and the Analysis of Drugs of Abuse," Anal. Addict. Misused Drugs 1995, 267.

(3) Grogg-Sulser, K.; Helmlin, H.; Clerc, J. "Qualitative and Quantitative Determination of Illicit Heroin Street Samples by Reversed-Phase High-Performance Liquid Chromatography: Method Development by CARTAGO-S," J. Chromatogr., A 1995, 692, 121.

(4) Bravenec, V.A. "A Post Run Macro for the Evaluation of an HPLC Column's Performance and Reproducibility," Microgram 1995, 28, 220.

(5) Clark, C.C. "A Basic Program for the Quantitation of Cocaine, Heroin, Diazepam, Methaqualone, Codeine and Oxycodone on the HP 5890A Gas Chromatograph," Microgram 1994, 27, 70.

(6) Wrisley, L. "Use of Computer Simulations in the Development of Gradient and Isocratic High-Performance Liquid Chromatography Methods for Analysis of Drug Compounds and Synthetic Intermediates," J. Chromatogr. 1993, 628, 191.

(7) Davidson, A.W. "Computerized Infrared Search System," Microgram 1992, 25, 298.

X) Sampling Plans

Problem/Issue:

Large drug shipments are almost invariably comprised of multiple units of a standard container size, e.g., several thousand 1 kilogram packages of cocaine. Current U.S. sentencing guidelines require accurate assessment of the makeup of an entire shipment; however, comprehensive analyses of such shipments is a daunting and prodigiously labor intensive task.

Solution/Summary:

Representative sampling plans which permit statistical inferences to be drawn with a pre-established, high degree of confidence were developed.

Recent Developments:

Representative sampling plans have survived numerous Courtroom challenges in the United States.

Summary:

Since 1991, several representative sampling plans have been reported (1-3).

References:

(1) Colon, M.; Rodriguez, G.; Orlando-Diaz, R. "Representative Sampling of 'Street' Drug Exhibits," J. Forensic Sci. 1993, 38, 641.

(2) Tzidony, D.; Ravreby, M. "A Statistical Approach to Drug Sampling: A Case Study," J. Forensic Sci. 1992, 37, 1541.

(3) See also: Frank, R.S.; Hinkley, S.W.; Hoffman, C.G. "Representative Sampling of Drug Seizures in Multiple Containers," J. Forensic Sci. 1991, 36, 350.

XI) Vapor and Particle Detection (Portable 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 analytical equipment.

Solution:

Development of portable and highly sensitive vapor and/or particle detectors for drug analyses allows 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 single ion monitoring instruments (such as the Barringer IONSCAN) have 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 or GC/MS-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 1991, a variety of new, portable vapor and/or particle detectors have been reported for drug analyses (1-9).

References:

(1) Sobotka, A.J. (U.S. Drug Enforcement Administration, South Central Laboratory, Dallas, TX, USA) "MAX-2; Field Testing of the Barringer IONSCAN," Personal Communication, 1995.

(2) Hilpert, R.; Binder, F., et al. "Biosensor Technology for the Detection of Illegal Drugs (I). Objectives, Preparatory Work and Drug Enrichment," Proc. SPIE-Int. Soc. Opt. Eng. 1994, 2276, 120.

(3) Wendel, G.J.; Jadamec, J.R.; Su, C.-W. "Recent Developments in the SENTOR Drug Detection System," Proc. SPIE-Int. Soc. Opt. Eng. 1994, 2276, 98.

(4) Holland, P.M.; Mustacich, R.V. et al. "Correlated Column Micro Gas Chromatography Instrumentation for the Detection of Contraband Drugs in Cargo Containers," Proc. SPIE-Int. Soc. Opt. Eng. 1994, 2276, 79.

(5) Fetterolf, D.D; Donnelly, B.; Lasswell, L.D. "Portable Instrumentation: New Weapons in the War Against Drugs and Terrorism," Proc. SPIE-Int. Soc. Opt. Eng. 1994, 2092, 40.

(6) Brown, D.R.; Gozani, T. et al. "Application of Pulsed Fast Neutrons Analysis to Cargo Inspection," Nucl. Instrum. Methods Phys. Res., Sect. A 1994, 353, 684.

(7) Fytche, L.M.; Hupe, M.; Kovar, J.B.; Pilon, P. "Ion Mobility Spectrometry of Drugs of Abuse in Customs Scenarios: Concentration and Temperature Study," J. Forensic Sci. 1992, 37, 1550.

(8) Watson, G.; Horton, W.; Staples, E. "Portable Detection System for Illicit Materials based on SAW Resonators," Proc. - IEEE Ultrason. Symp. 1992, 1, 269.

(9) Elias, L.; Lawrence, A.H. "On-Site Sampling and Detection of Drug Particulates," Anal. Drugs Abuse 1991, 373.

XII) Miscellaneous

References (10):

(1) O'Gorman, M. ((U.S. Drug Enforcement Administration, Northeast Laboratory, New York, NY, USA) "Unique Drug Smuggling Techniques and Problems Associated with Analyses," Personal Communication, 1995.

(2) Arap Sogomo, W.K. (Government Chemist's Department, Nairobi, Kenya) "The Narcotic and Psychotropic Drugs Situation in the Republic of Kenya," Personal Communication, 1995.

(3) Bobev, K. (Research Institute of Forensic Science and Criminology, Sofia, Bulgaria) "A Survey of Drug Seizures in Bulgaria, 1990 - 1993," Personal Communication, 1995.

(4) de Bruin, P.R.D.B. (Forensic Science Laboratory, South African Police Service, Pretoria, South Africa) "A Survey of Recent Drug Seizures in South Africa," Personal Communication, 1995.

(5) White, M.J. "Safety Training for Clandestine Laboratory Investigators," Australian Criminal Intelligence Digest 1994, 44.

(6) Pakulniewicz, K.J.; Matkovich, C.E. "Intralaboratory Precision of Cocaine Analysis by Capillary Gas Chromatography," Microgram 1993, 26, 71.

(7) Joern, W.A. "Ion Ratio Instability of a GC/MS System is Related to Fluctuations in Room Air Flow," J. Anal. Toxicol. 1993, 17, 122.

(8) Casale, J.F.; Klein; R.F.X. "Illicit Production of Cocaine," Forensic Sci. Rev. 1993, 5, 95.

(9) Lomonte, J.N. "Determination of Volumes in Laboratory Vessels," J. Forensic Sci. 1992, 37, 1380.

(10) Heagy, J.A. "Decontamination of Biohazardous Evidence by Autoclaving," Microgram 1992, 25, 238.