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Forensic Science

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It is the aim of this article to present a concise survey of articles appearing in publications that primarily appeal to forensic practitioners. To accomplish this objective, we have focused our attention on the following journals: *Journal of Forensic Sciences, Journal of the Forensic Science Society, Forensic Science International, Journal of the Canadian Society of Forensic Science, Arson Analysis Newsletter, Analytical Toxicology*, and *The Microscope*, as well as *Chemical Abstracts Selects: Forensic Chemistry*. Our survey encompasses the period from October 1982 through October 1984. Because of normal delays in the abstraction of journal articles by Chemical Abstracts, some work covering this period will inadvertently be omitted. Hopefully these references will be included in the next biennial review.

The format selected for this survey divides coverage into three distinct areas: Drugs and Poisons, Forensic Biochemistry, and Trace Evidence. Within the scope of each of the areas, articles have been selected to describe current forensic science practices in analytical chemistry and to outline relevant forensic science research interests. To keep our discussion concise and meaningful, we have limited our survey to drugs regulated under the United States Controlled Substances Act, ethanol, and common poisons. Furthermore, to eliminate unnecessary duplication of effort, citations of articles appearing in *Clinical Chemistry*, the *Journal of Pharmaceutical Sciences*, and other pharmaceutical journals have been avoided. We believe that ample coverage of these journals is provided within the pharmaceuticals and clinical chemistry sections of this journal and recommend that interested readers consult these sections in order to obtain a complete survey of the drug-abuse subject.

DRUGS AND POISONS

Ethanol and Volatiles. The role of the forensic scientist in the application of chemical tests for alcohol in traffic law enforcement has been reviewed (*1*). The stability of ethyl alcohol in forensic blood specimens has been tested by headspace gas chromatography (*2*). The effects of short-term storage conditions (i.e., time, temperature, and NaF preservative) on ethanol concentrations in blood from living human subjects has been investigated using gas chromatography (*3*). A procedure incorporating improvements in the recovery and stability of ethanol in automated headspace analysis has been described (*4*). The relation of ethanol and acetaldehyde conversion in whole blood to blood age (in vitro) has been studied (*5*). Tests for the accuracy of back-calculated blood ethanol values according to an evaluation of 198 blood samples showed that coefficients currently used for back-calculations of blood ethanol values in the German Democratic Republic, as well as proposed new coefficients, give unreliable results (*6*). Inter- and intraindividual differences of alcohol distribution in humans have been studied by experimental determination of Widmark's factor r by intravenous alcohol administration (*7*). The rate and kinetic order of ethanol elimination was evaluated in human volunteers by breathalyzer tests and headspace gas–liquid chromatography (GLC). The results indicate that the rate of ethanol elimination increases with drinking experience (*8*). Elimination of ethanols in humans has been reviewed by Holsbecker and Wells (*9*). Liquid/air partition coefficients were determined for dilute solutions of ethanol in water, whole blood, and plasma at various equilibrium temperatures by GC (*10*). Endogeneous ethanol in blood and breath has been determined by gas chromatography–mass spectrometry (GC/MS) (*11*). An experimental study on the ratio of breath and blood ethanol showed the respiratory ethanol concentration tended to be higher in the absorption phase and lower in the elimination phase (*12*). The role of rebreathing in the determination of the blood–breath ratio of expired ethanol has been studied (*13*). Effects of temperature and humidity of inhaled air on the concentration of ethanol in a man's exhaled breath have been investigated (*14*). Collections of expired air and chemical determinations of ethanol concentrations in inspired and expired air by mass spectrometry showed, that during prolonged inspiration of ethanol vapor containing air, about 55% was absorbed by adult human subjects (*15*). A study of purge readings and the reproducibility and accuracy for repeated sampling of standard vapors with the Breathalyzer 900 and 900A was reported (*16*). The effects of nonethanolic vapors on readings of blood ethanol concentrations taken by an A.L.E.R.T. roadside screening device with a J-cal calibration accessory showed substantial readings can be produced only at volatile concentrations which are expected to be toxic to humans (*17*). The response to breath-alcohol analyzers to acetone has been studied. Except for one device employing solid-state sensing, acetone is not considered to be a significant problem in breath-alcohol analysis for traffic law enforcement purposes (*18, 19*). Radio frequency interference (RFI) from available frequencies in the 150–170 MHz band has been identified as affecting the analysis of vapor samples using the Alco-Analyzer GC. Various effects on the recordings of the ethyl alcohol concentration curve have been discussed and demonstrated for identification of the rf-induced changes (*20*).

The unreliability of using a urine ethanol concentration to predict a blood ethanol concentration has been shown (*21*). Nomagrams based on the Widmark equation for estimating blood or urine alcohol concentrations from alcohol consumed have been presented (*22*). Ethanol levels in postmortem body fluids (*23, 24*) as well as ethanol levels in the brain from postmortem samples (*25*) have been determined. A study was

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conducted to determine how long and at what concentrations alcohol can remain in the cornea and ocular humours of the eyes from intoxicated donors after their storage by the regular eye bank procedures (*26*). Comparative studies of the ratio of ethyl alcohol in blood to vitrous humour have been reported (*27, 28*), also the ratios of ethyl alcohol of blood to vitrous humour and to urine (*29, 30*) have been reported. The effects of prolonged immersion in water on the ethanol concentration of vitrous humour has also been studied (*31*). Blood, bone marrow, and eye fluid ethanol concentrations in putrefied rabbits have been studied to investigate the feasibility of estimating the blood alcohol level at the time of death from bone marrow or eye fluid ethanol levels obtained from a putrefied body (*32*). A direct injection GC technique has been employed to determine the ethanol concentration in postmortem blood and bile specimens to determine the influence of physical properties and lipid content of bile on the human blood-bile ethanol ratio (*33*).

A discussion of what levels of blood alcohol are lethal has been reported which include a case history of a fatal blood alcohol level in an acute alcohol poisoning (*34*). A study on the effects of low doses of alcohol on driving performance has been reported which, the authors believe, raises questions about the validity of 80 mg/100 mL being a legal limit for driving (*35*). The use of marijuana, ethanol, and other drugs among drivers killed in single-vehicle crashes has been studied (*36, 37*). A comprehensive and systematic screening procedure, for the analysis of samples collected from injured and fatally injured drivers which uses radioimmunoassay (RIA), GC, and HPLC has been recently described (*38*). The extent and nature of the combined use of drugs and alcohol in drivers who have undergone a blood test on suspicion of driving under the influence of alcohol in The Netherlands have been reported (*39*). The effects of ethanol in fatal carbon monoxide poisonings has been ascertained by a survey of over 200 fatalities involving carbon monoxide (*40*). The forensic and biochemical implications of endogeneous 2-propanol have been discussed (*41*). Other studies have also been reported on alcohol cogeners in blood (*42, 43*) and blood and urine (*44*). Headspace GC has been used to detect solvent abuse deaths (*45, 46*), and a headspace method has also been used to identify and quantify acetone and toluene in the blood of a suspected impaired drive (*47*). The role of the laboratory in the investigation of solvent abuse as well as the GC and MS techniques used to detect abused solvents in the breath has been discussed (*48*).

Morphine and Related Narcotics. A review of laboratory methods for the analysis of opiates and diluents in illicit drug traffic has been reported (*49*). Analysis of heroin samples has been performed by capillary GC (*50–52*) and HPLC (*53*). Analytical studies in illicit heroin for the identification of O^3-monoacetylmorphine has been performed using a thin-layer chromatographic (TLC) and HPLC method (*54*), as well as capillary GC and HPLC procedures (*55*). The characterization and comparison of illicit heroin have been performed by HPLC (*56*), capillary GC (*57, 58*), and a combination of GC and HPLC (*59–61*). A dual column HPLC method has been used to quantify the opiate and sugar content of illicit heroin preparations (*62*). Manufacturing impurities in illicit heroin after derivatization with heptafluorobutyric anhydride have been determined at picogram levels by using fused silica capillary column GC in the splitless mode with an electron capture detector (*63, 64*). The specific identification of benzoyltropeine, an isomer of tropacocaine and a unusual substance to find in street heroin samples, was obtained by NMR and GC/MS (*65*). The synthesis and characterization of a novel impurity, $\Delta^{16,17}$-dehydroheroinium chloride, detected in illicit heroin have been described (*66*). The application of zero-order and second derivative ultraviolet spectrometry (UV) for the analysis of mixtures of heroin and morphine has been described (*67*). A rapid-scanning multichannel detector for HPLC has been exemplified by the detection of a model system of diacetylmorphine and its principal metabolites and degration products (*68*). A rapid procedure for the separation and quantification of major alkaloids of opium using isothermal GC has been described (*69*).

Rapid analysis of opium alkaloids has also been performed by ion-pair HPLC (*70*). A simple HPLC method for the determination of morphine in poppy straw has also been reported (*71*). A model system of noscapine and papaverine was used to measure a novel technique for peak recognition and deconvolution by computer-aided photodiode array detection in HPLC (*72*).

A simple and rapid method for the detection of heroin and its metabolites in blood and urine has been described. It involves hydrolysis with two-dimensional TLC analysis using ferric chloride/ferricyanide visualization (*73*). Analysis and identification of heroin and related opiates in biological fluids has been accomplished by GLC (*74, 75*), HPLC (*76–81*), GC/MS (*82*), thin-layer immunoassay (*83*), and a combined enzyme immunoassay–LCEC method (*84*). Pholcodeine, a common component in cough medicines, has been reported as a possible interfering compound in the immunoassay for opiates in urine (*85*).

Cannabinoids. Eight sensitive and specific spot tests for the identification of cannabis materials have been described (*86*). A simple and sensitive assay for the cannabinoids has been presented using a dansylation derivatization with 4-(dimethylamino)azobenzene-4′-sulfonyl chloride (*87*). The synthetic route used and the identification of the precursors and reaction products in a clandestine laboratory manufacture of tetrahydrocannabinol have been presented (*88*). The refined procedures developed for the taking and processing of swabbings from hands to detect trace marijuana by GC/MS has been presented (*89*). TLC methods for the separation and identification of cannabinoids in plant material have been presented (*90, 91*). The potency of confiscated marijuana, hashish, and hash oil over a 10-year period has been discussed by ElSohly et al. (*92*). GC methods have been used to determine the cannabinoid levels in cannabis products (*93, 94*). High-resolution GC (HRGC), packed column GLC, and HPLC were evaluated for the analysis of cannabis constituents. HRGC gave better results than GLC with packed columns and HPLC revealed the presence of cannabinoid acids in fresh cut influorescences of cannabis plants (*95*). A tentative identification of components in the essential oil of *Cannabis sativa L.* by a combination of GC, negative ion chemical ionization MS (NICIMS), and retention indices has been reported (*96*). A comparative study by capillary GC/MS of the composition of the basic fraction of marijuana and tobacco condensates has been performed (*97*). Different batches of cannabis resin from Lebanon have been differentiated by comparing the principal cannabinoid contents by HPLC (*98*). Samples of cannabis were tested by HPLC for indicating a common origin (*99*). GLC and HPLC analyses on the effects of leaf treatment as well as the conditions for cannabinoid extraction were examined in two clones of *Cannabis sativa* (*100*). The physical and chemical features of some cannabis plants have been examined. TLC, GLC, and HPLC were used in the chemical analysis (*101*). Successive generation studies on cannabis have been performed by analyzing the TLC cannabinoid profiles (*102*). A histofluorescent procedure for identifying marijuana cannabinoids has been described which uses a solution of choral hydrate and propylene glycol (*103*).

The stability of Δ^9-THC and two of its metabolites has been determined in blood and plasma (*104*). A pilot study has been conducted to ascertain the range of induced hemolyzed blood/serum Δ^9-THC concentrations in humans. This pilot study may lay the groundwork for a program designed to determine the epidemology and behavior correlates of marijuana used in motorists (*105*). The methods for quantification of Δ^1-THC in tissues and biological fluids have been reviewed (*106*). The detection, identification, and/or confirmation of cannabinoids and their metabolites has been reported using TLC (*107*), TLC and fluorometry (*108*), TLC and MS (*109*), HPLC (*110, 111*), GC-ECD (*112*), HPLC and GC-ECD (*113*), GC/MS (*114–117*), GC and negative CIMS (*118, 119*), RIA (*120, 121*), and RIA and HPLC (*122–124*), and RIA and GC/MS (*125, 126*). Confirmation of EMIT cannabinoid assay results by bonded phase adsorption with TLC has also been reported (*127*). An integrated multimethod approach for urine cannabinoid analysis has been presented which used EMIT, HPLC, and high efficiency TLC (HETLC) (*128*). An evaluation of immunoassay for cannabinoids in urine has been reported (*129*). Apparent half-life of excretion of cannabinoids in man has been reported (*130*). Determination of urinary cannabinoid metabolites following incidental exposure to marijuana smoke has been reported (*131*), as well as studies on the passive inhalation of marijuana smoke by analysis of excreted urinary cannabinoids by EMIT (*132*), and by the

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analysis of Δ^9-THC with GC/MS (*133*). The detection of Δ^1-THC in the breath of human subjects by GC/MS has also been reported (*134*).

Cocaine. A simple, rapid, and highly specific presumptive identification test for cocaine, in either its salt or free base forms, has been described which is based on an ion-pair extraction into an organic phase and is virtually unaffected by diluents, fillers, or excipients and which gives a fair estimation of the purity of cocaine by color comparison (*135*). Use of color reactions for benzyl alcohol when screening cocaine and other benzoyl compounds following their reduction with lithium aluminum hydride has been described (*136*). Samples containing cocaine have been analyzed by HPLC (*137*) and MS (*138*). Synthetic cocaine impurities have been detected in clandestine synthetic cocaine samples by GLC and spectroscopic analysis (*139*). Sampling errors in the determination of cocaine in seized drugs have been discussed (*140*). The study of the stability of cocaine and benzoylecognine in blood samples has been performed (*141*). Cocaine and its metabolites have been detected in biological fluids by UV spectrophotometry (*142*), GC/MS (*143–146*), and GC/MS, EMIT, and HPLC (*147*).

Amphetamines. The microcrystalline properties of the diliturate (5-nitrobarbituric acid) derivative of certain stimulant drugs found in illicit street preparations have been determined (*148*). An excellent article on the differentiation of side chain positional isomers of amphetamines has been reported. This article reported that 11 side chain positional isomers of amphetamines can be distinguished using a combination of color tests, TLC, and MS (*149*). The differentiation of 2,3-methylenedioxyamphetamine from 3,4-methylenedioxyamphetamine has been accomplished using a sulfuric acid color test, GC, IR, MS, and ^{13}C NMR (*150*). The specificity of GLC, UV, IR, ^{1}H NMR, and MS for the identification of methoxy-*N*-methylamphetamines which were synthesized and characterized by these methods has been discussed (*151*). Toxicology data of interest to the forensic chemist for 4-bromo-2,5-dimethoxyamphetamine have been given in a recent report along with its chemical properties and various methods of quantitative and qualitative analysis including HPLC, MS, and IR (*152*). Simple microcrystalline and spectrodensitometry were used to confirm results of spectrophotometric tests of counterfeit Ionamin capsules (*153*). A modification of a clandestine synthesis of phenyl-2-propanone from phenylpropenes has been reported (*154*). Analyses of Leukart-specific impurities in amphetamine and methamphetamine have been performed by LC-MS (*155*) and GC-MS (*156*).

A rapid screening for methamphetamine in urine by color reaction in a Sep-PAK C_{18} cartridge has been developed (*157*). A headspace GC method for the determination of methamphetamine and amphetamine in 1 mL of urine has been described (*158*). Capillary column GC has been used to determine the chemical signatures of different batches of methamphetamine (*159*). HPLC has also been used in the analysis of amphetamine impurities (*160*) as well as for amphetamine determinations as derivatives (*161, 162*). *N*-Mono(trifluoroacetyl) derivatives of amphetamines have been analyzed by GC and MS (*163*). Confirmation of amphetamines as pentafluorobenzoyl derivatives with extractive benzoylation and GC-ECD has been described (*164*). A CI–mass fragmentographic method for the analysis of methamphetamine and amphetamine in human autopsy tissues has been reported (*165*). A RIA has developed for the detection of amphetamine and its analogues in blood and urine without any pretreatment of the samples (*166*). Second derivative UV spectrophotometry has been used to distinguish between amphetamine and some other phenethylamines (*167*). A method has been described for the separation and determination of methamphetamine, amphetamine, and some other drugs using overpressured TLC (*168*). Separations of amphetamine enantioners have been accomplished using HPLC (*169*) and capillary GC/MS (*170*).

Barbiturates. 5,5-Disubstituted barbiturates have been identified with the aid of a field color test (*171*). Analysis of barbiturates in biological fluids has been performed using GC (*172, 173*), HPLC (*174, 175*), and RIA (*176*). An evaluation of a rotating disk multiwavelength UV HPLC detector for the identification of barbiturates and other drugs has been performed (*177*). A field effect transistor sensitive to the phenobarbital anion has been described (*178*). The stability of some barbiturates in blood and liver has been studied using GC and HPLC (*179*). Blood concentration ranges for barbiturates have been discussed to understand the significance of toxicology analyses (*180, 181*) and their interaction with alcohol (*182*).

Miscellaneous Drugs and Poisons. A comparison of methods of analysis for phencyclidine was undertaken utilizing GC, EMIT, TLC, and GC/MS (*183*). A review on the analysis of phencyclidine and its metabolites in biological material has been published (*184*). As part of a search for phencyclidine antagonists, a series of 11 aryl-substituted PCP analogues were prepared and characterized by TLC, GC, MS, NMR, UV-Vis, and IR (*185*). A sensitive capillary GC-NPD procedure has been developed for the analysis of phencyclidine (*186*). Phencyclidine and nine of its analogues have been separated and identified using TLC, HPLC, GC/MS, and IR (*187*).

Methods of the analysis for benzodiazepines in dosage forms and biological fluids have included HPLC (*188–193*), GLC (*194*), RIA (*195*), and a combination of TLC and HPLC (*196*). The decomposition of benzodiazepines during their analysis by capillary GC/MS has been discussed (*197*). Clorazepate presents several problems in identification. In addition to rapid acid decarboxylation to *N*-desmethyldiazepam, a noncontrolled substance, extracts of the pharmaceutical forms of clorazepate contain substances that interfere with isolation of intact and unaltered clorzepate. These substances have been identified by an IR method (*198*). The postmortem stability of benzodiazepines in blood and tissues have been studied (*199*).

A homogeneous polarization fluoroimmunoassay has been described for LSD in serum or urine using a highly fluorescent conjugate of lysergic acid and fluoresceinamine (*200*). LSD has also been detected in human biological fluids by HPLC followed by fluorescence detection (*201*). Capillary GC has been used to separate LSD and LAMPA (*202, 203*). A low-cost

differential fluorometer for the detection and determination of LSD in illicit preparations under field conditions has been described (*204*). A procedure for the analysis of lysergide, applicable to the illicit material dispersed on papersheets and in tablets, has been described using TLC and HPLC (*205*). Methods of examining dried fragments of hallucinogenic fungi have been given with a key to the preliminary identification of suspected hallucinogenic mushrooms (*206*). HPLC continues to be the method of choice for the screening and quantification of psilocybin in hallucinogenic mushrooms. Several methods have recently been reported which use HPLC for this purpose (*207–209*).

The potential of tandem MS utilizing collisionally activated dissociation for molecular structure determination has been illustrated with α-methylfentanyl ("China White"), whose complex structure required several methods for its original elucidation (*210*). Reversed-phase HPLC has been used to separate fentanyl homologues and analogues (*211*). Two simple RIA's have been described for methadone in blood and urine (*212*). A method has been described for the determination of promethazine and some of its major metabolites in postmortem specimens by enzymic digestion followed by HPLC of the extract (*213*). Capillary and packed column GC have been used to determine propoxyphene in biological fluids (*214–216*). A new method for the analysis of ritalinic acid, the major metabolite of methylphenidate, in urine has been described which involves solid-phase extraction, derivatization, GC, and TLC (*217*). The EMIT-st (single test) drug detection system was determined to be reliable and simple to perform for the analysis of methaqualone and several of its metabolites (*218*). A method has been described for the determination of chlorpromazine and some of its major metabolites in postmortem specimens by enzymic digestion of the tissues followed by HPLC (*219*). Toxicological and pathological findings in fatalities involving pentazocine and tripenlenamine have been described including procedures such as TLC, GC, UV, and spectrofluorimetry (*220*). Pentazocine has also been detected by GC/MS (*221*, *222*), and HPLC (*223*).

A headspace GC method for the quantitative analysis of cyanide in blood has been developed (*224*). The effect of thiocyanate on the pyridine–pyrazolone method for the spectrophotometric determination of cyanide has been investigated (*225*). A semiquantitative screening method has been evaluated for the detection of heavy metals in blood and urine by emission spectroscopy (*226*).

Arsenic has been determined in biological fluids for forensic purposes by using direct hydride atomic absorption spectrophotometry (AAS) (*227*), furnace atomic absorption (*228*), with use of a Gutzeit apparatus to rule our external contamination (*229*), and using the silver diethyldithiocarbamate colorimetric method (*230*, *231*). Strychnine in biological fluids has been determined by HPLC (*232*), and spectrophotometric methods have been used to determine carboxyhemoglobin (*233*, *234*). A recent report has described three chromatographic techniques (TLC, GC/NPD, and GC/MS) which may be applied to the analysis of succinylcholine in human tissues (*235*). A recent paper reviewed the biochemistry of insulin and experiences in detection and intrepretation of insulin residues in overdose and poisoning cases (*236*). Comments on this work have also been published (*237*). An interesting article has described the series of events which led to the involvement of FDA's elemental analysis research center in the homicide investigation of the Tylenol tampering incident. The use of ICP-AES analysis to trace the source of the KCN powder is well described (*238*). A study of biological samples obtained from victims of the MGM Grand Hotel fire has been reported. The blood and tissue fluid samples were analyzed for carboxyhemoglobin, oxyhemoglobin, methemoglobin, and total hemoglobin. Outgassing studies were done on the tissue samples using GC/MS and heavy metal analysis on inhaled soot was done by X-ray fluorescence (*239*).

General Procedures. A review of the forensic analysis of drugs of abuse has been published recently (*240*). Gough and Baker (*241*) have updated their previous review (1982) of the identification of major drugs of abuse using chromatography by presenting a reference guide to TLC, GC, and HPLC of major drugs of abuse.

The use of high-resolution GC in forensic science has been discussed (*242*) and applications of its use in forensic drug and toxicology analysis have been reported (*243–248*). The isolation of specific solutes from a urine extract prepared for forensic drug scanning has been used to illustrate a discussion on the state-of-the-art in GC instrumental design (*249*). A comprehensive review covering the evaluation of 29 TLC systems for the chromatography of 794 basic, neutral, and acidic drugs has been reported (*250*). Applications of TLC in drug analysis and forensic toxicology have also been reported (*251–253*).

Identification of drugs by principal components analysis of R_f data obtained by TLC in different eluent systems has been reported (*254*). The impact of biological matrix and isolation methods on the detectability and interlaboratory variations of TLC R_f values in systematic toxicological analysis has been discussed (*255*).

Reviews on the use of TLC (*256*) and HPLC (*256*, *257*) in forensic toxicology have been published as well as reviews in the use of bonded-phase columns (*258*), RIA (*259*), and fluorescence techniques (*260*). Applications of the use of HPLC in forensic science have appeared (*261–263*) as well as forensic applications in the use of LC/MS (*264*), MS (*265*) X-ray diffraction (*266*), and Fourier transformed infrared photoacoustic spectroscopy (*267*). A rapid, sensitive tandem MS/MS technique has been described for the screening and confirmation of trace amounts of drugs and metabolites in blood serum (*268*). The general toxicology unknown often presents challenges and interests to toxicologists. A systematic analytical approach to search for drugs and poisons has been presented (*269*). Other screening strategies and extraction systems have been presented for the detection of drugs in biological fluids (*270–272*). A review of current drugs of abuse screening methods available in Canada in 1982 has been published (*273*). A survey of drug screening in the urine specimens of 45 autopsy cases to determine whether urine is a suitable material for the preliminary screening of drugs in autopsy cases has been reported (*274*). The feasibility of detecting drugs in saliva samples obtained from impaired drivers has also been studied (*275*). The toxicological feasibility of determining drug concentrations in postmortem vitreous humour has been confirmed in a recent study (*276*).

Therapeutic and toxic drug concentrations in blood have been reported by various investigators (*277–279*). The mean list length approach has been elucidated and applied to the screening for basic drugs by TLC and GLC (*280*). A proficiency testing outline has been given which can be feasible for a national proficiency testing program in forensic toxicology (*281*). A review of clandestine laboratory seizures during the period of 1978–1981 has been reported which is intended to familiarize forensic chemists with current information on the types of laboratories being seized in the USA and the methods of synthesis being used (*282*).

FORENSIC BIOCHEMISTRY

RIA for the platelet-specific protein β-thromboglobulin (BTG) has been assessed for use as a sensitive marker of human blood (*283*). The identification of human blood with an hybridoma-derived antibody to human immunoglobulin G has been reported (*284*). Species identification of blood and saliva stains has been accomplished by an indirect enzyme-linked immunoassay (ELISA) method using a monoclonal antibody (*285*). A study has been published on the effect of prolonged water immersion on blood-stained cloth of different types for the detection and origin of blood stains (*286*). A field technique for the identification of deer blood has been evaluated (*287*). Crossed immunoelectrophoresis (CIEP) patterns have been examined for the purpose of individualizing bloodstains (*288*). The determination of the sex from bloodstains has been described by several methods (*289–292*). The determination of sex has also been accomplished in human hairs by using cortical cell nuclei chromatin staining (*293*). The forensic identification of human urine has been accomplished by a solid-phase RIA (*294*). A rapid and inexpensive method for the identification of human urine stains has been developed by identifying the presence of urinary specific Tamm-Horsfall glycoprotein (THG) (*295*). A radial gel diffusion method utilizing urease and bromothymol blue has also been developed for urine stain identification (*296*). The preliminary evaluation of a commercially available test kit in the identification of saliva has been reported (*297*). The test strip Rapignost -Amylase (Behring) for the rapid determination of α-amylase in the urine is also suitable for the de-

termination of salivary amylase in stains stored up to 6 weeks at room temperature (*298*).

The identification of choline in human semen has been accomplished by a simple TLC method (*299*) and an enzymic fluorometric method (*300*). A simple qualitative color test for identifying seminal stains based on high levels of zinc in human semen has been described (*301*). The detection of p^{30} by means of an indirect thin-layer immunoassay has been described (*302*). A comparison of p^{30} and acid phosphatase levels in postcoital vaginal swabs from donor and casework studies has been described (*303*). Sodium thymolphthalein monophosphate has shown that it can serve as an effective substitute for presumptive seminal acid phosphatase (SAP) testing (*304*). RIA has been used to detect prostatic acid phosphatase in vaginal swabs (*305*) and has been compared to an enzyme assay for the measurement of human acid phosphatase in cases of sexual assault (*306*). Alternative methods for the identification of SAP by electrophoresis has been described (*307*). The precise conditions for the separation of SAP and vaginal acid phosphatase (VAP) by isoelectric focusing patterns have been described (*308*).

Aberrant group B reactions detected in mixtures of semen and vaginal secretions have been reported (*309*). The feasibility of HLA typing of dried serum (*310*) and dried bloodstains (*311*) has been described. An excellent, simple, and inexpensive capillary tube method for the Lewis typing of red blood cells has been described (*312*). Attempts to determine the Lewis phenotype of dried bloodstains have been described (*313*). The detection of ABO isoagglutinins in saliva can be facilitated by the addition of toluene to saliva specimens (*314*). An unusual Rhesus Haplotype $-D^{-}$ has been reported in Iceland (*315*). An ELISA technique for the identification of Km(3) in dried bloodstains has been successfully demonstrated (*316*). Gm(11) grouping in dried bloodstains has been reported in two instances (*317, 318*). The L-fucose binding lectin in extracts of *U. europoeus* seeds was isolated by affinity chromatography using sepharose - 6B derivatized with fucose (*319*).

Sensabaugh (*320, 321*) has reviewed the utilization of polymorphic enzymes in forensic science. The relative indices of efficiency for selected enzyme methods of bloodstain analysis have been compared. The methods are listed according to their efficiencies with enzyme group I system being the most efficient and enzyme group III system being the least (*322*). Statistics and probabilities in paternity testing have been discussed in the forensic literature and applied in a variety of ways (*323–332*). A new peptidase isoenzyme which may assist in the identification of vaginal debris has been detected (*333*). The lactate dehydrogenase (LDH) isoenzyme patterns given by cellulose acetate membrane electrophoresis have been found suitable to differentiate between stains of menstrual blood and bloodstains that have become mixed or contaminated with vaginal secretors (*334*).

Isoelectric focusing (IEF) has become a valuable technique in the field of forensic serology, and the use of ultrathin gel is making it even more powerful (*335*). IEF has been used to type esterase D (ESD) (*336–338*), group specific component (Gc) (*339–343, 335*), erythrocyte acid phosphatase (ACP_1) (*344*), transferrin (Tf) (*345*), and phosphoglucomutase (PGM) (*346–352, 335*). The value of ultrathin polyacrylamide gels for the simultaneous separation of PGM and EAP by isoelectric focusing of human red cell lysates and bloodstain extracts has been investigated (*353*). The simultaneous electrophoretic analysis of isoenzymes has also been reported for a number of systems, such as EsD and PGM (*354*), hemoglobin (Hb) and glyoxylase I (GLO-I) (*355*), the third component of complement (C3), Tf, Gc, and BF (*356*). Other methods of electrophoretic analysis have been developed for PGM (*357, 358*), Gc (*359–361*), ESD (*362*), leucine aminopeptidase (LAP) (*363*), and C3 (*364*). An alternative overlay reaction mixture for the development of GLO-I isoenzymes on starch–agarose gel multisystem GLO/PGM/ESD plates has been developed (*365*). A method for the detection of fetal hemoglobin in bloodstains by means of thin-layer immunoassay has been described (*366*). A silver staining method has been developed for the detection of polymorphic proteins in minute bloodstains after IEF (*367*). A method to increase the volume of sample applied to IEF gels has been described which makes IEF of extracts from diluted or aged bloodstains more successful (*368*). The identification of trout and salmon blood on the basis of the enzyme superoxide dismutase has been accomplished by IEF (*369, 370*). Enzyme phenotyping of Alaskan bears for wildlife law enforcement has been accomplished by examining LDH-2 and 6-phosphogluconate dehydrogenase (6PGD) by cellulose acetate electrophoresis (*371*).

Results of a population frequency study conducted on 14 polymorphic blood grouping systems in North Carolina white and black populations have been reported (*372*). Population frequency data have also been reported for ESD (*373*), Hp (*374*), phosphoglycolate phosphatase (PGP) (*375*), PGM_1 (*348, 352*), Tf (*345*), and Gc (*343*).

TRACE EVIDENCE

Petroleum Products. The collection of hydrocarbons on charcoal was evaluated as a technique in arson investigation. This technique is more sensitive than headspace analysis. Solvent desorption of the hydrocarbons from the charcoal was found to be as sensitive as thermal desorption (*376*). The sampling of organic vapors from arson residues using different types of adsorbants was studied. Sampling tubes packed with Poropack Q and Tenax GC were evaluated along with Curie point pyrolysis coated with carbon. The detection limits were found to be in the order of 1 μL of accelerant (*377*). The comparison of heated headspace and a purge–trap charcoal elution procedure was made by Kurtz et al. (*378*). Very similar responses for gasoline by the two techniques were found under the studies' experimental conditions. However, the authors concluded that under actual case conditions the charcoal elution's sensitivity can be enhanced. Furthermore, headspace sensitivity is dependent on the volatility of the petroleum residues present in the sample. Klosterman (*379*) found that direct insertion of charcoal adsorption tubes into the sample container allowed for improved extraction of the debris sample and avoided contamination problems. Five sample recovery techniques useful for the detection of petroleum-based residues were compared for the purpose of determining their relative sensitivities. Studied were headspace analysis, solvent extraction, steam distillation, charcoal elution, and sorbent trap/thermal desorption. Charcoal elution was approximately 25 times more efficient than headspace sampling and over 100 times more efficient than solvent extraction (*380*). An inexpensive devise was designed by Webber et al. (*381*) to facilitate the recovery of petroleum residues utilizing a charcoal trap. In preparation for headspace analysis Higgins et al. (*382*) reported that heating fire-scene debris in a microwave oven offers a number of advantages over a conventional convection oven. Additionally, tests showed that the replacement of metal cans with polyester bags for sample storage can result in a high recovery of petroleum residues. Polyethylene containers were demonstrated to be unsuitable packaging for accelerant evidence collection. Polyethylene's permeability to hydrocarbons causes significant accelerant loss (*383*).

Clausen (*384*) reported on the development of an atmospheric sample collection procedure at fire scenes that uses air pumps. It was concluded that fire atmosphere sampling could be of value in establishing the presence of arson in a large percentage of accelerant-initiated fires. The composition of gasoline changes as it is exposed to heat. This change is caused by the evaporation of volatile chemicals. Chromatograms of gasoline at different stages of evaporation were evaluated by Guinther et al. (*385*). Capillary GC/MS has been applied to analyzing arson accelerants. Improved identification of accelerants was accomplished through gas chromatography by selecting ions from major hydrocarbon families (*386*). Common products frequently used as accelerants were readily identified by GC/MS. This approach was further enhanced by using selected ions to generate chracteristic spectral profiles of common accelerants (*387*). Trimpe et al. (*388*) has also demonstrated the applicability of mass chromatography as an aid for the identification of petroleum-based residues. Hydrocarbons present in the headspace sample of carpet material recovered from a fire were identified by GC and GC/MS and were not mistaken from an accelerant (*389*). A fluorometric method named by the authors "variable separation synchronous excitation fluorometry" (VSSE) when augmented by other fluorometric techniques has been used to differentiate a number of commercial motor oils (*390*). Conventional, synchronous, and VSSE fluorometric techniques were also shown to be highly discriminatory for differentiating used automobile engine oils (*391*). Petroleum-based lubricants may be used in cases of rape or forcible sodomy. Sixteen such

commercial products were distinguished by the combination of GC and synchronous fluorescence, and as little as 0.5 mg of product could be identified by these techniques (*392*).

Explosives and Lachrymators. Work was done to develop a procedure to detect ethylene glycol mononitrate and monomethylamine nitrate from post explosion debris. The approach involved a trap and purge procedure for the former and an aqueous extraction for the latter (*393*). Similar quantities of nitroglycerine as those encountered in cardiovascular tablets can be transferred to the hands on contact with explosives or firearms. Hence, any positive result for explosives or propellant trace due to nitroglycerine in a hand swab should be interpreted with allowance for the possession of a tablet (*394*). Factors affecting the persistance of military explosive residues on hands were studied. Low levels of explosives were found after 24 h; however, attempts to detect either RDX or TNT after 48 h were unsuccessful (*395*). A comparison of the relative efficiencies of several methods for extracting common explosives from hand swabs was made. A centrifugal microfilter procedure was recommended (*396*). A microfilter extraction assembly containing a mixture of alumina and octadecylsilylsilica was used in the preparation of small volumes of cleaned-up extracts from hand swabs. Explosive components were screened by HPLC with electrochemical detection at a mercury dropping electrode. Detection limits were on the order of 1–10 ng per swab (*397*). The pendent mercury dropping electrode in combination with HPLC gives detection limits for many organic explosives in the range of 7–49 pg per 20 μL of injected sample (*398*). Nitrocellulose was detected in trace amounts with a size exclusion chromatograph coupled to an electrochemical detector (*399*). HPLC conditions were reported for screening and identifying common explosives, e.g., nitroglycerine, ethylene glycol dinitrate, RDX, and TNT. Minimum detectable limits were found to be in the range of 10 ng (*400*). Also, HPLC utilizing photolysis–electrochemical detection was applied to the detection of explosives of forensic interest (*401*). A thermal energy analyzer interfaced to both a GC or HPLC was shown to be selective for nitro-based explosives at a sensitivity level of 4–5 pg. This approach was applied to the examination of postexplosion debris, hand swabs, and human blood (*402*). An HPLC was linked with a mass spectrometer using a direct liquid introduction interface for the purpose of examining explosives (*403*). A series of explosives were studied by this approach. The resultant chemical ionization spectra obtained included many protonated molecular ions, adduct ions, and typical fragment ions which made positive identification of the compounds possible (*404*). Use of fused silica capillary GC columns in conjunction with a thermal energy detector allowed for the detection of explosives at low picogram levels (*405*).

The major metabolite of TNT was detected in urine by GC analysis. Detectable levels were found in the urine samples of munition workers. However, it was not possible to detect TNT metabolite through a single contact with TNT (*406*). Trioxane was identified as an adulterant in an improvised explosive device. Trioxane was identified by its infrared (IR) spectrum and by proton magnetic resonance spectroscopy (*407*). Paterson et al. (*408*) reported that explosives sensitized with nitrostarch, nitrocellulose, ethylene glycol mononitrate, aluminum, and alkylammonium nitrates are displacing explosives sensitized with nitroglycerine and nitroglycol. The alkylammonium nitrates can be uniquely identified by three TLC systems. Reutter et al. (*409*) have reviewed the analysis of explosives and explosion debris by ion chromatography. Raman spectra were obtained from trace amounts of nitroglycerine and TNT absorbed onto silica gel and charcoal (*410*). Acetone or ethanol swabbing was used to collect traces of explosives from postexplosion debris. The samples were analyzed by HPLC and TLC (*411*).

An HPLC method was developed for determination of capsaicin in oleoresin capsicum and in mineral oil based aerosol formulations intended for personal protection (*412*). Electron impact (EI), positive ion chemical ionization (PICI), and negative ion chemical ionization (NICI) mass spectrometry were studied for their abilities to detect and identify 1-(methylamino)anthraquinone, (*o*-chlorobenzal)malononitrile (CS tear gas), and chloroacetophenone (CN tear gas). A direct comparison of these ionization techniques showed that CI methods are more sensitive than EI for these chemicals (*413*).

Gunpowder and Primer Residue Detection. The use of predetermined threshold levels of barium and antimony to identify the presence of gunshot residues was challenged. Priming mixtures were found to have widely varying barium and antimony compositions. This precludes the establishment of a threshold level for either or both metals (*414*). A scheme was presented for characterizing gunshot residue particles by morphology and elemental composition. The possibility of discriminating between discharge residue from cartridge-operated industrial tools and from firearms was examined (*415*). By use of scanning electron microscopy (SEM) with an energy dispersive X-ray analyzer, glue-lift collections of gunshot residue particles from the hands of a firer were used to reconstruct a suicide victim's hand position at the time of firing (*416*). Neutron activation analysis was used by Brandone et al. (*417*) to investigate the chemical composition of lead and jacketed bullets. Trace elements were used to identify the bullet's manufacturer and to differentiate different batches. Trace levels of antimony, copper, arsenic, and tin in bullet lead were also analyzed by neutron activation analysis (*418*). Shooting distances were estimated by measuring the concentrations of antimony and lead on the target (*419*). The effects of weapon, ammunition, and target surface on the determination of the distance from the muzzle to the target through trace elemental analysis was evaluated by Eigendorf et al. (*420*). A kit consisting of sodium rhodizonate and rubeanic acid was devloped to characterize bullet hole (*421*). Thieme (*422*) discussed the determination of copper, lead, and antimony by X-ray fluorescence for the purpose of evaluating shooting distances. Firearm discharge residues from ammunition of Indian origin have been analyzed by neutron activation analysis (*423*). Detectable amount of nitrites on a shooter's hands were found utilizing a modified Greiss reagent (*424*).

Fibers and Hairs. Melting point and refractive index determinations were made on colorless polyester fibers to assess the comparative value of such data. Melting points proved to be of little comparative value, but refractive indices do allow for discrimination between samples (*425*). A combination of IR spectroscopy and melting point determination was found suitable for identifying polyolefin fibers. Polypropylene fibers can be discriminated from all the other olefin types and mixtures by IR spectroscopy (*426*). Pairs of fibers not distinguishable by common examination procedures were shown to be differentiated by UV microfluorometry. This was achieved by coupling a fluorescence microscope with a microspectrofluorometer (*427*). Single fibers less than 1 mm long were thermally shrunk with a hot stage mounted on an optical microscope. In general it was found that this approach may be used to discriminate acrylic fibers (*428*). Garger (*429*) described an improved technique for preparing solvent cast films suitable for producing IR spectra from fiber samples as small as 1 μg. A method for measuring the birefringence of fibers with a microspectrophotometer was reported. The method has advantages for measuring the birefringence of highly birefringent and thick fibers (*430*). The identification of single fibers and fiber blends by pyrolysis GC was described by Perlstein (*431*). A sequential extraction scheme and TLC analysis of the extract were shown to correctly classify dyes from polypropylene fibers. Pigment colored polypropylene fibers were characterized by visible microspectrophotometry (*432*).

Grieve (*433*) reviewed methods for collecting and examining fiber evidence. The forensic value of fiber evidence was discussed as well. Methods for searching and recovering forensic fiber evidence were suggested by Fong (*434*). Identifications and comparisons were effected by microscopic techniques. Experiments were conducted on the persistence and transfer ability of fibers between articles of clothing. Experiments confirm that caution must be exercised in interpreting fiber distribution in casework situations (*435*).

A technique for examining hair's medullary microstructure with a light microscope was discussed. The technique confirmed that medullary microstructure has significant taxonomic value in animal hair identification (*436*). A blind study was conducted utilizing hairs collected from individuals for the purpose of assessing the forensic value of hair comparisons. The results affirm that with a "good degree of certainty" one can associate hair(s) to a particular individual in a crime-related case (*437*). Scalp hair samples from twins and triplets

were used in a blind study to evaluate the significance of forensic hair comparisons. In some cases in which two or three questioned hairs were compared with several known hairs, the hairs were matched with known samples that were not the source of the questioned hair. The experiments illustrate the caution necessary in the comparison of common featureless hairs (*438*). A case example showing the utility of the comparison microscope for hair examination was reported by Strauss (*439*). The cuticular scale pattern and diameter of adult human hair were used to categorize hair into three different types: scalp, eyebrow and eyelash, and the rest of the body. Diameter studies confirmed that androgen-dependent sizes had consistently wider hairs (*440*). Measurements of medullary fraction and width were used to devise a computer program to assist in determining whether an unknown hair could have come from a cat or dog (*441*). Hairs tested with several chemical reagents were examined by SEM. The authors suggested that this approach could be useful in the discrimination of human hairs since chemically induced topological changes on the hair shaft apparently exhibit a high degree of intraindividual consistency (*442*). Mudd (*443*) described a chromosome staining procedure using quinacrine mustard that permits hair root sheath cells to be reliably sexed. The results suggest that the sex of an individual could be determined from hair roots maintained for at least 100 days.

The detection of methamphetamine and amphetamine in hair was accomplished by GC/MS (*444–446*). Gas chromatography and GC/MS were used to detect methamphetamine, amitriptylene, imipramine, nicotine, and their metabolites in human hair (*447*). Hair samples were analyzed for opiates by radioimmunoassay. By sectioning the hair, the approximate period of drug use could be determined (*448*). By use of TLC, GC, and GC/MS, chloroquine and its major metabolite were identified in hair samples (*449*). Esterase D and glyoxalase I were detected in freshly plucked hair roots. The enzyme intensity was less in cadaver hair roots than in those removed from living individuals (*450*). PGM types determined from hair samples were unaltered by blood transfusions (*451*). Erythrocyte acid phosphatase (EAP) was also determined in human hair root sheaths (*452*). The structure of bird feathers was reviewed. Their examination by SEM and the numerical assessment of node density were found to increase the objectivity of feather identification (*453*).

Glass and Plant. Refractive index data for glass samples collected by English forensic science laboratories have been collected and published (*454*). In many instances annealing may provide a more uniform refractive index distribution over a glass surface. Locke et al. (*455*) described equipment for annealing glass particles. Refractive index variations in glass objects were determined. Refractive index variations were largest in thick shop windows but were also detectable in window panes. Annealing significantly reduced variations in refractive index (*456*). A computer program was proposed for interpreting the significance of refractive index data obtained from glass fragments (*457*). Regression analysis was used to estimate the density of glass samples from a determined refractive index value of glass (*458*). A statistical analysis made on control and recovered glass fragments was extended to deal with the possibility that the recovered fragments may originally have come from more than one source (*459*).

Hickman (*460*) reviewed different analytical techniques that are useful for the identification and comparison of glass particles. The determination of a number of elements in glass samples has proved to be very successful for both classification and discrimination purposes. Seven elements proved to be most useful for discriminating modern sheet glasses (*461*). The concentrations of 22 elements in glass were determined. The single best element for classifying sheet, container, and tableware glasses was magnesium. A combination of six elements was found to provide a firm basis for a classification scheme for glass samples (*462*). Elemental and refractive index data were collected for 11 types of glass used within Australia. The elemental composition was determined with an energy dispersive X-ray spectrometer (*463*). Zorro (*464*) reported on the distribution of glass fragment sizes flying backward from a broken window. An interference objective has been used to microscopically examine glass surfaces for characteristic features. Preliminary studies showed that curved surfaces from tableware and bottles could be distinguished from plain sheet glass (*465*). Interference filters were investigated as alternatives to sodium vapor lamps to establish their suitability for making refractive index measurements. It was found that variations in the characteristics of the filters and their method of mounting could give rise to systematic errors (*466*). Locke (*467*) has discussed the application of interferometry to the examination of glass particles. A novel tilting and transferable microscope stage was described which was specifically designed for handling and examining irregularly shaped particles. to reduce subjectivity, a numerical criterion was devised to assess the curvature of glass particles and this was then applied to a survey of different types of glass (*468*).

Many single layer household paints of varying colors were successfully discriminated by their solubilities in various reagents (e.g., acetone, chloroform, sulfuric acid, and nitric acid) (*469*). A scheme for the characterization of automotive finishes based on solubility tests was discussed. This scheme permits solvent-thinned lacquers to be distinguished in most instances from nonaqueous dispersion lacquers (*470*). A computerized automotive paint library along with a search and retrieval system was developed. The system was based on the classification of the paint's chemical components and the colors of the paint (*471*). In order to implement the computerized search, a collaborative study involving five Canadian forensic laboratories was undertaken. The participants used IR spectroscopy to identify major chemical components in paint (*472*). A visible microspectrophotometer has been used to study the variations in topcoat paint color over the surface of an automobile. This variation proved to be small and thus would not prevent the comparison of paint by this technique (*473*). The reflectance spectra of small paint chips were measured with a visible microspectrophotometer. These data can be used in combination with a computer program to identify the pigments present in paint samples (*474*). The results of a study indicated that the Munsell Matte collection can be used successfully for the color classification of automotive paint primers (*475*). A simple method utilizing polyester resin was presented for the cross sectioning of paint chips (*476*). Methods for the analysis of small quantities of paint samples obtained in criminal investigations were described by Nielsen et al. (*477*). A survey of crow bars showed them to have wide variations in blade tip shapes, dimensions and paint formulations. The latter were determined by visible microspectrophotometry and pyrolysis mass spectrometry (*478*).

Fingerprints. Experience gained with an argon-ion laser has shown that it is a valuable technique to be used in conjunction with other fingerprint detection methods (*479*). A fluorescence spectrometer utilizing a laser excitation source has been designed for the electronic development of latent fingerprints (*480*). Increased fingerprint ridge detail was obtained from ninhydrin-developed prints by dipping the print in zinc chloride reagent. Luminescence was produced with a xenon arc lamp following cooling of the print to liquid nitrogen temperatures (*481*). Strongly luminescent fingerprints were produced on paper by initial treatment of the latent print with 4-chloro-7-nitrobenzofurazan (NBD) chloride followed by excitation with a 150-W xenon arc lamp. The quality and detail of the print were equal to that obtained with an argon-ion laser (*482*). NBD chloride was found to be a more sensitive developing reagent than ninhydrin for 3–9 month old fingerprints and at least equal to ninhydrin in all other cases (*483*). A solution of iodine and 7,8-benzoflavene was found to be an extremely sensitive reagent for a developing old latent fingerprints on porous surfaces (*484*). A very pronounced enhancement in detectability of latent fingerprints was obtained when ninhydrin treatment is combined with the application of two hydrolytic enzymes, trypsin and pronase (*485*).

A rapid method for developing latent fingerprints by exposure to cyanoacrylate ester fumes was described (*486*). Investigations indicate that fuming latent fingerprints with glue containing cyanoacrylate ester can on its own or in combination with laser illumination, add substantially to latent print detectability. In addition, glue treatment can be effectively combined with dusting using fluorescent powder, staining using fluorescent dye, and ninhydrin/zinc chloride treatment (*487*). Two staining procedures were recommended for improving the quality of fingerprints developed with cyanoacrylate ester fumes (*488*). Through heating, a high concentration of cyanoacrylate vapors was produced to reduce

the time required for the development of latent prints (*489*). Organic-based fluorescent powders were used for developing or enhancing latent fingerprints on nonporous surfaces (*490*). Organic-based fluorescent powders were also used for developing latent prints on smooth surfaces (*491*).

Miscellaneous. The applicability of high-performance TLC for ink analysis was evaluated. The technique was found to be superior to conventional TLC in terms of speed, sensitivity, and resolution (*492*). HPLC techniques were developed for the semiquantitative analysis of pigment components and manufacturing artifacts present in alkali blue pigment which is commonly found in black letterpress and offset inks (*493*). The application of visible microspectrophotometry to document examination was reviewed. Visible spectra from ball-point, fiber-tip, and fountain pen ink and printing ink were measured (*494*). Visible spectra have been measured for common ball-point and fiber-tip inks using a visible microspectrophotometer. Comparison of these spectra provided a high degree of discrimination between similarity colored inks (*495*). Ink writing on photographic paper has been analyzed by TLC (*496*). Chromatographic differences between pen inks were revealed by IR luminescence photography of TLC plates (*497*). Ink toners used in photocopying machines were classified and characterized by IR spectroscopy (*498*). The chemical composition of these ink toners was assessed by evaluating their characteristic IR adsorption bands (*499*). The application of the SEM to document examination problems was evaluated by Baier (*500*). Minerals used as fillers in paper making were were characterized by X-ray diffraction. The crystallinity of the cellulose constituent of paper is a function of the raw material used and the manufacturing process. Various types of paper were characterized by measuring their cellulose crystallinites (*501*). Spark source mass spectrometry was used as an analytical technique to ascertain the origin of paper samples (*502*). Argon laser examination was applied to document examination problems (*503*). Optimum conditions were obtained for the examination of documents using an electrostatic detection apparatus device to visualize indented writings (*504*). The theory and applications of the electrostatic detection process for the detection of indented writings on documents were reviewed (*505*).

HPLC was shown to be capable of providing for significant discrimination in the comparison of lipstick smears. However, care must be exercised in the interpretation of these chromatograms. Some difficulties arise from the disappearance of component peaks in the chromatogram of worn smears or the occasional appearance of spurious peaks (*506*). A combination of TLC and GC was demonstrated to be suitable for the characterization and discrimination of small quantities of lipsticks (*507*). A general scheme of analysis for suspected cosmetic smudges, including extraction from a substrate, sample preparation, and determination of major organic and inorganic components, has been described (*508*). A computerized search program was developed to evaluate X-ray diffraction patterns in an on-line mode permitting mixture components to be determined from a data file of forensically relevent substances (*509*). The applicability of pyrolysis capillary GC for the discrimination of paints, rubbers, adhesives, polyurethane, foams, and fibers was demonstrated. This method gave long-term reproducible retention time values (*510*). Comparative analysis of synthetic polymers was performed by pyrolysis IR spectrometry, pyrolysis GC, and energy dispersive X-ray analysis. After pyrolysis the residual material was analyzed by the latter technique (*511*). Pressure-sensitive adhesives were characterized by pyrolysis GC and IR spectrometry (*512*). Markings on mass produced plastic bags allowed sequentially made bags to be related to each other (*513*). Capillary and packed column GC were used to differentiate paraffin waxes and to identify different types of engine lubricants (*514*).

Pyrolysis mass spectrometry was used as a method for characterizing strains of *Streptococcus salivarius*. This approach has potential application for identifying the origin of bite marks (*515*). A microdiffractometer designed to provide forensic analysts with the capability of analyzing small samples was described by Banks et al. (*516*). The diffractometer was used mainly for drug analysis. Dixon (*517*) discussed comparative physical microscopic techniques for linking a torn match to a single matchbook. Cut ends of aluminum and copper wires made by different pliers have been compared by SEM to correlate the two pieces of wire and identify the cutting tool (*518*). Methamphetamine and amphetamine were detected in nail clippings by GC/CIMS (*519*). A computer-retrieved system was recommended by Brown (*520*) to aid in the identification and quantification of forensic substances, as well as with the comparisons of results obtained from different cases.

Sieving techniques used in conjunction with the forensic examination of soils were reviewed. A wet sieving technique was recommended for the production of accurate and precise results (*521*). Density-gradient methods for the comparison of soils was reviewed. It was shown that the techniques cannot be used to reliably prove the origin of soil. The authors concluded that its test results be used with caution and it is preferable that the test be avoided altogether (*522*). Strauss (*523*) discussed the pros and cons of using statistical probability to evaluate the significance of physical evidence in the courtroom. The proper application of statistics to the interpretation of forensic evidence was discussed by Stoney (*524*).

BOOKS

There have been some noteworthy books published on forensic science topics since 1982. An overview of topics relevant to the scientific examination of physical evidence is contained within "Forensic Science-An Introduction to Criminalistics" by De Forest, Gaensslen, and Lee (*525*). A three volume set aimed at giving the reader a basic understanding of pertinent topics in forensic science and legal medicine is to be found within "Forensic Sciences" edited by Wecht (*526*). The classic text "Kirk's Fire Investigation" has been revised by DeHaan (*527*), as has "The Crime Labroatory" by Osterburg (*528*). The latter contains a comprehensive pictorial collection depicting techniques for examining items of physical evidence. In the area of toxicology and drug analysis "Advances in Analytical Toxicology-Volume I" edited by Baselt (*529*) presents chapters on current topics of interest in forensic toxicology that are written by noted authorities. "Topics in Forensic and Analytical Toxicology" edited by Maes (*530*) presents the proceedings of 1983 Annual European meeting of the International Association of Forensic Toxicologists. "High-Performance Liquid Chromatography in Forensic Chemistry" edited by Lurie and Wittwer (*531*) reviews the fundamentals of HPLC and details application of HPLC to forensic drug analysis, toxicology, explosives, and ink analysis. Chromatographic and spectral data for 600 selected drug compounds are contained within Volumes I and II of "Instrumental Data for Drug Analysis" by Mills et al. (*532, 533*). "An Eight Peak Index of Mass Spectra of Compounds of Forensic Interest" by Ardrey et al. (*534*) contains mass spectral data for over 2000 selected compounds. Gaensslen (*535*) has published an exhaustive work detailing both the theoretical and practical aspects of forensic biochemistry. "Proceedings of the International Symposium on the Analysis and Detection of Explosives" (*536*) is a collection of papers of current interest on analytical techniques related to the detection and characterization of explosives.

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Rubber

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This review covers methods for identification, characterization, and determination of rubber and materials in rubber. Literature which became available to the author between November 1982, the end of the period covered by the last review in the series (*47*), and November 1984 is reviewed. Abbreviations recommended in ASTM Designation D1418-18 have been used (*3*). There are listed in Table I.

Table I. Abbreviations Recommended by ASTM (*3*)

BR	butadiene rubber
CR	chloroprene rubber
EPDM	terpolymer of ethylene, propylene, and a diene with residual unsaturated portion of the diene in the side chain
IR	isoprene synthetic rubber
NBR	nitrile–butadiene rubber
NR	natural rubber

GENERAL INFORMATION

The American Society for Testing and Materials published the 1983 annual edition of test methods for rubber (*3*).

Some of the new standards issued by International Organization for Standardization (ISO) are

1. International Standard 6101/1-1982. Rubber—Determination of Metal Content—Flame Atomic Absorption Spectrometric Method—Part 1: Determination of Zinc Content.
2. International Standard 6235-1982. Rubber, Raw-Determination of Block Polystyrene Content—Ozonolysis Method.

The third edition of the book "Analysis of Rubber and Rubber-like Polymers" was published (*97*). This book presents a balanced picture of the current technology in the rubber and rubberlike polymers. Chapters are given on sampling and sample preparation, extractions, analysis of extracts, chemical analysis for polymer type, quantitative elemental analysis, solution methods, instrumental polymer analysis, polymer characterization, inorganic fillers and trace metal analysis, carbon black, formulation derivation and calculation, blooms and visually similar phenomena, and finally validity of results.

The review on analysis of high polymers (*88*) presented important references which have bearing on the subject of this review as well. An account of the approach and methods used in an industrial laboratory for analysis of rubber compounds was presented (*57*). Chemical analysis of plastics and elastomers was the subject of a book published recently (*46*). One chapter in a recent publication delineates the various isomerization and cyclization reactions which occur with rubber under pyrolytic and nonpyrolytic conditions (*33*).

Various aspects of polymer characterization are detailed in the recent publication edited by J. J. Dawkins (*21*).

MICROSCOPY

Some of the techniques used by microscopists to isolate and identify contaminants in rubber by light and electron microscopy were discussed (*72*). Techniques for isolation use forceps, needles, replicas, selective dissolution, thin sectioning, and direct transfer. Identification by using polarized light microscopy, morphology, and IR analysis was detailed.

Phase contrast produced in electron microscopy by defocus techniques was used to obtain the first unstained images of styrene–isoprene and styrene–butadiene diblock and triblock copolymers (*36*). Theoretical image calculations based on square-wave and circular cross sectional one-dimensional models were used to demonstrate the effects of mean inner potential difference, interface width, and microscope optics on resultant images. Experimental phase contrast images of microtomed block copolymers with ordered lamellar, cylindrical, and disordered spherical morphologies were in good agreement with theory and experimental scattering contrast images (osmium tetraoxide stain). The phase contrast technique was sufficient to visualize the phase-separated regions of polymers of similar atomic composition and for density.

Transmission electron microscopy studies with ultrathin sections of cords embedded in rubber were shown to provide valuable information about resorcinol–formaldehyde–latex and predip locations which could not be obtained by standard optical methods (*31*). A scanning electron microscopy study was made of the tear structure of SBR cross-linked with either a sulfur-curing system or a peroxide-curing system (*83*). The effect of the addition of ISAF carbon black and the influence of cross-link density in the case of the sulfur-cured vulcanizates were examined.

The particle size distribution of BR latex was studied using a transmission electron microscope coupled to a computer-controlled image analyzer by means of a fixed focus television camera and a fiber optic fluorescent screen (*34*). By a special agglomeration program, it was possible to distinguish highly agglomerated particles as single particles, approximating their shape to a spherical one.

A description of the principles of Nomarski double beam interface contrast microscopy was given and the use of this technique in the study of polymeric materials was illustrated (*74*). Scanning electron microscopy was used to study the ozone resistance of NR and NR/EPDM blends (*61*). Advances in SEM of polymers was the subject of a recent review by White (*99*).

NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY (NMR)

The use of NMR for studying complex molecular structures of diene polymers and copolymers was reviewed (*37*). Polymers examined were BR, BR copolymers, IR, IR copolymers, polypentadiene, pentadiene copolymers, CR, CR copolymers, and other modified diene polymers. A model was established to illustrate the magnetic relaxation properties of proton pairs linked to strongly entangled chains (*1*). This model was compared with NMR spectra of real chains like *cis*-1,4-BD. This indicated that the splitting of the chain relaxation spectrum into two well-defined dispersions may be perceived

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