

California Association of Criminalists

SEPTEMBER 1981

OFFICERS 1981-82

President EDWARD RHODES Los Angeles Sheriff's Office 2020 W. Beverly Blvd. Los Angeles, California 90057

President-Elect FRED A.J. TULLENERS California Department of Justice Criminalistics Laboratory P.O. Box 3679 Riverside, California 92519

Recording Secretary JOHN D. DE HAAN California Department of Justice Technical Support Unit P.O. Box 13337 Sacramento, California 95813

Treasurer DUANE LOVAAS California Department of Justice 2213 Blue Gum Avenue Modesto, California 95351

Regional Director — North MARY GIBBONS Forensic Science Associates P.O. Box 8313 Emeryville, California 94608

Regional Director — South JAMES N. STAM San Diego Police Department 801 West Market Street San Diego, California 92101

Immediate Past President ROBERT OGLE Institute of Criminalistics 1413 Georgia Street Vallejo, California 94590

Membership Secretary DOROTHY NORTHEY Contra Costa County Sheriff's Office P.O. Box 391 Martinez, California 94553

Editorial Secretary GEORGE F. SENSABAUGH School of Public Health University of California Berkeley, California 94720

CONTENTS

Association Activities 2
Committee Appointments 3
McCrone Course 4
Employment 4
Upcoming Meetings 5
Pan American Association of Forensic Sciences 5
Inter-American Congress 6
Revisions in Alcohol Analysis Regulations - Kathi Holmes . 7
Separation of Cocaine from Common Excipients and Adulterants - Jerry O'Donnell
Ethical Dilemma - Peter Barnett
Kinetics of the Reaction of Ninhydrin with Phenethylamines of Abuse - Beth Hendrickson, John Thornton, and Ralph Maloney
Laboratory Aid: A Method to Aid in the Precision of Trigger-Pull Determination - Frank Cassidy20
ATF Charcoal Tube Method for Extracting Accelerants from Arson Debris - Darryl Tate21

This mailing includes the following items:

- 1. Membership Roster and Student Affiliate Listing
- 2. Proxy for Fall Seminar Business Meeting
- 3. Replacement Pages for Forensic Safety Guidelines The preceding printing was missing the table of contents; the two replacement pages correct this.

ANNOUNCEMENT REGARDING CAC ABSTRACTS

The second printing of the CAC ABSTRACTS should be ready by the Fall Seminar. Members who have not yet received their copy should contact either the membership secretary (Dorothy Northey) or their regional director (Jim Stam, South; Mary Gibbons, North).

ASSOCIATION ACTIVITIES

Southern Section Meetings

A meeting hosted by DOJ-Riverside on Psycho-motor testing to detect alcohol impairement was a success. Over 50 people attended the meeting held at Don Jose's in Riverside. This was the second Southern Section dinner meeting in a row that has had high attendance.

Another Southern Section meeting is scheduled for Friday, September 25, at the Twin Inns in Carlsbad. The meeting will be hosted by the San Diego Police Dept. The talk will be given by Lance Martini on the "Forensic Evaluation of Accelerator Cartridges".

Northern Section Meetings

The famous Contra Costa Home Cooked Feed and Volleyball Tournament scheduled for 12 Sept. was cancelled. The next meeting will be in October and will be hosted by the San Mateo Police Lab.

Study Group Meetings

1. Northern Trace Group. The July meeting was hosted by Oakland Police Dept. At this meeting, tests used to discriminate between generic fiber types were discussed. The tests discussed included polarizing microscopy, solubility testing, melting point determinations and instrumental analysis.

At the August meeting hosted by San Mateo City Laboratory, results of the fiber characterization exercise begun earlier in the year were discussed.

The next meeting will be held September 24 at the Contra Costa Co. Sheriff's Office. Dr. Joseph Di Giorgio of California State University at Sacramento will discuss infra-red analysis of fibers.

- 2. Southern Trace Group. Jim White is the Chairman of the newly-formed Trace Evidence Study Group. The first meeting was held August 14 and discussed cross-sectioning techniques as well as the direction the study group was to take. The study group will tentatively meet in October and aim for meetings every six weeks.
- 3. Southern Serology Group. The group's last meeting covered the topics of GLO \overline{I} development and the systematic analysis of rape evidence and its significance. Also covered were problems encountered with P-30 analysis. The next meeting will be held in late September/early October and will cover Lewis typing among other topics.

COMMITTEE APPOINTMENTS 1981-82

TRAINING & RESOURCES

Steve Shaffer* Linda Baxter Richard Berger Marty Blake Bill Casper Benny Del Re Heram Evans Mary Gibbons Michael Grub Lucien Haag Jim Hall John Hartman Celia Hartnet James Kurrasch Warren Loomis Greg Matheson James Norris

John Patty
George Roche
Gary Sims
Theresa Spear
Jim Stam
Jim White
Sandy Wiersema
Brian Wraxall

ETHICS

Cecil Hider* Charles Morton Lucien Haag

BY-LAWS

Peter Barnett*
Parker Bell
Frank Fitzpatrick

PUBLIC RELATIONS

Jim Stam* Peter Barnett Norm Wallis

ACCREDITATION LIAISON

Enrico Togneri

AWARDS

Karen Sheldon* Bruce Fukayama Celia Hartnet

HISTORICAL

Paul Dougherty* Duayne Dillon Norm Wallis

PUBLIC HEALTH LIAISON

Kathy Holmes* Lance Gima

SEMINAR - Fall 81

Enrico Togneri Richard Berger

*Chairperson

McCRONE COURSE ON MICROSCOPY OF EXPLOSIVES

A course on the microscopy of explosives is being planned for 18-22 Jan. 1982; the location in California has not yet been designated. The fee will be about \$350; McCrone will supply all equipment and materials.

The course will focus on the use of polarized light microscopy for the study of forensic explosives: propellants, high explosives, boosters, detonators and fuses.

The objective will be characterization of any such material for the purposes of identification and comparison, quality control and trouble shooting. The relationship between performance (power, sensitivity, etc.) and composition or structure will be covered as well as the effects of polymorphisms.

Monday: Use of polarizing microscope

Crystal morphology

Tuesday: Crystal optics

With polarizer only and with crossed polars

Wednesday: Polymorphisms

Mixed explosives

Thursday: Study of high explosives and boosters

Study of primary explosives and detonators

Friday: Study of propellants and fuses

If you are interested in the above course, please contact Nancy Daerr (McCrone Research, 2820 S. Michigan Ave., Chicago, Ill., 60616, (312)842-7100) or Ed Rhodes (LASO Crime Lab, 2020 W. Beverly Blvd., Los Angeles, CA 90057, (213)974-4611). If there is sufficient interest, separate Northern and Southern classes can be given. Also, McCrone Research would be willing to do a second course on another topic (botanicals, soils, fibres, etc.) if there is sufficient interest; contact Nancy Daerr or Ed Rhodes.

EMPLOYMENT OPPORTUNITIES

- 1. SUPERVISING CRIMINALIST, El Cajon. Requires B.A. or B.S. with a major in Criminalistics or a relevant natural or physical science and progressively responsible experience in the field. For applications or information, contact the Personnel Dept., City of El Cajon, 200 E. Main St., El Cajon, CA 92020.
- 2. CRIMINALIST, Las Vegas. Requires B.A. or B.S. in chemistry, bacteriology, microbiology or closely related field. Also experience in serology, hair and fiber identification, and general trace evidence analysis. Contact T.W. Biggs, Personnel Bureau, Las Vegas Metropolitan Police Dept., 400 East Stewart, Las Vegas, Nevada 89101, (702) 386-3111.
- CRIMINALISTS, Dade County, Florida. Openings in serology, drugs-trace, firearms, and scanning electron microscopy. Contact Ed Whittaker, Crime Lab Bureau Commander, 1320 NW 14th St., Miami, Florida 33125.

UPCOMING MEETINGS

- 1. California Association of Criminalists Northwest Association of Forensic Scientists Joint Meeting, 4-7 November, 1981.

 Sahara Tahoe-South Shore. Contact Rico Toqueri, Floyd Whiting or Richard Berger, Washoe County Sheriff, P.I. Box 2915, Reno, NV 89505.
- 2. California Association of Criminalists, Spring Seminar, 1982.

 Note this meeting's venue is to be changed to the south; further details are to be announced.
- 3. California Association of Criminalists Concurrent Meeting
 with the Pan America Congress 2-5 November, 1982. Sacramento, CA.
 Contact John DeHaan, Calif. Dept. of Justice Laboratory, Box 13337,
 Sacramento, CA. 95813.
- 4. Midwestern Association of Forensic Scientists 14-17 October, 1981, Kansas City, Kansas. Contact Bill Chapin, Johnson County Crime Lab. 6000 Lamar, Mission, Kansas, 66202.
- 5. Midwestern Association of Forensic Scientists 12-14 May, St. Louis, Mo. Contact Robert Briner, SEMO Regional Crime Lab, Cape Girardeau, Mo 67301.
- 6. Southwestern Association of Forensic Scientists, 6-7 November, Oklahoma City, OK. Program Chairman is Don Flynt, OSBI, Oklahoma City, P.O.Box 11497, Oklahoma City, OK 73136.
- 7. Northwestern Association of Forensic Scientists, 23-24 October, Allentown, PA. Contact: Alex Stirton, NEAFS, Box 133, Limeport, PA 18060, (215) 691-6110 ext. 256.
- 8. Society of Forensic Toxicology, 4-6 November,
 Chicago, Illinois. Contact Dr. Eleanor Berman, Division of Biochemistry, Cook County Hospital, 1835 W. Harrison St., Chicago, IL 60612.
- 9. Canadian Society of Forensic Science, 24-28 August,
 McMaster University, Hamilton Ontario. Program Chairman is
 Brian Dixon, Centre of Forensic Sciences, 25 Grosvenor St.,
 Toronto, Ontario, Canada M7A 2G8.

PAN AMERICAN ASSOCIATION OF FORENSIC SCIENCES

This new organization grew out of discussions held at the Pan American Conference on Forensic Sciences held in Mexico City in 1977. Its purpose is to foster communication between forensic scientists in North, Central, and South America. The first inter-America Congress in Forensic Medicine and Sciences will be held in Sacramento, Nov. 1982 (see announcement elsewhere in this issue). The Secretariat is c/o Dr. William Eckert, Milton Helpern Center, Box 95, Wichita State Univ., Wichita, KA 67208.

First Inter-American Congress of Forensic Sciences

The California Department of Justice, Bureau of Forensic Services, is pleased to announce that they have been selected to host the First Inter-American Congress of Forensic Sciences in November 1982. John DeHaan, the Bureau's Information Officer, has been selected as President of this Congress and will be responsible for organizing it. This will be the inaugural meeting of the Pan-American Association of Forensic Sciences and promises to be an exciting debut of a new cooperation between forensic scientists from all of the Americas -- South America, Central America, the Caribbean, Canada and the U.S. It is anticipated that this meeting will be a joint meeting between the Pan-American Association, the California Association of Criminalists, the California Association of Toxicologists and the American Society of Questioned Document Examiners, thus bringing together scientists from all disciplines.

A number of Bureau personnel are already involved in making plans for this meeting: Robert Prouty has offered to organize the Questioned Documents Section; Victor Reeve will coordinate the technical exhibits; Spiro Vasos will organize the identification and fingerprint programs; and Tom Corcoran will coordinate the audio-visual resources. From outside the Bureau, Jan Bashinski of the Oakland PD Crime Lab will be responsible for the criminalistics program and Dr. Brian Parker from California State University, Sacramento will be one of the meeting Vice-Presidents and will be responsible for coordinating the activities of the other organizers. Headquarters hotel will be the Capitol Plaza Holiday Inn in downtown Sacramento. A fully packed five-day technical and social program is planned for the first week of November 1982. Every effort will be made to keep the registration fees as low as possible but they will include luncheons and a barbecue, wine and cheese tastings and various other social events.

It promises to be a most exciting week for the four or five hundred forensic scientists involved. You are invited to participate either as an attendee or as a presenter of a paper. Themes for the meeting will include: Homicide: California Style, Communications in Forensic Science and Sex Crimes. Plan now to attend. This is a rare opportunity for criminalists at the bench level to have an impact on the international forensic scene and it will be a most exciting adventure. We hope you will all participate. For further information contact John DeHaan in Technical Support Unit, (916) 322-3968.

REVISIONS IN FORENSIC ALCOHOL ANALYSIS REGULATIONS

The Department of Health Services held a meeting of a subcommittee of the Advisory Committee on Alcohol Determination on August 5, 1981. The purpose of the meeting was to review the Health and Safety Code sections which govern forensic alcohol analysis and breath alcohol analysis for necessary revisions.

A copy of the first progress report summarizing the results of the August meeting has been included here. Some of the proposed changes impact CAC members.

The Department of Health Services has also asked the CAC for input regarding any desired revisions in the Title 17 regulations. A copy of the letter from Health Services is included and gives the format for suggested revisions.

Members who wish to comment on the proposed Health and Safety Code changes or who wish to suggest revisions in the Title 17 regulations should contact Kathi Holmes, 729 Castro Street, Martinez, CA 94553.

FIRST PROGRESS REPORT

Program Area:

PUBLIC HEALTH LABORATORY SERVICES

Program:

FORENSIC ALCOHOL ANALYSIS AND BREATH ALCOHOL ANALYSIS

Authority:

HEALTH AND SAFETY CODE, SEC. 436.50 - 436.63

I. Mission and Goal Statement

Over 50% of traffic fatalities are associated with drivers under the influence of alcohol. Adding to this toll are the accidental injuries attributed to drunk drivers. One form of control over this problem was the enactment in California of provisions in the Motor Vehicla Code which require arrested drunk drivers to submit to chemical tests to determine blood alcohol concentrations, together with the designation of a blood alcohol concentration above which a person is presumed to be under the influence of alcohol. The effectiveness of this form of control is dependent on the competence of the tests. The H & S Code assigns to the Department of Health Services the responsibility for maintaining the competence of laboratory testing and of breath testing by police officers. The goal of this program is to ensure the reliability of the testing so that courts can reduce the number of deaths and injuries due to drunk drivers.

II. Recommendations to Date

- A. Amendments intended to result in "housekeeping", non-substantive changes.
 - 1. These amendments would delete language, needed when the statute was enacted in 1969, to determine the date by which the Department was to adopt regulations which implemented the statute (July 1, 1970) and the date by which the regulations were effective (January 1, 1971). This language is now considered to be obsolete.

 These recommendations would result in amending Sections 436.50, 436.51, 436.54 and 436.55 as follows:

436.50. Which without 1414 11 1910/ Yine State Department of Health Services shall adopt and publish such rules and regulations to be used in approving and governing the operation of laboratories engaging in the performance of tests referred to in Sections 436.51 and 436.52, including the qualifications of the employees of such laboratories who perform such tests, as it determines are reasonably necessary to insure the competence of such laboratories and employees to prepare, analyze, and report the results of such tests. The rules and regulations shall be adopted, only after the State Department of Health Services has consulted with at least one member of each of the following groups: district attorneys, public defenders, coroners, criminalists, pathologists, analytical chemists, and such other persons deemed by the department to be qualified.

436.51. On or affect landary 1/ 1971/ *The testing by or for law enforcement agencies of blood, urine, or tissue for the purposes of determining the concentration of ethyl alcohol in the blood of persons involved in traffic accidents or in traffic violations shall be performed only by a laboratory approved and licensed by the State Director of Health Services for the performance of such tests.

436.52. The testing of breath samples by or for law enforcement agencies for purposes of determining the concentration of ethyl alcohol in the blood of persons involved in traffic accidents or in traffic violations shall be performed in accordance with regulations adopted by the State Department of Health Services.

The rules and regulations shall establish the procedures to be used by law enforcement agencies in administering breath tests for the purposes of determining the concentration of ethyl alcohol in a person's blood. Such rules and regulations shall be adopted and published in accordance with the provisions of Chapter 4.5 (commencing with Section 11371) of Part I of Division 3 of Title 2 of the Government Code.

436.53. Each laboratory in this state which performs the tests referred to in Sections 436.51 and 436.52, shall be licensed by the State Director of Health Services. Each such laboratory, other than a laboratory operated by the state, city or county or other public agency shall upon application for licensing pay a fee to the State Department of Health Services in an amount, to be determined by the department, which will reimburse the department for the costs incurred by the department in the issuance and renewal of such licenses, but not to exceed one hundred dollars (\$100). On or before each January 1 of each year thereafter, each such laboratory shall pay to the department a fee so determined by the department, not to exceed one hundred dollars (\$100), for renewal of its license.

436.54. On od divided Jidulide 1/1411/ The department shall enforce the provisions of this chapter and such rules and regulations as are adopted by the board.

436.55. On but differ January 1/1911/ fine department shall annually publish a list of approved and licensed laboratories engaging in the performance of tests referred to in Sections 436.51 and 436.52.

(436.55 through 436.63, no change, excepting any instances in which "Services" has not been added to "Department of Health.)

- B. Amendment intended to require that retesting of blood, urine or tissue samples by the defense (i.e., "referee" testing) be performed in accord with the same regulations which govern testing which is done by or for law enforcement agencies.
 - This amendment would terminate the practice of obtaining referee tests
 which are of undetermined reliability because they are performed in
 unregulated laboratories.
 - This recommendation would result in amending Section 436.51 as follows:

436.51. On or after January 1, 1971, the testing by or for law enforcement agencies of blood, urine, or tissue for the purposes of determining the concentration of ethyl alcohol in the blood of persons involved in traffic accidents or in traffic violations shall be performed only by a laboratory approved and licensed by the State Director of Health for the performance of such tests. Any Jaboratory which performs referee, repeat tests shall also be approved and Licensed by the State Director of Health Services.

- C. Amendment intended to clarify the role of breath alcohol concentrations relative to blood alcohol concentrations and to the extent of impaired driving ability.
 - This amendment would bring the statute into correspondence with the egulations [Sec. 1220.4(1)(f), Title 17, California Administrative Code] by acknowledging that the breath sample does not measure actual blood alcohol concentration, since no blood was actually tested. The breath measurement is converted to a blood value using a mathematical factor which relates a breath concentration to an equivalent blood concentration.
 - 2. This amendment would prepare the way for an amendment of the Vehicle Code which now designates 0.10 grams percent, blood alcohol concentration as the level at which impairment begins [Section 13354(f), Vehicle Code]. The amendment in the Vehicle Code would define the impaired level in terms of both breath alcohol concentration and blood alcohol concentration. This would terminate the present practice of courtroom arguments over the mathematical treatment which was used to convert a breath measurement to a blood value.
 - 3. This recommendation would result in amending Section 436,52 as follows: 436.52. The testing of breath samples by or for law enforcement agencies for purposes of determining the <u>equivalent</u> concentration of ethyl alcohol in the blood of persons involved in traffic accidents or in traffic violations shall be performed in accordance with regulations adopted by the State Department of Health.

The rules and regulations shall establish the procedures to be used by law enforcement agencies in administering breath tests for the purposes of determining the <u>equivalent</u> concentration of ethyl alcohol in a person's blood. Such rules and regulations shall be adopted and published in accordance with the provisions of Chapter 4.5 (commencing with Section 11371) of Part I of Division 3 of Title 2 of the Government Code.

- D. Amendment intended to require that testing of breath samples, captured for later analysis by the defense, be performed in accord with the same regulations which govern testing which is by or for law enforcement agencies.
 - 1. This amendment looks forward to the time (considered imminent) when the courts will require, on constitutional grounds, that breath samples be retained for testing by the defense. This amendment would establish the new requirement that such testing be performed according to procedures set forth in regulations adopted by the State Department of Health Services.
 - 2. This recommendation would result in amending Section 436.52 as follows: 436.52. The testing of breath samples by or for law enforcement agencies, or the testing of breath samples captured for later analysis by the defense, for purposes of determining the concentration of ethyl alcohol in the blood of persons involved in traffic accidents or in traffic violations shall be performed in accordance with regulations adopted by the State Department of Health.

The rules and regulations shall establish the procedures to be used by law enforcement agencies and the defense in administering breath tests for the purposes of determining the concentration of ethyl alcohol in a person's blood. Such rules and regulations shall be adopted and published in accordance with the provisions of Chapter 4.5 (commencing with Section 11371) of Part I of Division 3 of Title 2 of the Government Gode.

III. Coneral Comments and Observations

The following persons were asked to participate in the Working Group:

Affed A. Biasoffi
Criminalist, Bureau of Forensic Sciences
State Department of Justice
Lloyde W. Bradley, Jr.
Manager, Driver Improvement
Division of Driver's Licenses
State Department of Motor Vehicles
Kathryn J. Holmes
Supervising Criminalist
Office of Sheriff-Coroner, Contra Costa County
Jack Katz
Chief Criminal Deputy City Attorney
San Diego City Attorney's Office
Daniel R. Morales, Ph.D.
Chief, Clinical Chemistry Laboratory Soction
State Department of Health Services
(Convener)

These persons were selected from the membership of the Department's Advisory Committee on Alcohol Determination. (Mr. Katz was unable to attend the Working Group meeting at which these recommendations were made.) In making its recommendations, the Working Group nevertheless expressed strong feelings of reticence relative to proceeding with these amendments until specific guidance is sought from the Attorney General's Office. The ills that the substantive amendments are intended to remove may be tolerable in light of other problems that may be caused by changing the statute.

Although the other members of the Advisory Committee on Alcohol Determination were asked to mail or telephone their comments or recommendations concerning this statute, no responses were received by the convener by the date of this meeting (August 5, 1981).

IV. Work Process Information

It is difficult to estimate amount of person hours/days invested in this project, since it is interwoven with other work over a long period. A reasonable estimate of this stage would be something of the order of 14 person-days (DHS staff).

V. Future Action

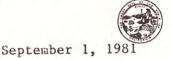
- 1. Short-range.
 - Submission of this document to the Working Group for comment, clarification or revision.
 - b. Submission of the document, reviewed by the Working Group, to the Attorney General's Office for critique.
 - Submission of a revised document to consultants for review and written commentary.

2. Long-range.

- a. Presentation of the recodification project, and its proposals relative to this statute, to the Advisory Committee on Alcohol Determination.
- b. Drafting an amended statute.

DEPARTMENT OF HEALTH SERVICES 2151 BERKELEY WAY BERKELEY, CA 94704

(415) 540-2219



Kathryn J. Holmes Supervising Criminalist Office of Sheriff-Coroner, Contra Costa County 729 Castro Street Martinez, CA 94553

Dear Ms. Holmes:

In preparation for a meeting of the Advisory Committee on Alcohol Determination, I am writing you and other interested persons to request your input to revisions in the regulations for forensic alcohol analysis and breath alcohol analysis (Sections 1215 through 1222.2, Title 17, California Administrative Code). The goal of this work is to update the regulations as needed. After consultation with your group, please send me your recommendations.

Even though you may have made recommendations in the past, I would appreciate your taking the trouble to review them again and submit them again at this time in order that I can be sure that they represent the current thinking of your group.

So that your recommendations can be worked readily into an agenda, submit each individual recommendation in the following form:

- 1. Section number: [e.g., Sec. 12204(1)(f)].
- Designate whether this is an amendment of an existing Section, a deletion of an existing Section, or the addition of a new Section.
- 3. Show the exact wording of the revised Section, using strike-outs to designate deletions, and underlining the designate additions.
- 4. Present succinctly the change or improvement you intend to achieve by the revision.

Use a separate page or set of pages for each proposal. Thank you for your assistance. A copy of the regulations is enclosed for your use.

Sincerely,

Daniel R. Morales, Ph.D. Chief, Clinical Chemistry Laboratory Section

Staff to the Committee

Enclosure: Booklet, "Forensic Alcohol Analysis and Breath Alcohol Analysis"

cc: John M. Heslep, Ph.D.

Chief, Laboratory Services Branch Chairman, Advisory Committee on Alcohol Determination

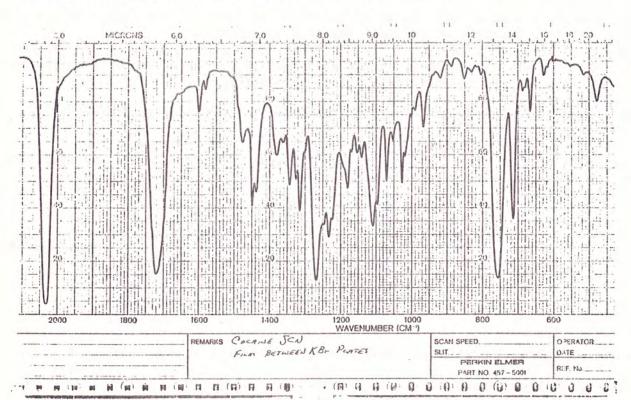
SEPARATION OF COCAINE FROM COMMON EXCIPIENTS AND ADULTERANTS

Jerry O'Donnell Albuquerque Police Dept. Albuquerque, N.M.

(Reprinted by permission from the Southwestern Forensic News.)

Take an amount of sample which will contain about 20 mg of cocaine. (I normally use 50 mg of street cocaine.) Add sufficient water to effect dissolution, then add 1 ml of the Scott reagent (1% cobaltous thiocyanate in 50:50 water:glycerol) and mix well. Add concentrated HCl dropwise until the blue color is just discharged; avoid an excess of acid, which may cause hydrolysis of the thiocyanate salt. Add 5 ml chloroform and extract in a separatory funnel, filtering the blue chloroform layer through filter paper. (Note: Do not use phase-separating filter paper unless it has been thoroughly washed with chloroform, as it contains silicones which will botch the spectrum.) Repeat extraction with 3 ml chloroform. The IR spectrum of the thiocyanate salt may be obtained directly, which is what we do by evaporating the chloroform on KBr plates, or the cocaine may be recovered by hydrolysis with aqueous base. An IR spectrum of the cocaine thiocyanate salt is shown below. Note that the 1215 and 760 peaks are residual chloroform; drying chloroform from the salt is not easy.

This procedure will separate cocaine from mannitol, sugars, starch, and the other 'caines, unless the latter are present in amounts greater than the cocaine. Lidocaine is the chief offender; an IR spectrum thus obtained from a 50:50 cocaine:lidocaine mixture contains several small peaks not present in pure cocaine thiocyanate. However, unless the adulterants are present in large amounts, the interference is minimal.



ETHICAL DILEMMA

Peter D. Barnett

The CAC Code of Ethics does not specifically address the question of technical competence as it may relate to ethical or unethical behavior. For this reason "Ethical Dilemmas" which have previously been presented have, for the most part, avoided technical issues. Criminalistics is, however, a technical business and there are situations in which technical questions must be considered in resolving ethics questions. The case presented below is an effort to combine an ethical dilemma with a technical issue.

In examining vaginal swabs obtained from the victim of an alleged rape a criminalist finds acid phosphatase activity, numerous spermatozoa, and high levels of H antigen on an extract of a portion of the swab which was obtained less than 3 hours after the assault. Debris recovered from a centrifuged extract of a portion of one swab is smeared on a microscope slide and stained, revealing 50-100 sperm per 400% microscope field. Using standard procedures the criminalist determines the victim to be ABO type O nonsecretor. On a cell-free extract from the swab, absorption-inhibition testing results in 8-fold reduction in titer of H lectin. The actual data from the absorption inhibition is shown in the accompanying table. Investigative information indicates the victim has not had sexual intercourse for at least 1 week prior to the rape.

	Anti-A (1/64)	Anti-A (1/128)	Anti-A (1/256)	Anti-A (1/512)	Anti-B (1/64)	Anti-B (1/128)	Anti-B (1/256)	Anti-B (1/512)	H Lectin (1/4)	H Lectin (1/8)	H Lectin (1/16)	H Lectin (1/32)
ontrol (blank)												
10 min.	3+	3+	2+	1+	4+	3+	2+	2+	3+	2+	2+	1+
20 min.	4+	4+	3+	2+	4+	4+	3+	2+	4+	3+	2+	2+
30 min.	4+	4+	3+	3+	4+	4+	3+	3+	4+	4+	3+	2+
wab Extract												
10 min.	2+	1+	1+	-	2+	2+	1+	1+	1+	-	-	-
20 min.	4+	4+	2+	1+	4+	3+	3+	2+	2+	1+	-	-
30 min.	4+	4+	3+	2+	4+	3+	3+	3+	3+	1+	-	-

In examining this data it can be seen that there is some apparent reduction in the titer of the anti-A and anti-B sera. There are two possible interpretations of these data: First, the semen on the swab is from an ABO type O secretor individual. This interpretation assumes that the H-activity is not from the victim and assumes that the apparent A and B activity is due to normal experimental variation in this type of test. An alternative interpretation of the data is that there is strong indication of H antigen from an ABO type O secretor semen donor, but there is also some indication of A and B antigen on the swab, which could only come from semen on the swab.

It is hoped that the data are not so ambiguous as to cause a great deal of difficulty in their interpretation. (It is recognized, however, that alternative interpretations are possible and, if a report were written at this stage of the investigation, different criminalists might write different reports.) The report that is written states, "the semen on the vaginal swabs originated from an ABO type O secretor individual". Subsequently, a suspect is apprehended who turns out to be an ABO type AB secretor.

A supplemental report is written which states "Although the high levels of H-antigen are most consistent with the semen being from an ABO type O secretor, there is some indication of A and B antigen present which could be from an ABO type AB secretor individual". With this additional information the prosecuting attorney can argue that the suspect (type AB) is compatible with the evidence from the victim.

In this situation both technical and ethical judgements need to be made. If the first of the reports is the <u>only</u> technically defensible report, the second report would seem to be unethical under sections II B., II F., II G., II I., or other sections of the CAC Code of Ethics. On the other hand, if the second report is technically the correct one, the first must be unethical under sections II E., II G., II J. of the Code of Ethics.

In commenting on this dilemma it is necessary first to decide the technical questions of which is the technically correct interpretation of the data. Then a decision must be reached as to whether or not either or both of the reports is in violation of the Code of Ethics.

Reponses to the July "Ethical Dilemma" agreed that no discussion should take place with the prosecutor without some further authorization or Court Order. Mike Grubb contends that this issue is a matter of rules of evidence rather than Code of Ethics. (But, whose rules of evidence, Alaska or California?) Steve Shaffer says that it is necessary to determine one's obligations under Alaska law, and Dave Sanchez would require either written authorization from the defense attorney, or a Court Order from an Alaska Court.

RESPONSE TO OCTOBER ETHICAL DILEMMA

First, how would you resolve the technical issues? Which is
the correct report:
O Alternative A: The report states "The semen is from an
ABO type O secretor".
O Alternative B: The report states "The H-activity indicates
semen from an ABO type O secretor; however,
there is also some indication of A and B anti-
gen so the semen could be from an ABO type AB
secretor".
O Both A and B may be correct, given the data
Second, is there an ethical dilemma and how do you resolve it?
O This is purely a technical question and not an ethical one.
Only one interpretation of the evidence is possible: A O
or B. O Therefore, the other interpretation is technically
in error and is O or is not O a violation of the
Code of Ethics.

Comments:

Send To: Peter Barnett
Forensic Science Associates
P. O. Box 8313
Emeryville, Calif. 94608

KINETICS OF THE REACTION OF NINHYDRIN WITH PHENETHYLAMINES OF ABUSE

Beth Hendrickson, John Thornton, and Ralph Maloney
Forensic Science Group
School of Public Health
University of California
Berkeley, California 94720

INTRODUCTION

The reaction of ninhydrin with amines, amino acids and peptides has been used extensively in qualitative and quantitative biochemical investigations. In the forensic domain, ninhydrin has been used in particular as a detection reagent for latent fingerprints, and to a lesser extent as a test for certain drugs of abuse. It has been used for the visualization of drugs of abuse on TLC plates¹⁻³, and as a spot test to differentiate among phenethylamines⁴.

The chemistry of the ninhydrin reaction with amines is the subject of an extensive review by McCaldin⁵, and the mechanism of the reaction with secondary amines is described by Yamagishi and Yoshida⁶.

The reaction of ninhydrin with primary amines is thought to proceed as follows:^{5,7}

OH + RCH₂ NH₂
$$\stackrel{\text{H}^+}{\longrightarrow}$$
 OH $\stackrel{\text{H}^+}{\longrightarrow}$ OH $\stackrel{\text{H}^+}{\longrightarrow}$ CHO

(I) NINHYDRIN

(III) $\stackrel{\text{H}^+}{\longrightarrow}$ (III)

(IV) (V) RUHEMANN'S FURFLE

FIGURE 1. Mechanism of reaction of ninhydrin with primary amines

In the case of N-methylamines, such as methamphetamine it is generally accepted that the mechanism proceeds through the formation of formaldehyde and methylamine:^{5,6}

FIGURE 2. Mechanism of reaction of ninhydrin with secondary amines

In this reaction, the methylamine then reacts in the manner of primary amines to yield the characteristic "Ruhemann's Purple". It is difficult to reconcile these mechanisms, however, in light of the sensitivity reported for primary and secondary amines of abuse by Cashman et al.⁴. Cashman and co-workers report a sensitivity of 3 micrograms for primary amines (amphetamine) and 1 microgram for secondary amines (methamphetamine). This apparent anomaly prompted the present work, which was directed at elucidating the kinetics of the reaction of ninhydrin with phenethylamines of abuse. Amphetamine was selected as the representative primary amine, and methamphetamine was selected as the representative secondary amine.

EXPERIMENTAL

Materials:

Acetate buffer, 4N, pH 5.5. For reproducibility, the buffer must be made up critically. To 100 ml H_2O add 136 g sodium acetate (NaOAc·3 H_2O) and stir on a water bath until the solution is homogeneous. Cool to room temperature, add 25 ml glacial acetic acid, then add H_2O to a total volume of 250 ml. The pH should be 5.51 \pm 0.03. The solution should be stored in a plastic container in a refrigerator.

Ninhydrin reagent (triketohydrindene). Dissolve 2.0 g ninhydrin and 0.3 ml hydrindantin in 75 ml 2-methoxyethanol by stirring. Add 25 ml of the acetate buffer solution and store in a lightproof glass container in

a refrigerator. Solutions should be used within 48 hours, after which fresh reagent must be made.

Phenethylamine standards. The amphetamine standard (20 mM) was prepared by dissolving 0.7370 g amphetamine sulfate in 100 ml deionized H₂O. The methamphetamine standard (20 mM) was prepared by dissolving 0.3714 g methamphetamine hydrochloride in 100 ml deionized H₂O.

Procedure:

A standard curve was determined following the method described in Moore and Stein⁸. Four spectrophotometric tubes were set up for each concentration and 1 ml amphetamine or methamphetamine were added to each tube after being appropriately diluted. The final dilutions used in the analysis were 2,4,8, and 10 mM for amphetamine and 4,10 and 20 mM for methamphetamine. One ml of the ninhydrin solution was added to each tube, which was then loosely capped and shaken for 10 seconds. The tubes were then placed in a boiling water bath at 1 minute intervals with timing started when the first tube was placed in the water. The tubes containing amphetamine were heated for 15 minutes and the tubes containing methamphetamine were heated for 1 hour. The tubes were then taken out sequentially at 1 minute intervals followed by dilution and quenching with 2 ml ethanol:H₂O (1:1) solution. The tubes were then cooled to room temperature and read spectrophotometrically at 570 nm against a reagent blank treated in the same way as above.

The same procedure was used to obtain data for the reaction of 10 mM concentrations of both amphetamine and methamphetamine as a function of time. Each tube was measured spectrophotometrically at 570 nm against a reagent blank.

RESULTS

The results of the reaction of ninhydrin with varying concentrations of amphetamine and methamphetamine were analyzed and the y-intercept, slope and correlation coefficient were computed for each. The reaction involving amphentamine has a y-intercept at an absorbance of 0.02, a slope of 0.07 and a correlation coefficient of 0.996. The reaction involving methamphetamine has a y-intercept at an absorbance of 0.07, a slope of 0.01 and a correlation coefficient of 0.979. The graphs of the data for both amphetamine and methamphetamine can be seen in Figures 3 and 4, respectively.

The results of the reaction of ninhydrin with a 10 mM solution of amphetamine and methamphetamine as a function of time were analyzed and the y-intercept, slope and correlation coefficient were computed for each. The reaction involving amphetamine has a y-intercept at an absorbance of 0.134, a slope of 0.008 and a correlation coefficient of 0.987. The reaction involving methamphetamine has a y-intercept at an absorbance of 0.006, a slope of 0.0007 and a correlation coefficient of 0.993. The graphs of this data for amphetamine and methamphetamine can be seen in Figure 5.

DISCUSSION

The mechanism of the reaction of ninhydrin with amines is generally considered to have been elucidated, and the kinetics of the reaction with amphetamine and methamphetamine follow the classical reaction. The kinetics of the reaction, however, do not explain the greater sensitivity of ninhydrin toward methamphetamine previously reported. It must be recognized, also, that there are several anomalies connected with the ninhydrin reaction. The reaction of ninhydrin with enamines, e.g., β -aminocrotonic acid, does not follow the classical reaction, nor can the

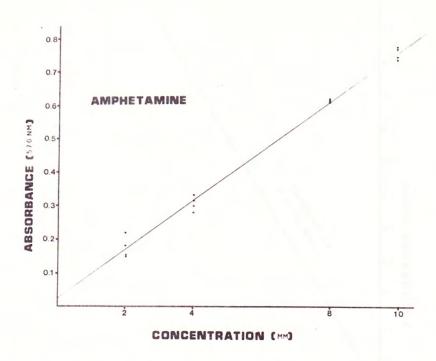


FIGURE 3. Ninhydrin reaction as a function of concentration of amphetamine

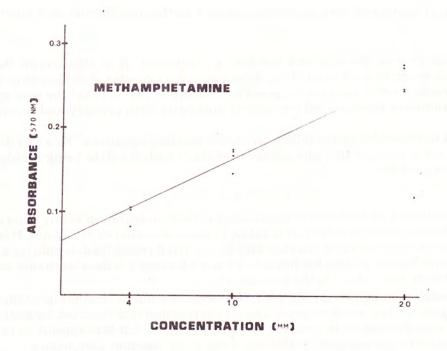


FIGURE 4. Ninhydrin reaction as a function of concentration of methamphetamine

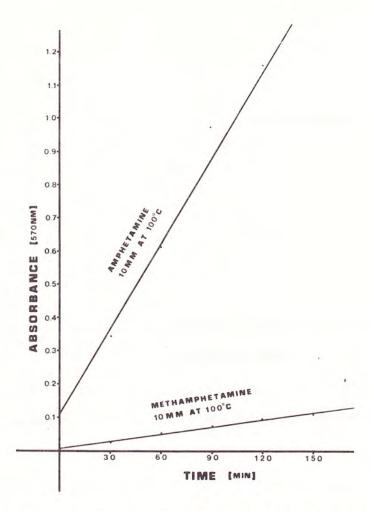


FIGURE 5. Reaction of ninhydrin with amphetamine and methamphetamine as a function of time

failure of aromatic amines to give the expected reaction to explained. It is also known that diamino and aminothiol acids, although reactive with ninhydrin, do not give the expected stoichiometric amounts of Rhuemann's Purple⁸. The present work is yet another point in evidence of the fact that the reaction mechanism is more involved than is assumed in the classical reaction of ninhydrin with primary and secondary amines.

Both amphetamine and methamphetamine follow zero-order reaction equations. This was determined by the fact that the data generated a straight line plot (linear over time) with the data being highly correlated. The equation for zero-order reactions is:

$$c - c_0 = k_0 t$$

where c_0 is the initial concentration at time zero (y-intercept), c is the concentration at any other time and k_0 is the slope. For the purpose of this paper, concentration is taken to mean absorbance at 570 nm. It is an easy step to convert absorbance to concentration utilizing the standard curve. The formula for determining absorbance when either time or concentration is known utilizes the formula y = mx + b where y is the absorbance at 570 nm, m is the slope, x is the concentration or time, and b is the y-intercept.

As expected from the postulated reaction mechanism, the secondary amine, methamphetamine, has a slower reaction rate than the primary amine, amphetamine. The slower reaction rate observed for methamphetamine is interesting in view of the greater sensitivity reported by Cashman *et al.*, but this appears to be a system where sensitivity and rate of reactivity are not manifestations of the same reaction parameters.

The standard curve regression lines plotted for both amphetamine and methamphetamine (Figures 3 and 4, respectively) show a deviation from Beer's Law, which stipulates that a plot of absorbance versus concentration should give a straight line *passing through the origin*. This deviation is probably the result of air oxidation, to which ninhydrin is highly susceptible. It should also be noted that a regression line is a compromise generated from different statistical considerations.

CONCLUSION

The reactions for both amphetamine and methamphetamine follow zero-order reaction equations since they produce a plot that is linear over time. Methamphetamine has a slower reaction rate which is consistent with the proposed reaction mechanisms reported in earlier works, but which is surprising in view of its greater sensitivity. It is apparent that the sensitivity of phenethylamines toward ninhydrin is a function of some other unspecified parameter or parameters.

REFERENCES

- l. Bastos, M., G.E. Kananen, R.M. Young, J.R. Monforte, and I. Sunshine, 1970, Clin. Chem., 16, 931.
- 2. Mule, S.M. Bastos, D. Jukofsky, and E. Saffer, 1971, J. Chromatogr., 63, 289.
- 3. Mule, S., 1969, J. Chromatogr., 39, 302.
- 4. Cashman, P.J., J.D. Beede and J.I. Thornton, 1979, J. Forens. Sci. Soc., 19, 137.
- 5. McCaldin, D., 1960, Chem. Rev., 60, 39.
- 6. Yamagishi, M. and T. Yoshida, 1954, J. Pharm. Soc. Japan, 74, 1042.
- 7. Johnson, A. and D. McCaldin, 1958, J. Chem. Soc., 1958, 817.
- 8. Moore, S. and W. Stein, 1948, J. Biol. Chem., 176, 367.
- 9. Freidman, M. and C.W. Sigel, 1966, Biochem., 5, 478.

LABORATORY AID A METHOD TO AID IN THE PRECISION OF TRIGGER-PULL DETERMINATION

by Frank Cassidy
Santa Barbara Regional Lab
(Reprinted from the DOJ TIELINE by permission.)

Our method for determination of trigger pull utilizes a 3-lb coffee can (as the weight recepticle) supported by a heavy cord which connects to the load-bearing member that contacts the trigger. The heavy cord is approximately three feet long. The weights are added as integral one-lb. or one-ounce units (Rob Cranston, when working at SBRL, packaged lead shot in small boxes for the one-lb units and small glass vials for the one-oz units.) The weights are carefully added to the coffee can until the hammer is actuated.

When adding the weights, great care and a steady hand are required to prevent addition of spurious momentum. When the load approaches the actuation level, this becomes especially critical. Recently, we have introduced a stabilizing technique that enables achieving greater precision by essentially eliminating the spurious momentum that occurs during the weight addition. This was achieved by placing a scissors-type laboratory support jack under the can. Prior to adding the weight, the tension on the supporting cords is removed by raising the jack under the can. Thus, when the weight is added no momentum is added. The jack is slowly lowered and the true load is transmitted to the trigger without being adversely affected by any spurious momentum.

The jacks are available from several lab supply houses, for example:

VENDOR	CATALOG NO.	PAGE	PART NUMBER
C.M.S.		1027	335-281
Fisher	81	1162	14-673-10
Sargent Welch	128	1271	S-79151

ATF CHARCOAL TUBE METHOD FOR EXTRACTING ACCELERANTS FROM ARSON DEBRIS

BY DARRYL R. TATE, SANTA BARBARA LAB (Reprinted from the DOJ TIELINE by permission.)

In June I had the opportunity of attending the Bureau of Alcohol, Tobacco and Firearms (ATF) Course in the "Detection and Identification of Accelerants Found in Arson Debris". Part of the course centered around the comparison of the four common methods used today in extracting accelerants from arson debris. These included steam distillation, heated headspace, solvent extraction and the ATF Charcoal tube method. The advantages, disadvantages, lower limits of detection and limitations of each method were compared. Table I is a summary of the findings on each technique.

Based on this information an analysis scheme was discussed and developed by the group to deal with arson samples coming into the laboratory. (See schematic) The sample is opened to determine what type of material is present (carpet, foam rubber, soil, etc) and whether an accelerant odor can be detected or not. If there is a strong odor of an accelerant, it is possible that heated headspace is sufficient to identify the sample. The Charcoal Tube Method should be used in all other cases except in the instance of a possible heavy petroleum distillate where solvent extraction would be the method of choice.

The conditions and equipment that the ATF laboratory uses are reprinted in Table II. It must be noted that ATF uses a Perkin Elmer Sigma 1 Gas Chromatograph with the Sigma 10 Data Station and Santa Barbara uses a Perkin Elmer 3920 GC. The conditions that they use cannot be duplicated on the PE 3920 so they were adjusted to fit our equipment. These conditions give good separation and resolution of peaks but every laboratory is cautioned to adjust their programs to suit their instrumentation and special conditions.

The charcoal tube method has many advantages. It is simple to set up and run and it is more sensitive than the other methods. Samples take very little preparation time and it gives you a collected sample for later analysis of further analysis by other techniques (IR, etc.) It has a greater recovery and it can capture a broader range of the light to heavy petroleum based distillates than the other methods. It also can distinguish between kerosene and diesel fuel (No. 1 and 2 fuel oils). It is not severely affected by the presence of water and it can be performed using materials that can be found around the laboratory or that are easily ordered. The disadvantages can be overcome by preparing samples in an area free of volatile vapors and by keeping the charcoal away from these vapors and the lid tightly closed. Overheating is not a problem if you make sure that the samples are not heated much over 100°C. (Overheating will result in a loss of the light fraction). ATF has developed an acid stripping method for removing the pyrolysis products by treating the carbon disulfide extract with acid but so far the results have been less than satisfactory.

The method employs the use of the following materials:

Activated Cocoanut Charcoal 50-200 Mesh (available from Fisher Cat. #5-690-B) Carbon Disulfide (Fisher C-573)

Cotton

5 3/4 inch disposable pipets

Paint cans (pt., qt., gal., sizes)

Brass & inch pipe to hose adaptors

00 rubber stoppers with one hole

Thermometor (Santa Barbara has a Markson model A-20 digital thermometer with a probe that works perfect!)

Heating mantle (connected to a Variac, optional)

Vacuum system (Santa Barbara uses a Cole Parmer Air Cadet Pump)

Silicone tubing (Cole Parmer #C-6411-74)

½ dram screw cap vials.

The figure (1) shows the materials used and how they are assembled to perform the method. In theory the volatilized accelerant is removed from the can by vacuum and trapped on an in-line charcoal tube. The accelerant is removed from the tube with carbon disulfide and analyzed.

Method

- 1. Evidence is placed in a sealed metal can having a special lid with a hose adaptor and a rubber stopper epoxied on. (These lids must be baked in the oven to remove all vapors prior to use). 3-4 holes are punched in the bottom of the can or above the water level if water is present.
- A vacuum line is attached to the top of the can and a thermometor is inserted into the one holed rubber stopper.
- 3. A disposable pasteur pipette is plugged at one end with a small amount of cotton. $2\frac{1}{2}$ inches of activated cocoanut charcoal * are added and the other end is plugged with cotton. This tube is placed in the vacuum line with the pointed end towards the vacuum.
- 4. The can is heated until the contents reach 100°C and held at that temperature for approximately 10 minutes. Vacuum is pulled throughout the entire heating cycle and also during a cool down period of about 5-10 minutes.
- 5. The tube is removed and extracted with approximately 2 mls of carbon disulfide into a $\frac{1}{2}$ dram screw top vial.
- 6. The extract is then injected into the GC or used on the IR.

So far the results have been excellent (see chromatogram) and Santa Barbara's laboratory aid, Monica Tokunaga, is now in the process of running standards through this method of all the common accelerants. Also, we plan to run the accelerants after weathering them to various degrees by using a hair dryer. Further research and results will be reported

^{*} According to the ATF laboratory the charcoal must initially be put in an oven at 300°C overnight to remove contaminants then you can place some in a smaller closed container for everyday use.

. TABLE I

TECHNIQUE	DISADVANTAGES	COMMENTS
STEAM DISTILLATION	Distilling time (1-4 hrs.)	Lower limit of detection: 50-100 µl/quart
	Loss of light fraction	
	Interference of distillation products	
HEATED HEADSPACE	Overall diminishment of all distillate fractions	Quick and easy
	Complete loss of heavy distillates past C13	Lower limit of detection: 10-20 µl/quart
	Swamping effect on light fraction	
	Less resolution of peaks	
	Pyrolysis products	
SOLVENT EXTRACTION	Large amount of expensive solvent needed	Great for heavy fraction
	Loss of light to medium fraction	Recovery of sub- strate varies with sample
	Dissolves dirt etc. that can clog and reduce life of column	Lower limit of detection .5 µl/ quart
CHARCOAL TUBE ABSORPTION—ELUTION	Possible contamination of charcoal	greater than 95% recovery
	Loss of very heavy fraction past Cl8	Lower limit of detection -3-1 µl/quart
	Overheating affects light fraction	
A.		

Pyrolysis products

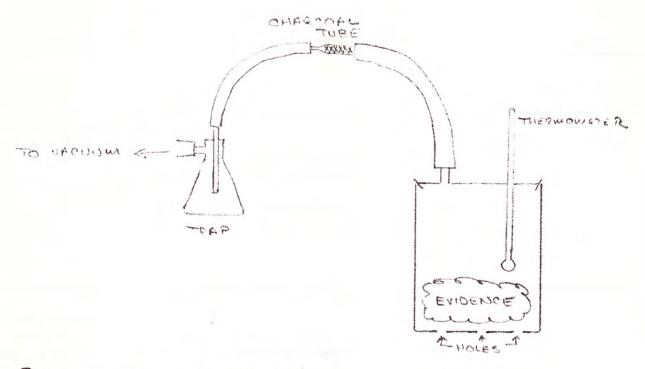


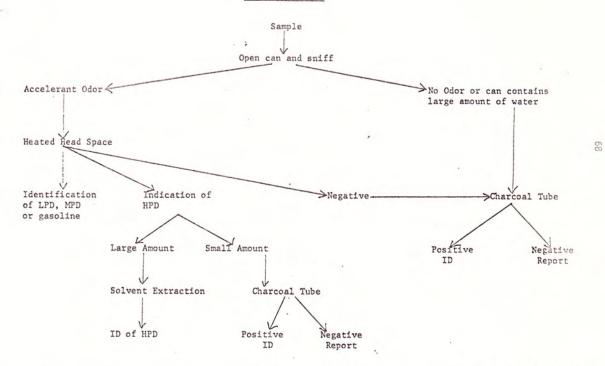
FIG. 1. CHARCOAL TUBE SETUR

TABLE II

GAS CHROMATOGRAPH CONDITIONS

	ATF	SANTA BARBARA
INJECTOR	250°C	250°C
DETECTOR	300°C	300°C
INITIAL OVEN TEMPERATURE	50°C	50°C
INITIAL HOLD	6 minutes	8 minutes
TEMPERATURE PROGRAM RATE	12°C/minute	16°C/minute
FINAL TEMPERATURE	250°C or 280°C	250°C or 280°C
FINAL HOLD	varies	4 minutes
COLUMN GAS FLOW	25 ml/minute	25 ml/minute
CHART SPEED	1 cm/minute	1 cm/minute
COLUMN	20 ft 1/8 inch I.D. Stainless Steel	10 ft 1/8 inch I.D. Stainless Steel
	3% SP-2100 Methyl Silicone on 80/100 Mesh Supelcoport	10% SP-2100 Methyl Silicone on 80/100 Mesh Supelcoport
ĠAS CHROMATOGRAPH	Perkin Elmer Sigma 1 with the Sigma 10 Data Station	Perkin Elmer 3920 Hewlett Packard 3380A Integrator Soltec Recorder





LPD = Light Petroleum Distillate

MPD = Medium Petroleum Distillate

HPD = Heavy Petroleum Distillate

