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Rules

RULES

Department of Agriculture
Bureau of Entomology and Plant Industry

Supplement to the Sweet-potato Weevil
Quarantine and Regulation

In accordance with the authority vested in the Louisiana Department of Agriculture of Part 2 of Chapter 12 of Title 3 of the Louisiana Revised Statutes of 1950, the Sweet-potato Weevil Quarantine and Regulation is hereby supplemented as follows:

III. Quarantined Areas

1. In the United States

   a. The areas hereby quarantined on account of the sweet-potato weevil shall be the portions of all states in which sweet-potato weevil infestations are known to occur, and so officially designated as quarantined or regulated areas, by the sweet potato quarantines of the states of Alabama, Florida, Georgia, Louisiana, Mississippi, Texas, and South Carolina.

2. In Louisiana

   a. Quarantined areas in Louisiana are hereby declared to be the entire parishes of Acadia, Allen, Ascension, Assumption, Avoyelles, Beauregard, Calcasieu, Cameron, East Baton Rouge, East Feliciana, Evangeline, Iberia, Iberville, Jefferson, Jefferson Davis, Lafayette, Lafourche, Livingston, Orleans, Plaquemines, Pointe Coupee, Rapides, Sabine, St. Bernard, St. Charles, St. Helena, St. James, St. John the Baptist, St. Landry, St. Martin, St. Mary, St. Tammany, Tangipahoa, Terrebonne, Vermilion, Vernon, Washington, West Baton Rouge, West Feliciana, and those parts hereinafter listed:

   Bienville Parish–Ward 4; that portion consisting of a one mile radius of and including the property of Enis Lowe, Section 12, R5W, T16N; that portion consisting of a one mile radius of and including the property of Henry Lowe, Section 13, R5W, T16N; that portion consisting of a one mile radius of and including the property of Mary Willis, Section 29, R4W, T15N; and that portion consisting of a one mile radius of and including the property of David Williams, Section 6, R4W, T14N;

   Caddo Parish–Wards 6 and 7; that portion consisting of a one mile radius of and including the property of Dr. Joe White, 115 Lucia Lane, Shreveport; that portion consisting of a one mile radius of and including the property of T. B. Boyter, Section 29, R14W, T18N; that portion consisting of a one mile radius of and including the property of Mrs. L. M. Laborde, Section 32, R13W, T16N; and that portion consisting of a one mile radius of and including the property of Tony Scarpinatto, Section 16, R13W, T16N;

   Caldwell Parish–that portion consisting of a one mile radius of and including the property of Ed Hilburn, Section 28, R3N, T14N; and that property consisting of a one mile radius of and including the property of Clifton Hilburn, Section 28, R3N, T14N;

   DeSoto Parish–that portion south of T15N;

   Jackson Parish–that portion consisting of a one mile radius of and including the property of Loretta Denton, Section 26, R4W, T16N; and that portion consisting of a one mile radius of and including the property of Lee Watkins, Section 11, R4W, T15N;

   Lincoln Parish–that portion consisting of a one mile radius of and including the property of James Kay, Section 13, R3W, T17N;

   Natchitoches Parish–that portion west and southwest of the Red River;

   Webster Parish–that portion of Ward 4 bounded on the west by Bayou Dorcheat, on the east by Highway 159, and on the south by Highway 80;

and/or such other area or areas as may hereafter be designated as quarantined areas by notice in the Register and Journal of the State of Louisiana by the State Entomologist, with the approval of the Commissioner.

b. Non-sweet potato areas shall be infested properties in the area north of Avoyelles and Rapides Parishes, east and northeast of the Red River line at Grant Parish, northeast of the Red River in Natchitoches Parish, north of the Natchitoches Parish line west of the Red River and north of the Sabine Parish line, and such other area or areas as may hereafter be declared non-sweet potato areas by publication in the Official Journal and the Louisiana Register by the State Entomologist, with the approval of the Commissioner.

The above supplement to the Sweet-potato Weevil Quarantine and Regulation shall be revised effective on and after June 20, 1977.

Richard Carlton, State Entomologist
Bureau of Entomology and Plant Industry

RULE

Department of Commerce
Radio and Television Technicians Board

Rule 10

Each member of the Examining Committee shall receive a per diem of thirty dollars for the performance of his duties while conducting examinations, and twenty-five dollars per day for any official meetings attended. He shall also be paid all necessary travel expenses and all actual and necessary subsistence expenses as set forth in the guidelines of the Division of Administration.

Nicholas J. Lapara, Administrator
Radio and Television Technicians Board

RULES

Board of Elementary and Secondary Education

Rule 4.01.50

Standards for Approval of Nonpublic Schools

Philosophy and Need

Nonpublic schools differ from public schools in philosophy, objectives, and approach. They come in many sizes and shapes and stress variety rather than uniformity. They develop as responses to particular educational needs within their community. They offer a broad range of learning options commensurate with the diversity of human needs, interests, and potentialities. All share a common concern for the individuality of students and for individualizing and personalizing the learning process. Each allows the student to make meaningful choices about his education.

Procedures for Approval of Nonpublic Schools

1. The nonpublic school board or governing body shall pass a resolution establishing the nonpublic school and setting forth its goals and objectives.

2. Nonpublic schools are designed to meet the needs of a specific group of students. Each nonpublic school will be evaluated on the basis of its stated goals and objectives. Evidence as to the attainment of each school's specific goals and objectives will be presented by the school to the State Board of Elementary and Secondary Education.

3. In addition, the basic goals and objectives of the nonpublic school will be evaluated by the nonpublic school system or governing body. The nonpublic school shall have an on-going evaluation of their school program with a written report to the State Board of Elementary and Secondary Education at least once every five years. This evaluation shall insure that the nonpublic school is maintaining a sustained curriculum or specialized course of study of quality at least equal to that prescribed for similar public schools.

4. It is recommended that the State Board of Elementary and Secondary Education establish a Commission of Nonpublic Elementary and Secondary Schools to evaluate these schools and to make

recommendations to the State Board of Elementary and Secondary Education as to approval or non-approval.

5. The Department of Education shall submit to the Board a yearly report evaluating nonpublic schools in accordance with the nonpublic school standards.

Criteria for Approval

Because of the diversity inherent in nonpublic schools, established public school standards are often neither appropriate nor fully applicable to their operation.

Standards for nonpublic schools should permit meaningful decisions to be made concerning the quality of their educational program and should insure an educationally sound environment.

1. Each nonpublic school shall develop and maintain a written statement of its philosophy and the major purposes to be served by the program. The statement shall reflect the individual character of the school and the characteristics and needs of the students it serves.

2. The educational program shall be designed to implement the stated goals and objectives. It shall be directly related to the unique educational requirements of its student body.

Faculty

A. Principal—The local governing body shall provide that the operation of the school be under the direction of a proven educational administrator. The principal shall hold, at least, a master's degree.

B. Instructional Staff—All members of the instructional staff shall have received a bachelor's degree from an accredited institution. They shall also have completed a minimum twelve semester hours of professional study, except that a beginning teacher shall have a two-year period in which to meet this twelve semester hour standard. The teacher shall be required to have a certificate or college major in the field of work for which the teacher is responsible during a major portion of the school day. A teacher may work in areas other than the major field for a period of time that is less than the major portion of the school day provided that he has earned at least twelve semester hours in each such area. Exception may be made for teachers in trades and in other special classes.

Professional and/or technical personnel may teach less than one-half of a school day (i.e. certified public accountant, doctor, college or university professor, lab technician, lawyer, etc.).

Those teachers not presently holding a bachelor's degree, but having taught for a period of at least five years and are satisfactory in their teaching performance may be retained. However, upon retirement or replacement, they must be replaced by a degreed teacher.

No secondary teacher shall be required to teach more than seven hundred fifty pupil-hours per week.

Teacher recognition as described in this section for nonpublic schools shall not constitute any automatic granting of public certification, nor shall experience teaching in this status be countable for public certification unless that person has received public certification.

Curriculum

A. Elementary—Nonpublic elementary schools shall devote no less than fifty percent of the school day to the skilled subjects, which are reading, language arts, and mathematics. The remainder of the school day may be devoted to such subjects as social studies, arts, religion, science, physical education, or other electives.

B. High School—The units required for graduation shall include the following:

English Must be any three of the four courses, English I, II, III, IV. (A course in basic reading may be available as an elective course to enable students who need it to take the three English units required.)	3 units
Health and Physical Education Each required unit must include thirty hours of health instruction.	2 units
Mathematics	2 units
Science	2 units

Social Studies	2 units

Two units in social studies shall be required. One unit must be in American history; and one unit must be civics or an equivalent course in citizenship education as approved by the State Department of Education.

Minimum total required	11 units
Electives	9 units
Minimum total required for graduation	20 units

Instructional Time Requirement

The daily program shall contain a minimum of 5 1/2 hours of instructional time. Schools on a continuous progress or modular scheduling program, where the constant is achievement and time becomes the variable must devote at least two hundred fifty minutes per week per basic subject area.

Enrollment and Attendance

1. Kindergarten age shall be defined as beginning at four years, eight months with a birth certificate required upon enrollment. The kindergarten program is informal in nature and planned to meet the developmental needs of young children with structured and unstructured learning activities.

2. The maximum enrollment allowed in any class or section shall not exceed thirty-five students except in certain activity type classes such as physical education, music, art, etc.

3. Children shall be accepted into first grade according to the standards established in the Compulsory School Attendance Law, (R.S. 17:222).

4. In order to be eligible for advancement to the ninth grade, an eighth grade student must have successfully completed the eighth grade skill subjects and must demonstrate a functional literacy achievement level in reading.

5. Elementary children must be present a minimum of one hundred forty days to be eligible to receive credit for the courses taken. Exception can be made only in the event of extended personal illness as verified by a physician or at the discretion of the principal.

Secondary students must be present a minimum of seventy days per semester to be eligible to receive credit for the course taken. Exception can be made only in the event of extended personal illness, verified by a physician or at the discretion of the principal.

Library

A. Elementary schools with a centralized library shall have the services of at least a trained part-time librarian.

B. High schools with three hundred fifty students or more shall have the services of a full time graduate librarian. High schools with less than three hundred fifty shall have the services of a part-time graduate librarian.

C. High schools shall have at least ten volumes per student and an adequate number of magazines and periodicals.

D. Library expenditures must not be less than two dollars per student per year.

Physical Plant

School facilities shall assure that the health and safety of those served by the school are properly safeguarded. Applicable local and State regulations shall be followed.

Summer School

1. The minimum attendance for a student to receive credit or pass a subject shall be as follows:

   a. Seventy hours for one-half unit new credit
   b. Forty-seven hours for removal of one-half unit deficiency

2. The local system may impose a more strict minimum attendance policy.

3. For the acceptance of summer school credit, the receiving principal must approve the summer school course(s) in advance.

4. Summer schools with seven or more teachers must have a certified principal.

School Reports

1. Annual School Reports

At the end of the first reporting period during the session, the principal shall forward a report through the nonpublic superintendent's or administrator's office, to the State Department of Education, on forms provided for that purpose. This must include the name, degree, and experience of each teacher employed; the daily schedule of classes; the registration of students in elementary grades; the high school subjects taught. This report shall be signed by the principal and the nonpublic school superintendent or administrator. One copy will be filed in the nonpublic school superintendent's or administrator's office and the other in the principal's office.

2. Reports of High School Credit

Before a student may graduate from a nonpublic high school a certificate of high school credits shall be submitted to and approved by the Director, Bureau of Secondary Education, State Department of Education.

3. Reports to Local Public School Superintendents

Student registration, gains, losses, enrollment, and attendance shall be recorded on report forms furnished by the State Department of Education. A complete form must be sent to the local public school superintendent's office and a copy filed in the principal's office. The principal shall send the session report to the local public school superintendent at the end of the school year. A copy of this report shall be filed in the principal's office, the local public school superintendent's office, and shall be available to the State Department of Education upon request.

* * * *

Rule 5.00.50c

Adoption of policy limiting consulting fees for applications for Federal funds to five percent of the grant.

* * * *

Rule 3.01.52c

(This policy replaces policy presently in effect.)

Full-time secondary certified teachers in schools including grades 7-12 (or any combination thereof) may be allowed to teach a maximum of two periods in one subject out of their field of certification if they have earned twelve hours in that subject. This allowance shall not apply to the teaching of English I, II, III, American History, Civics, General Science, or Free Enterprise. Secondary teachers shall not teach below the seventh grade level. This requirement shall become effective for the school year 1978-79.

* * * *

Rule 3.01.70v (10)

No out-of-state graduate credit will be accepted by the Bureau of Higher Education and Teacher Certification for salary increment purposes, or for satisfaction of any teacher certification requirements if such credit is earned in a program conducted in a state other than that in which the granting institution is located. Any graduate credit earned at an out-of-state university, housed in state, which is acceptable in writing by a Louisiana graduate dean or the State Department of Education will not be subject to this provision.

Earl Ingram, Director
Board of Elementary and
Secondary Education

RULES

Department of Health and Human Resources
Board of Dentistry

(Editor's Note: The following rules were adopted by the Louisiana State Board of Dentistry on June 3, 1977, to become effective August 1, 1977.)

1. Notice of Employment of Dental Hygienists

Each dentist shall inform the Louisiana State Board of Dentistry of the name and license number of the dental hygienists employed by him or her. The Board must be notified of any change in employment within ten days of such change.

Anthony J. Milazzo, Jr.
Secretary-Treasurer
Board of Dentistry

RULE

Department of Health and Human Resources
Office of Family Services

The Department of Health and Human Resources, Office of Family Services, has adopted the following policy and procedures in the Medicaid Program relating to the method of reimbursement for outpatient hospital services. The primary objective of this policy is to make Medicaid payments more equitable to the Title XIX providers of outpatient hospital services. The adopted policy and procedures are as follows:

Reimbursement for outpatient hospital services will be based on costs or charges, whichever is lower. Hospital cost reporting periods beginning on or after July 1, 1977, will be adjusted to cost at their year-end closing.

William A. Cherry, M.D., Secretary
Department of Health and Human Resources

RULE

Department of Health and Human Resources
Office of Family Services

The Department of Health and Human Resources, Office of Family Services has adopted rules and regulations regarding revised Income Standards and Basis of Issuance in the Food Stamp Program effective July 1, 1977, in accordance with Federal regulations as specified in Federal Register, Volume 42, Number 85, Tuesday, May 3, 1977, pages 22,356-8. The revisions provide food stamp recipients with a cost of living increase.

Copies of the revised Income Standards and Basis of Issuance may be obtained without cost at the following address: Food Stamp Program, Office of Family Services, 333 Laurel Street, Room 301, Baton Rouge, Louisiana 70804, telephone number 389-2631.

William A. Cherry, M.D., Secretary
Department of Health and Human Resources

RULES

Department of Health and Human Resources
Office of Health Services and
Environmental Quality

(Editor's Note: Exhibits B, D, E, G, H, I, J, and K are samples of forms, logs, and certificates. They are not reproduced here, as per R.S. 49:954.1C)

Rules and Regulations for
Chemical Test for Intoxication

The Louisiana Department of Health and Human Resources, Office of Health Services and Environmental Quality, having published notice of its intention to adopt certain rules and regulations as amended to date pertaining to breath and blood alcohol analysis methods and techniques pursuant to R.S. 32:663, hereby adopts and publishes the following rules and regulations pertaining to the performance of chemical tests for intoxication.

1. The Office of Health Services and Environmental Quality, Louisiana Department of Health and Human Resources, is the successor to, and acts as, the State Department of Health and/or Department of Health, R.S. 46:1751 et seq.

2. After the Louisiana Department of Health and Human Resources, Office of Health Services and Environmental Quality, Bureau of Laboratories, has approved a prototype breath testing device as an acceptable model for chemical analysis in breath alcohol testing it shall be necessary for each individual instrument of the approved model to be given a numbered tag and to be checked out and approved for use by the State Police Crime Laboratory at least once every four months, and a machine recertification form shall be maintained for each machine in the State Police Crime Laboratory, as prima facie evidence of the operating performance of the device. A copy of this certificate shall be filed with the clerk of the applicable court in the respective parishes in which each device is used for breath testing.

   Any manufacturer of any apparatus, device or equipment made for the purpose of analyzing the alcoholic content of breath, may request the Office of Health Services and Environ-

mental Quality, Bureau of Laboratories, to approve such apparatus, device, or equipment. The Bureau will consider said request upon submission of such information, instructions for use, exemplars, and other pertinent data as the Board may request.

3. Analysis of breath specimens for the determination of the alcohol content therein will be performed with the Photo-Electric Intoximeter Model No. 400 single cylinder instrument manufactured by Intoximeters, Inc., St. Louis, Missouri, which has the approval of the Louisiana Department of Health and Human Resources, Office of Health Services and Environmental Quality, Bureau of Laboratories. The Photo-Electric . . . Model No. 400 single cylinder, is an approved technique or method for the performance of chemical tests for alcoholic influence.

4. The procedure for such analysis shall include the following:

   A. General observation of the subject for a period of twenty minutes prior to testing whereby the subject shall not have ingested alcohol, alcoholic beverages, regurgitated, vomited, or taken anything by mouth.

   B. The operator conducting breath analysis shall conduct such analysis in accordance with the "photo-electric intoximeter check list" which contains but is not limited to the following (See Exhibit A):

      (1) Completing the information section concerning such things as name of subject, time, witness, arresting and testing agency, instrument number and location, and State Police Crime Laboratory machine certification tag number.

      (2) A calibration check whereby the calibration of the instrument is checked by using a set of standard ampuls which accompany each instrument. A standard ampul of known value is used whereby the reading must be within the given range to show the calibrating section of the instrument is working properly.

      (3) Preparation of the instrument whereby temperature is checked and the ampul to be used in such analysis is checked to show it is within a certain tolerance plus or minus .010g%. This is to insure a good ampul will be used in the analysis.

      (4) A systems blank by which the instrument is shown to be free of contamination. Limitations here will be from +.010g% thru -.020g% whereby corrections from here will be made to produce the final reading.

      (5) Sample collection whereby the sample is taken and the twenty minute observation period is checked off.

      (6) Alcohol determination section whereby the instrument is flushed, scale zero checked and final reading taken. The ampul will be discarded after analysis since preservation will yield erroneous results after the ampul is opened, used in analysis, and exposed to continuous light. This ampul also contains acid which is very corrosive and may cause injury or damage if not properly disposed of.

      (7) Breath specimens collected for analysis should be substantially in equilibrium with pulmonary arterial blood, with respect to alcohol. That is, it should be essentially alveolar in composition.

      (8) Procedures for breath control analysis for the indirect determination of the blood alcohol concentration should include the following controls in conjunction with the testing of each subject.

         a. Continuous observation of the subject for at least fifteen minutes prior to collection of the breath specimen, during which period the subject must not have ingested alcohol, regurgitated, or vomited.

b. A system blank analysis.

(9) Results of analyses of breath for alcohol shall be expressed in terms of percent W/V (grams per deciliter) that is, grams of alcohol per 100 milliliters of blood, rounded downward to the second decimal place; for example 0.237 g/dl found should be reported as 0.23 g/dl or 0.23 percent.

(10) The quantity of breath analyzed for its alcohol content shall be established only by direct volumetric measurement, or by collection and analysis of a fixed breath volume at constant known temperature.

C. After each test the results will be recorded in the intoximeter log book (See Exhibit B). A copy of which is to be sent to the State Police Crime Laboratory at the end of each month and a copy to be retained at the testing agency.

D. A chemical test for intoxication of either blood or breath shall be administered after the arrest but within two hours of the violation, but the person arrested, having refused the test in writing properly acknowledged by two witnesses, or orally in the presence of two witnesses, shall not be administered the test, having once refused.

5. Each lot of ampuls shall be certified at the factory by the manufacturer as to their standard of quality. This certificate shall be prima facie evidence as to the standard of quality of the ampul.

6. Maintenance checks will be performed on a routine basis at least once every four months, by the Louisiana State Police Crime Laboratory. Items to be checked shall be but not limited to the following:

A. Each lot of ampuls shall be spotchecked for performance.

B. Clean instrument.

C. Calibration check of standard ampuls.

D. Running of a known alcohol solution in which results shall be within plus or minus .010g% or the known alcohol value. (See Exhibit C).

E. In the event any repair work is needed, it will be recorded in detail (See Exhibits D & E).

Repair work will be performed by technicians working for the Applied Technology Section of the Louisiana State Police Crime Laboratory who are certified by the Louisiana Department of Health and Human Resources, Office of Health Services and Environmental Quality, Bureau of Laboratories, to perform such maintenance. The Applied Technology Section of Louisiana Police Crime Laboratory shall have the authority to instruct other individuals to perform such maintenance (Exhibit F). Upon satisfactory completion of such training the individual shall be certified to perform maintenance by the Louisiana Department of Health and Human Resources, Office of Health Services and Environmental Quality, Bureau of Laboratories.

Records covering maintenance, etc., of the P.E.I. (photo-electric intoximeter) instrument will be kept by the Louisiana State Police Crime Laboratory.

7. Qualification for the certifications of individuals to conduct breath test analysis are as follows:

A. Employee of a Louisiana law enforcement agency or Federal law enforcement agency.

B. At least eighteen years of age.

C. Resident of the State of Louisiana at time of application.

D. Graduation from a State-accredited high school or satisfactory passing of the "general educational development test" or equivalent educational background.

E. Successful completion of a forty-hour operator's training course conducted by the State Police Crime Laboratory, or any other course approved by the State Police Crime Laboratory. Course material to be covered will be taken from

"Chemical Test for Intoxication Training Manual" (See Exhibits H & G).

F. To successfully complete the training course and become certified, the operator must:

(1) Obtain a seventy-five percent score on the written examinations covering course material.

(2) Obtain a seventy-five percent score on the actual operation of the P.E.I. and practical test (running of unknown solutions). Written test and practical test will be made up by instructors of the Louisiana State Police Crime Laboratory.

(3) All testing results will be recorded on the "chemical test for intoxication progress record" (See Exhibit I). One copy to be retained by the Crime Laboratory and one copy to be forwarded to the Office of Health Services and Environmental Quality.

G. Qualifications for certification of instructors will be as follows:

(1) Certified as a P.E.I. operator or t certified on any other approved instrument.

(2) Attendance of an additional forty-hour course approved by the Office of Health Services and Environmental Quality.

(3) Involved in a chemical testing program approved by the Louisiana State Police Crime Laboratory.

8. Upon determining the qualifications of individuals to perform such analysis and after submitting an application for certification (Exhibit J), the Office of Health Services and Environmental Quality may issue permits which shall be effective for a period of two years from the date inscribed thereon and the Bureau of Laboratories, Office of Health Services and Environmental Quality, shall keep a record of all permits and ready reference to the expiration of all certificates issued.

A. Permits (See Exhibit K) may be renewed after a refresher course, given by the Louisiana State Police Crime Laboratory or any other designated agency.

B. In addition to being certified on the P.E.I., an operator may also attend a specified course for certification on any new instrument that is approved by the Office of Health Services and Environmental Quality.

9. All persons seeking to be authorized to conduct blood analysis shall:

A. Make application to the Office of Health Services and Environmental Quality for permit.

B. Have a bachelor of science in chemistry, physics, biology, zoology, medical technology, or a related field.

C. Conduct proficiency testing set up by individual laboratories.

D. Be employed in either a crime laboratory, medical laboratory or analytical laboratory, and have at their access necessary instruments and equipment for analysis of blood for alcoholic content.

10. The methods approved for blood-alcohol analysis of blood are:

A. Gas Chromatography

(1) Headspace sampling with internal standard (See for example Exhibit L).

(2) Direct injection with internal control (See for example Exhibit M).

B. Distillation Method (See for example Exhibit N).

C. Permits shall be effective when issued for a period of five years from the date inscribed thereon. Permits may be renewed by making application to the Office of Health Services and Environmental Quality.

D. (1) Procedures shall include the follow-

ing controls in conjunction with each batch of samples analyzed:

(a) A system blank analysis.

(b) Analysis of a suitable reference or control blood sample of known alcohol content within the range of 0.01 to 0.30 g/dl; the result of which analysis must coincide with the known blood alcohol value of the reference specimen within ±0.01 g/dl if validity is to be assigned to the results for the batch analyzed.

(2) Replicate analyses shall be performed in order to minimize the possibility of undetected errors.

(3) Results shall be expressed in terms of percent W/V (grams per deciliter) that is, grams of alcohol per 100 milliliters of blood, rounded downward to the second decimal place; for example 0.237 g/dl found shall be reported as 0.23 g/dl or 0.23 percent.

(4) Analytical procedures for determining alcohol in blood shall meet the following performance requirements:

(a) The accuracy and sensitivity of the procedure shall be such as consistently to attain results within ±0.01 g/dl of the known value over the range of 0.00 to 0.30 g/dl in analyses of appropriate reference materials of known ethyl alcohol concentration.

(b) The precision of the procedure shall be such as consistently to attain a standard deviation not greater than ±0.003 g/dl in replicate analyses.

(c) The blank values yielded by the procedure in analyses of alcohol-free blood specimens consistently shall be not greater than 0.01 g/dl.

(d) The specificity of the procedure shall be adequate and appropriate for the analysis of biological specimens for the determination of the blood alcohol concentration in traffic law enforcement and highway crash investigations:

(i) Procedures for the analysis of biological specimens from living subjects shall respond only to ethyl alcohol and the other lower aliphatic alcohols and should not be susceptible to significant unrecognized interference by other substances.

(ii) Procedures for the analysis of postmortem biological specimens shall respond only to ethyl alcohol and shall not be susceptible to significant unrecognized interference by other substances.

11. Blood drawn for the purpose of determining the alcoholic content therein shall have been taken with the contents of the "B-D Blood Alcohol Kit" No. 4990 or No. 4991 for postmortem determination (manufactured by Becton-Dickinson Division of Becton, Dickinson and Company, Rutherford, New Jersey), or by a similar blood collection kit approved by Louisiana Department of Health and Human Resources, Office of Health Services and Environmental Quality, Bureau of Laboratories. "B-D Blood Alcohol Kits" or similar blood collection kits as approved will be made available to all law enforcement agencies by the Louisiana State Police Crime Laboratory.

12. Because of various problems in the interpretation of the results of analysis of urine for alcohol which cannot be readily overcome in law enforcement practice, urine analysis to determine equivalent alcohol concentration in blood is discouraged. Chemical tests of blood or breath are preferred.

Exhibit A
PHOTO-ELECTRIC INTOXIMETER CHECK LIST

Subject Tested _____ Drivers License No. _____

Date of Birth _____ Race _____ Sex _____ Ampul Lot No. _____

Operator _____ Date _____ Time* _____

Witness _____ Certification Tag No. _____

Arresting Agency _____ Testing Agency _____

Machine Location _____ Machine No. _____

FIRST SECTION: CALIBRATION CHECK

1. Both power switches on ☐. Galvanometer mechanically zeroed ☐.
2. Standard ampul of 0.000 g% value removed from case, wiped clean, shaken and placed in sample well. Scale set at zero. Button depressed and needle brought to center by means of KNOB K4. ☐.
3. Standard ampul of _____ g% value removed from case, wiped clean, shaken and placed in sample well. Standard ampul read: _____ g%.

SECOND SECTION: PREPARATION OF INSTRUMENT

4. Temperature in green area (105-110° F or 40.5-43.3° C) ☐
5. Sampling assembly mounted on vent and valve to Position I ☐
6. With scale set at 0.000 g% and REFERENCE ampul in sample well, button was depressed and needle brought to center by means of KNOB K4 ☐.
7. Stock ampul gauged, opened, wiped clean and placed in sample well. ☐
8. Stock ampul read: _____ g% (zero correction; note plus or minus limits: + .010 to -.010)
9. New and clean bubbler tube attached and inserted into ampul. New and clean mouthpiece attached to sampling assembly. ☐

THIRD SECTION: SYSTEMS BLANK

10. Valve to POSITION IV ☐; rod down ☐
11. Ampul read (bubbler partly withdrawn): _____ g% (Final correction). (Note plus or minus limits: +.010 to - .020)

FOURTH SECTION: SAMPLE COLLECTION

12. Shifted sampling assembly to take sample, bubbler tube reinserted, valve to POSITION III ☐.
13. Subject under observation 20 minutes and nothing taken by mouth ☐.
14. Breath sample was obtained according to operating instructions and the accepted sample met the following requirements: A deflated waste bag was used ☐. Sequence: waste bag filled ☐; indicator rod rose steadily ☐; rod fully up when valve was turned to POSITION IV ☐. (record time in space above, end of line 3*).

FIFTH SECTION: ALCOHOL DETERMINATION

15. Rod down ☐. Bubbler tube removed and discarded ☐.
16. At this point the instrument was flushed by turning valve to POSITION I to fill the cylinder ☐; then to POSITION II to discharge cylinder through sampling assembly ☐; then back to POSITION I to fill cylinder ☐; then to POSITION IV to flush delivery tube ☐.
17. First reading of ampul: _____ g%.
18. Scale zero checked with REFERENCE ampul (same as Item 6) ☐. If it has changed, reset with KNOB K4. Re-read the test ampul 3-5 minutes after Item 15. Second reading of ampul _____ g%.
19. Power switches off and sampling assembly stored ☐. Ampul discarded ☐.

RESULTS:

_____ g%

Second reading of ampul (Item 18): _____ g%
Final correction (Item 11): _____ g%
BLOOD ALCOHOL CONCENTRATION: _____ g%

DPSSP 4616 (6/76)

Exhibit C

Photo-Electric Intoximeter Machine Recertification Form

Date_____ Instrument No. _____ Technician _____

City_____ Time of Arrival _____

Agency: L.S.P. Troop_____ Sheriff's Department_____ City Police_____

Check List

Is machine kept covered? Yes_____ No
Was machine sealed? Yes_____ No
Are ampuls kept covered? Yes_____ No

1. A.C. Power Source_____ OK
2. Condition of Power and Lamp Switch_____ OK
3. Condition of P.E.I. dirty etc._____ OK. If not what _____
4. Warm-up time approximately fifteen minutes_____ OK
5. Thermometer_____ OK
6. Colorimeter_____ OK
7. Lens and light bulb wiped clean_____ OK
8. Sampling Assembly_____ OK
9. Mechanical zero and galvanometer _____ OK
10. Galvanometer_____ OK
11. Standard Ampuls_____ OK. Check for tolerance _____ OK
12. Alcohol simulator test – run known standard _____
    Known reading_____. Instrument read _____
    Tolerance ± .010 g%, if adjustment necessary
    Describe _____
13. Valve_____ OK 105 cc cylinder_____ OK
14. Interior of instrument checked for plumbing connection, wiring, acid spills, etc._____ OK

Supply issue: list all supplies left at agency; if adequate indicate with check.

Operator check list _____
Alcoholic influence form_____
Rights form_____
Refusal form_____
Intoximeter log book_____
Use this space for any additional notes:

Ampuls _____
Waste bags _____
C.E.B. bulbs_____
Plastic cover _____
Tees or tubing _____

_____
Signature of Technician

Exhibit F

Qualification of Persons Conducting Repair Work on the Photo-Electric Intoximeter

Persons who perform repair work on the Photo-Electric Intoximeter shall have spent no less than eighty hours covering, but not limited to, the following:

1. Proper cleaning of the 105 cc cylinder and piston.
2. Proper cleaning of the multi-port valve.
3. Proper care, cleaning, and painting of the cabinet.
4. Checking the calibration and trim of the scale.
5. Detecting switch trouble and replacing button.
6. Replacing of power and lamp switches.
7. Temperature regulation.
8. Heater replacement.
9. Air pump and plumbing replacement.
10. Repair and replacement of fan motors.
11. Remaking of standard ampuls.
12. Proper cleaning, painting and repairing of the colorimeter.
13. Checking of the photocells.
14. Detecting any malfunction of galvanometer and mechanical zero.
15. Wiring and circuit connections.
16. Neutralization of any acid spills.

* * * *

Exhibit L

Quantitative Gas Chromatographic Determination of Blood Ethanol by Headspace Sampling With Internal Standard

Instrumentation:

1. Chromatograph: A Hewlett-Packard 700 gas chromatograph, or similar instrument, is operated as a single flame instrument. The flame detector is operated at a temperature of approximately 250° C. Cylinders of hydrogen and compressed air are used. The hydrogen flow is set at a rotometer reading of 3 at 18 psi and air adjusted to give optimum response with stability. Cylinder nitrogen is used as carrier gas, set at a rotometer reading of 3.5 at 50 psi. The column, a 4' x ¼" stainless steel tube, is maintained at approximately 180° C. The column is packed with 8 g of Porapak Q originally conditioned at 200° C overnight. The injection port is maintained at a temperature of approximately 170° C. The electrometer is normally set at a range of 1, attenuation X 20.

2. Integrator. The electrometer signal is connected directly to a Hewlett-Packard 3370A Digital Integrator set at the following parameters:

Up Slope Sensitivity	0.01 mV/min
Down Slope Sensitivity	0.01 mV/min
Baseline Reset Delan	infinity
Peak Summation Level	1,000 mV
Front Shoulder Control	OFF
Rear Shoulder Control	1,000 mV
Recorder Presentation	1 mV

Noise suppression is adjusted according to need for the particular batch of samples being analyzed.

3. Recorder: The duplicated signal from the integrator is connected to a Tracor, Westronics model MT, 12 inch, 1 millivolt recorder, used at a speed of ½ inch per minute.

4. Calculations: A Wang 700A/701 Programmable Calculator with output writer is used for analysis of the data generated by the integrator. The program is written so that calibration data for known blood alcohol concentrations are fitted (by the method of least squares) to the line

$$\text{CONC} = a_1 \frac{R\,(\text{EtOH})}{R\,(\text{INT STD})} + a_0$$

The correlation coefficient is also calculated:

$$r = \frac{n\Sigma xy - \Sigma x \Sigma y}{\sqrt{[n\Sigma x^2 - (\Sigma x)^2]\,[n\Sigma y^2 - (\Sigma y)^2]}}$$

and the Standard Error of Estimate:

$$S_{y \cdot x} = \frac{1}{n}\sqrt{n\Sigma y^2 - (\Sigma y)^2 - \frac{(n\Sigma xy - \Sigma x \Sigma y)^2}{n\Sigma x - (\Sigma x)^2}}$$

Individual (x,y) data points are retained and plitted digitally. The regressed concentrations and estimated standard deviations for each are also calculated and tabulated for comparison with data. Estimated Standard Deviations are calculated by the formula:

$$S_y = S_{y \cdot x} \sqrt{1 + 1/n + x^2/\Sigma x^2}$$

The format of the calibration printout is given as figure 1.

The slope and intercept of the calibration line are retained in calculator memory and used for solving unknown blood alcohols by solving into the regression equation. A printout of each such calculation is generated for records. Samples may be calculated at any desired level of replication. (Normally, duplicates of each unknown blood are determined.) For the purpose of reporting, the lowest value obtained is truncated at the second decimal place. The format of this printout is shown in figure 2.

5. Glassware: Class A glassware is used throughout. Pure alcohol is measured from a burette. Solutions are measured by transfer pipet. Blood is measured by Ostwald-Folin pipet.

Procedure:

1. Alcohol Stock Solution: Alcohol standards are prepared in terms of grams per 100 cubic centimeters in accordance with Louisiana law. Absolute ethanol from a freshly opened bottle is used. For the purpose of preparing calibration solutions, the alcohol is considered 99% pure. This figure was derived from studies of specific gravity of the alcohol after opening the bottle. Overestimation of the purity of the alcohol results in overestimation of the unknowns. An alcohol solution of 50 g/l is prepared by measuring

$$\frac{50}{0.79 \times 0.99} = 63.9 \text{ ml}$$

into a 1000 ml volumetric flask. The volume is made to the mark with water, allowed to equilibrate at room temperature, and made again to the mark if necessary. This 5 g% solution is then dispensed into 20 ml glass ampuls and rapidly sealed by fusion. The entire liter quantity is thus preserved for use as needed. A random sample may be taken for oxidative analysis as described below.

2. Blood Standards: Citrated whole blood obtained when out of date for transfusion is used. One milliliter of the stock alcohol solution diluted to 100 c.c. with blood will provide a blood of 0.05 g% concentration. Multiples of this volume are used to prepare a calibration curve with values of 0.05, 0.10, 0.15, 0.20 and .25 g%.

3. Working Standards and Unknowns: Serum bottles of 30 c.c. capacity with rubber septum stoppers are used to contain the working standards. These are prepared by adding 3 ml of a sodium chloride solution approximately 25 g%. The solution is evaporated overnight in an oven, leaving a residue of 3/4 g NaCl. Exactly equal volumes of internal standard and blood are delivered into the dry bottles, which are immediately stoppered. (Ordinarily, 1.00 ml is the volume used.) *The internal standard is prepared by diluting 2 ml of 1-propanol to a liter with water.* The exact concentration of this solution is not important, but must be constant for the calibration curve and the batch of unknowns calculated from it. The 0.2 vol% concentration was chosen to give a peak height roughly equal to that given by the average alcohol determined (0.18 g%). The combined blood and internal standard are mixed by rotating the bottle on its side, care being taken not to allow any blood to contaminate the stopper. A hypodermic needle is used to bleed the headspace to atmospheric pressure, and the samples are allowed at least 20 minutes to come to equilibrium. If working standards are prepared in this manner and kept refrigerated while not in use, they may be kept for several weeks, removing as much as 10 ml of headspace daily without significant change in the ratio of ethanol/propanol in the samples. In practice, the working standards are not kept longer than one week.

4. Sampling: The sample is taken with an ordinary 3.0 c.c. disposable plastic syringe fitted with a 1.5 inch, 22 gauge needle. The syringe is filled to the 1.5 c.c. mark with air which is injected into the bottle being sampled. The plunger is then pumped vigorously 5 or more times and a 1.5 c.c. sample withdrawn. The volume is adjusted to 1 ml and injected swiftly and smoothly into the injection port of the chromatograph. The integrator is started immediately. Five 1 c.c. rinses with room air have been found sufficient to completely remove all traces of ethanol from the syringe.

* * * *

Calibration Data:

	R (EtOH)	R (INT STD)	RATIO	CONC
1	3011.00	16190.00	.1859	.050
2	4326.00	12210.00	.3542	.100
3	5534.00	9851.00	.5617	.150
4	8935.00	11990.00	.7452	.200
5				

Plot:

.050 .
.100 .
.150 .
.200 .

CONC = .2648 X Ratio + .002

r = .99927
Std Error of EST = .00212
Y Calc:

	RATIO	CONC	STD DEV
1	.185	.051	.00240
2	.354	.096	.00248
3	.561	.151	.00264
4	.745	.200	.00283

CONC = .2648 X Ratio + .002

r = .99927
Std Error of EST = .00212

R (EtOH)	R (INT STD)	RATIO	CONC	STD DEV
Blood of: John Doe				
8123.00	16350.00	.4968	.1342	.00259
6325.00	12620.00	.5011	.1354	.00259
RESULT: .13				
Blood of: Boyd Conners				
9615.00	15300.00	.6284	.1691	.00271
10150.00	16450.00	.6170	.1661	.00270
RESULT: .16				
Blood of: Jane Doe				
1546.00	12210.00	.1266	.0362	.00238
2651.00	14180.00	.1869	.0522	.00240
RESULT: .03				

* * * *

Exhibit M

Quantitative Gas Chromatographic Determination of Blood Ethanol By Direct Injection With Internal Control

Instrumentation:

(Note: All operating parameters should be adjusted for optimum performance as judged by response and linearity of calibration curve.)

1. Chromatograph: A Hewlett-Packard 7610 gas chromatograph or similar instrument is operated as a single column instrument. The flame detector is operated at a temperature of approximately 300°C. Cylinders of hydrogen and compressed air are used. The hydrogen flow rate is set at about 25 cc per minute, air flow rate is set to about 500 cc per minute. The column, ¼" X 4' glass tube, is maintained at about 170°C. The column is packed with Porapak Q, 80/100 mesh. (Glass wool packing is placed at both entrance and exit of the glass column. Observation of the glass wool after 20-30 injections of blood will indicate the need for changing. Performance of the column will indicate need for changing the Porapak Q.) The helium carrier flow rate is adjusted to give a retention time for ethanol of not less than one minute. The injection port is maintained at approximately 140°C. (The temperature is intentionally low to avoid, as much as possible, decomposition of blood in the needle of the syringe.) The electrometer is set at a range and attenuation adequate to give a full scale ethanol deflection of the recorder from injection of the 0.50 g% standard with internal control. This attenuated signal is connected to one pen of a dual pen recorder (see below).

2. Integrator: The unattenuated electrometer signal is connected directly to a Hewlett-Packard 3370B digital integrator set at the following parameters;

Noise Suppression	maximum
Up Slope Sensitivity	0.03mV/min
Down Slope Sensitivity	0.3 M
Baseline Reset Delay	zero
Area Threshold	1000 mV/min
Front Shoulder Control	on
Rear Shoulder Control	1000mV
Recorder Presentation	100mV

The 100 mV (fullscale) signal from the integrator is connected to the recorder.

3. Recorder: The recorder used is a Hewlett-Packard 12 inch, 1 millivolt dual pen model used at a chart speed of ¼ inch per minute.

4. Glassware: All volumetric glassware used is Class A Pyrex. All measurements are made at ambient temperature using standard technique. Blood is measured with Ostwald-Folin pipets (Hawk, Oser, Summerson, *Practical Physiological Chemistry*, 13th ed., New York, 1954, p. 542).

Procedure:

. Alcohol Stock Solution: Alcohol standards are prepared in terms of grams per 100 cubic centimeters in accordance with the Louisiana Implied Consent Law. Absolute ethanol from a freshly opened bottle is used. An alcohol solution of 50 g/l is prepared by measuring 62.5 ml of the alcohol into a 1000 ml volumetric flask. Considering the density of the alcohol to be 0.8 g/ml:

62.5 ml X 0.8 g/ml = 50 g ethanol

The volume is made to the mark with water, allowed to equilibrate at room temperature, and made again to the mark if necessary. This 5 g% solution is then dispensed into 5 ml glass ampuls and rapidly sealed by fusion. Each ampul is numbered in the order prepared. The entire liter quantity is thus preserved for use as needed. A 10% ordered sample is taken for oxidative analysis as described below (Analysis of Alcohol Standards. . .), in order that the exact alcohol content be established by relating it to the potassium dichromate primary standard.

2. Internal Control: For the purpose of controlling the volume of injection in the gas chromatograph, an internal control is used. One milliliter of l-propanol is diluted to 500 ml with water for this purpose. This solution is made up only as required, as it is quite stable. The exact concentration is not critical, but should be identical for all samples within a given batch of blood analyzed and should produce a peak of height approximately the same as a 0.10 g% ethanol solution.

3. Working Standards: One of the 5 g% stock ampuls is opened and 1.00 ml of the ethanol solution is delivered into volumetric flasks of 100, 50, 25, and 10 ml capacity. When made to the mark with water, the resulting solutions will have (nominal) concentrations of 0.05, 0.10, 0.20 and 0.50 g%, respectively. These solutions will be treated exactly as the unknowns and will be used for the purpose of establishing a calibration curve for the unknowns. Parker *et al.* (Anal. Chem., 34 p. 1234, 1962) have shown that aqueous standards may be used for calibration of a direct injection method but this fact should be explicitly demonstrated.

4. Unknowns: Exactly 1.00 ml of the unknown blood (measured by Ostwald-Folin pipet—working standards are measured by transfer pipet) is delivered into a glass or plastic vial of approximately 10 ml capacity fitted with a plastic snap cap. Exactly 1.00 ml of the internal control solution (measured with a single transfer pipet) is delivered into each vial. All unknowns are prepared in duplicate; standards are prepared as singlets. For the purpose of preparing duplicates, blood is drawn from separate tubes if more than one is available. If clotted blood must be analyzed, the clot is homogenized in a Ten Broeck tissue homogenizer. If serum or urine must be analyzed they are treated as blood, but reported as serum alcohol or urine alcohol. The vials are capped and mixed. A small hole is punctured in the center of each cap to allow introduction of the syringe needle without removing the cap. The loss of alcohol through this opening is insignificant over reasonable periods of time (several hours). The holes should be sealed with plastic tape if it is not possible to begin injections immediately.

5. Control Bloods: Out of date transfusion blood is obtained and checked by direct injection to determine that it is free of ethanol. In a 250 ml volumetric flask, place 5.00 ml of the 5 g% stock ethanol solution. Fill to the mark with blood at room temperature. After thorough mixing, the blood will contain 0.10 g% ethanol (nominal). This blood is dispensed into clean empty test tubes in 2 ml amounts, stoppered and frozen on their sides for later use. Each batch of unknown samples should contain one of these controls as well as an ethanol-free blood sample prepared similarly. The analysis of the 0.10 g% blood control should give results within 0.01 g% of the value determined by an oxidative method (e.g. modified Kozelka-Hine, such as in Kirk, P.L., Crime Investigation, Interscience, New York, 1953, p. 751), which confirms both the accuracy of the concentration and the adequacy of aqueous standards. The negative control blood should give a result less than 0.01 g%. If the blood controls do not meet these criteria the results should not be reported.

6. Sample for Injection: A Hamilton No. 701 microsyringe (or equivalent) is used for injection into the gas chromatograph using the following technique. Water is drawn into the syringe not quite to the 2 microliter mark, excluding air bubbles. The plunger is then drawn back to the 2 microliter mark, pulling a short "plug" of air into the needle of the syringe. The needle is then introduced into the sample and the plunger is withdrawn to the 3 microliter mark, pulling 1 microliter of sample into the needle. The plunger is then withdrawn sufficiently to pull half a microliter of air into the needle. An additional microliter of water is drawn into the syringe to rinse blood from the needle into the syringe barrel, followed by sufficient air so that the water "plug" is entirely visible. At this point the exact volume of the sample is observed under a magnifying lens in suitable lighting to determine that a one microliter

sample has been obtained. Immediately upon injecting the sample into the gas chromatograph the syringe is withdrawn from the injector port, the integrator started, and a syringe-full of water is pulled into the syringe. A wire of diameter just smaller than the bore of the syringe needle is now threaded through the needle until it is visible in the syringe barrel. This wire is moved back and forth several times to insure that all blood deposits have been removed from the inner surface of the needle. The wire is removed, the water forced from the syringe and the syringe is rinsed 10 times with water. Using this injection technique, no difficulty will be experienced with clogged syringe needles and good replication of injection is assured. The plunger of the syringe should be removed between injections and wiped thoroughly with a chem-wipe or clean tissue.

7. Protocol for Injections:

   a. The series of standards is injected in ascending order of concentration (single injections).

   b. The alcohol-free blood control is injected (single injection).

   c. The unknown duplicates are injected (single injections).

   d. The (nominal) 0.10 g% control blood is injected (single injection).

Calculation:

The ratio of the ethanol response to the propanol response will be directly proportional to the ethanol concentration since the propanol concentration is held constant in all samples. If response to both ethanol and propanol are linear over the range of application (0 - 0.5 g%) then the response ratios are described by the equation

$$y = a_o + a_1 x$$

where x = alcohol concentration in g%
y = response of EtOH/response of PropOH
a_o = intercept
a_1 = slope

This equation may be fitted to the calibration data by the method of least squares. Unknown values may be derived from the equation by solving the equation for x,

$$x = \frac{Y - a_o}{a_1}$$

and substituting the observed values of y for the unknown bloods. Treatment of the data in this manner will:

1. reveal any constant bias of the method, reflected in a_o.
2. reveal the sensitivity of the method, reflected in the slope a_1.
3. reveal non-linearity in the response of the ethanol or propanol in the correlation coefficient, r, derived in the method of least squares: $|r| = 1$ in the case of perfect fit, $|r| < 1$ in most real situations.

Further, the mathematics of the least squares approach lends the method to statistical analysis for the purpose of placing confidence limits on the values derived or for the purpose of statistical comparison with other methods.

Retention Times:

To distinguish ethanol from other volatiles which may be present in the blood the retention time difference of the ethanol peak and the propanol peak is determined. This approach is not useful for comparison of day-to-day results due to slight variations of operating parameters and column performance. For within-batch comparisons, however, the replication is excellent and obviates the need to accurately time the injection or column hold-up, since the difference is not affected by these values. The retention time difference for the unknown bloods should match that of the standards and the 0.10 g% control blood within 0.03 minutes (1.8 seconds), with an absolute retention time for ethanol on the order of one minute.

Analysis of Alcohol Standards by Potassium Dichromate Oxidation

General: Standard terminology is used throughout to indicate precision of measurement (e.g. $0.009 < 0.100 < 0.101$ but $0.09 < 0.10 < 0.11$) except for volumetric glassware. Pipets and volumetric flasks used are all Class A and appropriate precision is assumed, burets are of the Machlett Auto-Buret type, 10 ml capacity, $\pm$ .02 ml tolerance. Standard pipet technique is observed with a 15 sec. drain time.

Preparation of Samples: Ten per cent of the 5 g% stock ampuls prepared are chosen in order of preparation (i.e. 1, 11, 21, 31, etc.). These are opened in turn and 1 ml is immediately withdrawn by pipet and delivered into a 50 ml volumetric flask. The flask is filled to the mark with distilled water, rinsing down all stock solution into the body of the flask. This dilution is repeated for all

samples, yielding, when made to the mark with water and completely stirred, solutions of 1.10 g% ethanol concentration.

Method: The alcohol in 1 ml of diluted sample (1 mg) is oxidized by a known quantity of potassium dichromate in 45% sulfuric acid. The heat generated by diultion of conc. sulfuric acid is used to complete the oxidation. This is accomplished by layering 5 ml (approx. by pipet or graduate) conc. sulfuric acid under a solution of 5 ml (buret) 0.100 N Potassium dichromate and 1 ml (pipet) of sample, then swirling to mix. (This is entirely satisfactory for the oxidation of 1 mg of alcohol, but incomplete oxidation becomes apparent with samples containing more than 2 mg of alcohol.) The sulfuric acid used must be of the best quality—even reagent grade frequently contains reducing material and will be associated with high blanks. It is imperative that the same lot of acid be used for any batch of analyses.

The reaction mixture is then diluted to less than 10 per cent sulfuric acid concentration with distilled water and an excess of potassium iodide added. The liberated iodine is titrated with 0.1 N Sodium thiosulfate, using freshly prepared 1% starch solution (prepared by heating with constant stirring up to the boiling point) to indicate the equivalence point.

To avoid side reactions it is necessary that the sulfuric acid concentration be less than 10 per cent, and that local excess of thiosulfate not occur. Further error may be encountered if the solutions are allowed to stand after liberation of iodine—both through evaporation of iodine from the solution and auto-oxidation of potassium iodide in contact with air. Consequently, after addition of potassium iodide, the tritration is completed as quickly as is consistent with precaution against local excess of thiosulfate.

Chemistry: The reactions involved may be described by the following quations;

$$Cr_2O_7^{--} + 14H^+ + 6e^- \rightarrow 2Cr^{+++} + 7H_2O$$

$$CH_3CH_2OH + 4OH^- - 4e^- \rightarrow CH_3COOH + 3H_2O$$

$$2K_2Cr_2O_7 + 8H_2SO_4 + 3CH_3CH_2OH \rightarrow$$

$$2Cr_2(SO_4)_3 + 11\,H_2O + 3CH_3COOH + 2K_2SO_4$$

Calculations: It follows, therefore, that the equivalent weight of potassium dichromate is 1/6 the formula weight or 49.03 grams, and that a 0.100 N solution contains 4.903 grams per liter. The equivalent weight of ethanol is ¼ the formula weight or 11.52. The milliequivalents of dichromate consumed in the oxidation (which equals the milliequivalents of alcohol oxidized) is given by the expression

$$N_dV_d - N_tV_t$$

where N_d = normality of the standard dichromate
V_d = volume (ml) of the dichromate solution used in the sample oxidation
N_t = normality of the thiosulfate solution
V_t = volume (ml) of the thiosulfate used in the sample tritration

This may be converted into milligrams of alcohol by multiplying by the milliequivalent weight of alcohol, 11.52. The per cent of alcohol is the sample may be obtained from this by converting to grams, dividing the expression by the volume of the sample in milliliters and multiplying by 100 (g%). Thus (where V = sample volume):

$$x = \frac{11.52}{10\,V_s}\,(N_dV_d - N_tV_t)$$

Now, from the titration blank we know that

$$N_dV_d = N_tV_{bt}$$

where V_{bt} = volume of thiosulfate required by the blank. Solving for N_t and substituting, we obtain the expression from which the per cent alcohol may be calculated:

$$x = \frac{11.52}{10\,V_s}\,N_dV_d\,(1 - V_t/V_{bt})$$

Since adherence to the procedure causes V_d = 5.00 ml, V_s = 1.00 ml, and N_d = 0.100, then

$$x = 0.576\,(1 - V_t/V_{bt}) \qquad \text{q.e.d.}$$

Procedure: Twice the number of 250 ml Erlenmeyer flasks as there are samples, scrupulously clean, are loaded with 5.00 ml of 0.100 N potassium dichromate each. Exactly 1 ml of distilled water is run into each of half the flasks. The remaining flasks are loaded with exactly 1 ml of the diluted samples. Each flask in turn is treated as follows: Five ml of conc. sulfuric acid is carefully run down the side of the flask to form a layer under the potassium dichromate/sample solution; the neck of the flask is covered with an inverted beaker; the