Department of Health, Education and Welfare

Public Health Service

H S M H A

National Institute For Occupational Safety and Health

Division of Laboratories and Criteria Development

Cincinnati, Ohio 45202

INDUSTRIAL HYGIENE SERVICE LABORATORY QUALITY CONTROL MANUAL

TECHNICAL REPORT NO. 78

DRAFT

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PREFACE

We are indebted to the Literary Executor of the late Sir Ronald A. Fisher, F.R.S., to Dr. Frank Yates, F.R.S., and to Oliver & Boyd, Edinburg, for permission to reprint extracts from Tables III and IV from their book Statistical Tables for Biological, Agricultural and Medical Research.

PURPOSE

1.1 The purposes of this manual are: (1) To direct the establishment and provide guidance in the maintenance of a Quality Control System in the National Institute For Occupational Safety and Health (NIOSH) Service Laboratories. (2) To serve as a source of directive and instructive material for other participating laboratories in the establishment and maintenance of laboratory quality control within those laboratories.

This manual describes administrative systems and statistical techniques, but does not cover detailed and specific calibration procedures or specific analytical methodology.

1.2 The basis for use of statistical techniques may be summarized as follows: Statistical quality control involves application of the laws of probability to systems where chance causes operate. The technique is employed to detect and separate assignable (determinate) from the random (indeterminate) causes of variation. Statistics is the science of uncertainty; therefore, any conclusions based on statistical inference contain varying degrees of uncertainty expressed in terms of probability. Uncertainty can be quantified in terms of well defined statistical probability distribution which can be applied directly to quality control. The application of statistical quality control can most efficiently indicate when a given procedure is under control, and a continuing program that covers sampling, instrumentation and overall analysis of quality will assure the validity of the analytical program. Further development of statistical techniques and applications will be found in Section XI.

QUALITY OBJECTIVES

2.1 The objective of the Analytical Laboratory Quality Control program is to assure the medical and/or scientific reliability of laboratory data used in NIOSH activities and documents. Management, administrative, statistical, investigative, preventive and corrective techniques will be employed to maximize reliability of the data.

2.2 Specific objectives are:

2.2.1 To develop and/or put into service rugged methods capable of meeting the users' needs for precision, accuracy, sensitivity, and specificity.

2.2.2 To establish the level of quality of the laboratory's routine performance.

2.2.3 To make any changes in the routine methodology found necessary to make it compatible with performance needs in Par. 2.2.1.

2.2.4 To monitor the routine operational performance of the laboratory through an appropriate intra-laboratory program and to provide for corrective actions as necessary.

2.2.5 To participate in quality evaluation programs with peer laboratories to achieve and maintain consistent uniform levels of quality.

2.2.6 To improve and validate laboratory methodologies by participation in method validation collaborative tests.

QUALITY CONTROL POLICIES

This section lists policies to be implemented by the laboratory in order to achieve the objectives stated in Section II and in furtherance of the overall quality control program. Details for carrying out these policies appear in later sections of the manual.

3.2 These policies include:

3.2.1 Publication, distribution and maintenance of current and complete Laboratory Analytical Methods and Procedures, Sampling Data Sheets, Calibration Data Sheets and Analytical Instrument Operating Instructions.

3.2.2 Promulgation, distribution and retention of laboratory reports with provision for administrative/technical review.

3.2.3 Periodic calibration of instruments and equipments, both in the laboratory and in the field, Quality Control checks on analytical instruments to ensure proper function at all times; and a preventive maintenance program.

3.2.4 Assurance of appropriate, fresh reagents and chemicals and for appropriate, calibrated glassware.

3.2.5 Establishment and maintenance of total analytical quality control systems to assure continued precision and accuracy of laboratory reports, including, as appropriate, requirements that:

a. Each test shall be checked on each day of use.

b. At least one standard (may be an instrument standard) and one control sample (working value established and run through the entire test (analytical) procedure) shall be included with each run of unknown samples. Where the control sample is not subject to the interferences, etc., of the unknown samples, a previously run unknown will be included as a blind check sample. A blank sample (no added amount of the constitutent

being determined) shall also be run to aid in detecting reagent contamination and other problems important near the lower limit of operation of the method.

c. If the results on the standard, control,blank or recycle samples are not within acceptable limits, the entire batch of analyses must be repeated and control verified before reports are issued. Serious consideration should be given to the non-acceptance of samples where there is only enough material for a single analysis. There may be situations where this policy is waived. Consideration of the consequences of reporting results when the analytical system is apparently "out-of-control" should minimize such waivers.

3.2.6 Requirements for participation in inter-laboratory quality evaluation programs.

3.2.7 Requirements for training and qualifying personnel in quality control techniques and prior to running new tests. This qualification test is to be statistically valid and include evaluation of precision and accuracy. The qualification standard shall be the established level of quality of the laboratory.

ORGANIZATION FOR QUALITY

4.1 The establishment of a Quality Control Program as described in this manual will require the services of a Quality Control Coordinator within the laboratory to carry out the monitoring, record keeping, statistical techniques and other functions required in such a system.

4.2 This individual may have these duties as his sole responsibility in a large organization and need clerical or technical assistance, or, in a smaller organization the incumbent may wear this as "another hat".

4.3 The Quality Control Coordinator should be assimilated into the organization, reporting to the highest level possible. In no case, however, should the Quality Control Coordinating function be subordinate to an individual responsible for a direct conduct of analyses.

4.4 A representative organization chart illustrating the placement of the Quality Control function in a laboratory follows. (Figure IV-1)

4.5 The Job Description of the Quality Control Coordinator should, as a minimum, include responsibilities listed in Exhibit IV-1.

QUALITY CONTROL
LINE AND STAFF ORGANIZATION CHART

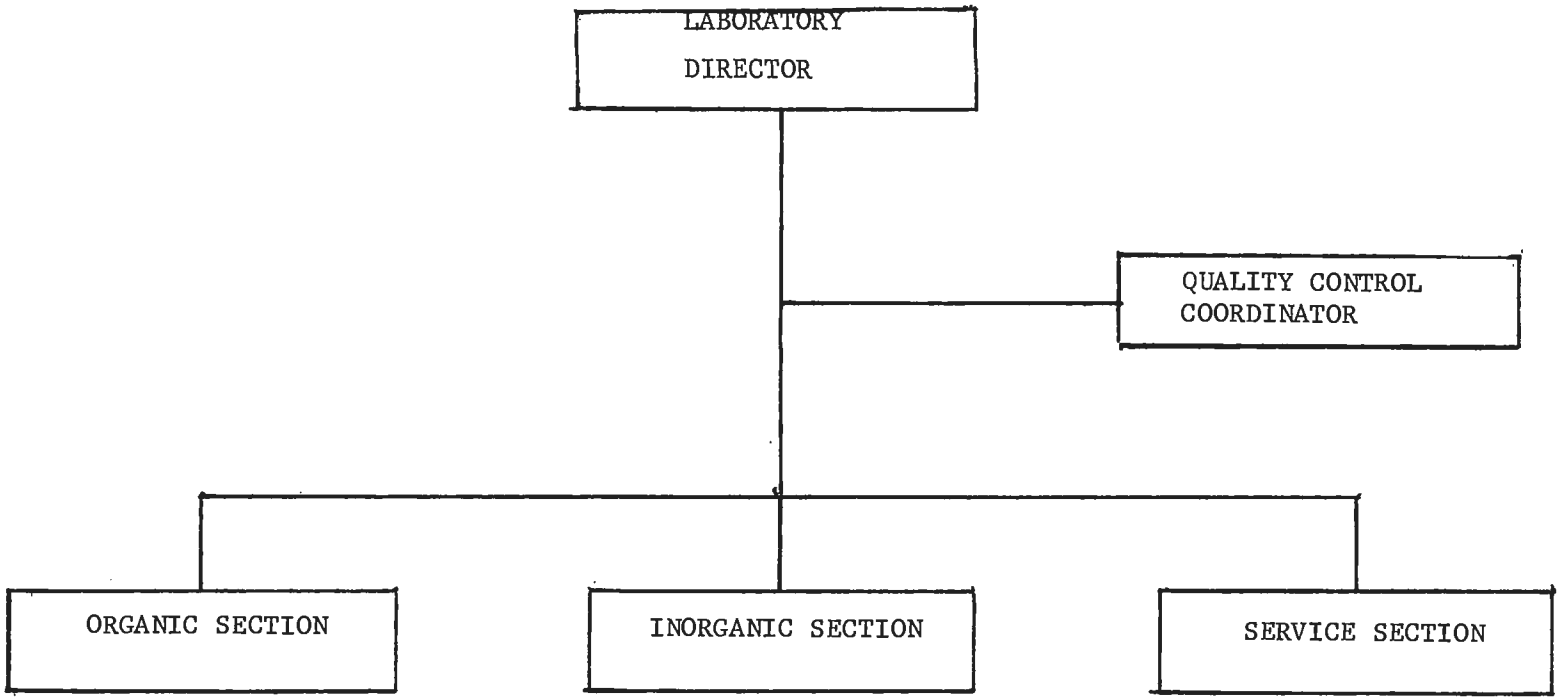


FIGURE IV-1

JOB DESCRIPTION

TITLE: Quality Control Coordinator.

1. Basic Function

The Quality Control Coordinator is responsible for the conduct of the Analytical Laboratory Quality Control program and for taking or recommending measures to ensure the fulfillment of the quality objectives of management and the carrying out of Quality Control policies in the most efficient and economical manner commensurate with ensuring continuing accuracy and precision of data produced.

2. Responsibilities and Authority.

2.a. Develops and carries out quality control programs, including statistical procedures and techniques, which will help laboratories to meet authorized quality standards at minimum cost; and advises and assists management in the installation, staffing and supervision of such programs.

2.b. Monitors quality control activities of the laboratory to determine conformance with authorized policy and procedures and with sound practice; and makes appropriate recommendations for correction and improvement as may be necessary.

2.c. Seeks out and evaluates new ideas and current developments in the field of quality control and recommends means for their application wherever advisable.

2.d. Advises management in reviewing technology, methods, and equipment, with respect to quality aspects.

2.e. Advises the Purchasing Section regarding quality of purchased materials, reagents and chemicals.

2.f. Recommends packaging materials and procedures.

2.g. Performs related duties as assigned.

QUALITY CONTROL RECORDS

5.1 Purpose

The purpose of this section is to describe the use of forms (Annex 1 - 5) used to request, record, check and report on the results of analytical tests performed to measure the degree of atmospheric contamination in samples taken by field industrial hygienists or NIOSH laboratory personnel. These are specific for NIOSH and the Department of Labor. Other laboratories may use this section as a guide for the use of their Quality Control Records.

5.2 Sample Analysis Requests

5.2.1 Department of Labor Field Industrial Hygienists shall submit requests for sample analyses to the NIOSH Salt Lake City Laboratory or to appropriate State Laboratory on USDL Forms LSB 00S 7 "Sample Identification" (Annex 2 - Section V) and LSB 00S 6 "Sample Accountability Record" (Annex 3 - Section V) both in two copies, one enclosed in the sample package, and the other mailed separately. The sample package is sealed with the Department of Labor seal.

5.2.1.1 Upon receipt the sample is assigned a laboratory number and logged in on the Test Sample Log (Annex 1 - Section V).

5.2.1.2 At the same time, USDL Form LSB 00S 5 (Annex 4 - Section V) "Analyst Worksheet" is accomplished in an original and two copies, all of which accompany the sample through all laboratory procedures. After the assigned analyst has completed the analysis requested, he fills out Parts 7, 8, 9, and 10 and returns all copies to the Chief Chemist of the Service Section together with the sample, if requested. One copy of the "Analyst Worksheet" is mailed to the requester, a second to the OSHA Region, and the original is filed and retained for not less than five years.

5.2.2 The Cincinnati Laboratory receives requests for analyses on the "Information on Samples For Analysis" Form (Annex 5 - Section V). These are logged in as in Par. 5.2.1.1 above. This form follows the sample through the laboratory procedure and is filed together with a copy of the report memorandum (Annex 6 - Section V) and retained for a period of not less than five years. The report memorandum "Results of the Analysis of - - -" (Annex 6 - Section V) is prepared in duplicate and the original is sent to the requester giving him results of the analyses and appropriate comments.

5.3 Statistical Quality Control Records

There are two principal kinds of statistical tools available for use in analytical laboratories: control charts and tests for differences including analysis of variance. See Section XI for a general discussion of these together with sample problems and a step-by-step procedure for the construction of $\overline{X}$ - R charts using actual laboratory data for analyses for mercury.

5.3.1 Control Charts. It shall be the responsibility of the Laboratory Quality Control Manager to establish control charts for each test method for blank analysis data, proficiency tests and recycled sample analyses for each of those test methods. Sample sizes are related to the number of replications. If the analyst uses several values as a single control chart point (an $\overline{X}$ chart), his chances of picking up small changes in the process average are increased. The protection against not detecting small changes in the process average increase as the sample size increases. The use of frequent sampling will detect changes more quickly with time. The ultimate in control would be large sample sizes taken frequently. However, an economic decision has to be made as to the value of closer control versus the increased cost of attaining that degree of control.

5.3.2 Tests for differences shall be computed by the Quality Control Coordinator as requested or as he feels necessary.

5.3.2.1 Sample sizes to be recycled shall be determined from the following table:

Data Availability No. of Tests	Sample Size
3 - 8	2
9 - 15	2
16 - 25	5
26 - 40	8
41 - 65	13
66 - 110	20
111 - 180	32

5.3.2.2 Larger samples may be used if past history or experienced judgment renders such a step advisable.

5.3.3 The Quality Control Coordinator shall institute and maintain a program of analysis checks.

5.3.3.1 Frequency of checks shall be determined by:

5.3.3.1.1 Ruggedness, precision and accuracy of the method.

5.3.3.1.2 Dependability of the analyst.

5.3.3.1.3 Precision and sensitivity of the instrument.

5.3.3.1.4 Difficulty and time length of the test method.

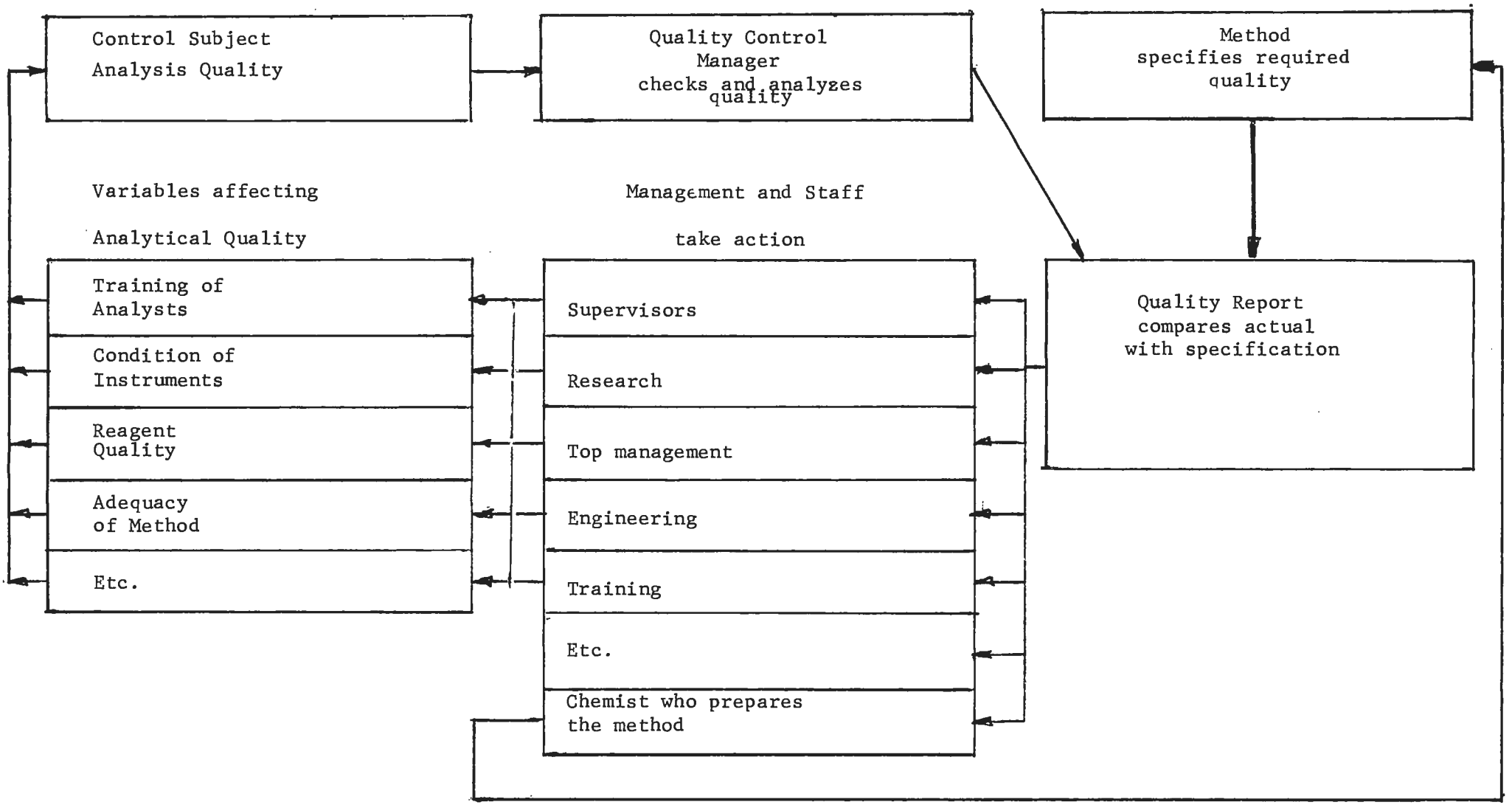
5.3.3.2 Check samples shall be selected from a method "family" at random and at irregular intervals.

5.3.4 The data from the check analyses shall be compared with that from the original analyses and tested for significant differences. Any significant differences shall be reported to the Director of the Laboratory. The Quality Coordinator shall prepare a monthly report showing, by test method, the percent of significantly different check analyses that occurred during that period and recommending corrective action. A copy of this report shall be furnished to the Chief, Chemical and Physical Analysis Branch.

5.4 Feedback and Corrective Action

The Quality Control Coordinator by identifying trouble spots, out of control conditions, and significant departures from normal results brings to management the need for corrective action and makes recommendations for such matters. This is illustrated by the following diagram:

SERVOMECHANISM DIAGRAM FOR QUALITY CONTROL



TEST SAMPLE LOG

Field No.	Log No.	Description	Req. By	Date Rec'd.	Assign'd. To	Report Date

U.S. DEPARTMENT OF LABOR
BUREAU OF LABOR STANDARDS
Office of Occupational Safety

SAMPLE IDENTIFICATION

1. SAMPLE NUMBER (REGIONAL)	2. DATE TAKEN	3. DATE SUBMITTED TO LAB
P-90	3-29-72	3-4-72

4. BY WHOM TAKEN	5. LABORATORY NUMBER
J. L. Torrey	

6. WHERE TAKEN

NAME OF ESTABLISHMENT: Fountain Foundry

STREET ADDRESS: 815 West 14th

CITY	COUNTY	STATE
Pueblo		Colo

7. OPERATION: Melter

8. SAMPLING TECHNIQUE

IMPINGER ____ BUBBLER ____ ESP ____ FILTER PAPER V D-39

MATERIAL ____ BAGS ____ SETTLED DUST ____ GRAB SAMPLE ____

9. AIR VOLUME SAMPLED (IN LITERS AND CUBIC FEET): 399 minute personal sample at 2.2 lpm

10. POSSIBLE INTERFERENCES

VM-1 clean filter 17.12 mg

11. ANALYSIS REQUIRED: SiO_2 - Gross dust

12. DATE REPORTED FROM LAB	13. CHEMISTS

14. REMARKS

Form LSB 008 7

U.S. DEPARTMENT OF LABOR
BUREAU OF LABOR STANDARDS
Office of Occupational Safety

SAMPLE
ACCOUNTABILITY RECORD

1. SAMPLE NUMBER	2. NAME OF ESTABLISHMENT	
3. ADDRESS OF ESTABLISHMENT		
4. DATE SAMPLE RECEIVED	5. BY WHOM RECEIVED	6. DATE RECORDS RECEIVED

7. METHOD OF SHIPMENT	A. PERSONALLY FROM	C. SHIPPED FROM
	B. VIA ☐ PP ☐ REA ☐ FREIGHT ☐ AIR	D. B/L NUMBER

8. DESCRIPTION OF SHIPMENT				
	A. SHIPPING CONTAINERS	NUMBER	TYPE	CONDITION
	B. SAMPLE PACKAGES	NUMBER	SIZE AND TYPE	CONDITION
	C. SEAL INSCRIPTION	COPY IN FULL		CONDITION

9. SAMPLE DELIVERED				10. SAMPLE RETURNED			
DATE	AMOUNT	FROM	TO	DATE	AMOUNT	TO	FROM

11. SAMPLE DISPOSITION

Form LSB 00S 6

U.S. DEPARTMENT OF LABOR
BUREAU OF LABOR STANDARDS
Office of Occupational Safety

ANALYST WORKSHEET

1. SAMPLE NUMBER	2. SEALS ☐ INTACT ☐ BROKEN ☐ NONE	3. DATE RECEIVED
4. RECEIVED FROM	5. REGION	
6. DESCRIPTION OF SAMPLE		
7. SUMMARY OF ANALYSIS		
8. RESERVE SAMPLE		
9. DATE REPORTED	10. ANALYST	11. CALCULATIONS CHECKED BY	12. DATE CHECKED

Form LSB 00S 5

Laboratory of Physical and Chemical Analysis Branch
NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH

INFORMATION ON SAMPLES FOR ANALYSIS

Chemist:_____ Date:_____

Lab. No.	Field No.	Type of Sample	Analyses Desired	Remarks	Results

Do you wish unused portion of sample returned to you? Yes / / No / /

_____
(Place sample was collected)

_____
(Name of person submitting sample)

_____

_____
(Affiliation)

MEMORANDUM

DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
PUBLIC HEALTH SERVICE
HEALTH SERVICES AND MENTAL HEALTH ADMINISTRATION
North Institute for Occupational Safety & Health

TO : Chief, Industrial Hygiene Services Branch DATE: April 12, 1972
Attention: Ron Mertz
Through: Chief, Physical and Chemical Analysis Branch

FROM : Chemist, Physical and Chemical Analysis Branch

SUBJECT: Results of the Analysis of Four Charcoal Tubes for Toluene (Lab. Nos. 59189, - 190, -192, -194) and Two Charcoal Tubes for Trichloroethylene and 1,1,1 Trichloroethane (Lab. Nos. 59191, -193); 6 tubes, 16 analyses.

Gas chromatographic procedures were used to analyze four tubes (Lab. Nos. 59189, -190, -192, -194) for toluene and two tubes (Lab. Nos. 59191, -193) for both trichloroethylene and 1,1,1 trichloroethane. All backup sections were also analyzed.

The results are given in the enclosed laboratory sheet and are expressed in milligrams per section of charcoal.

The holes in the ends of these six tubes were not large enough. When sampling, the ends of the charcoal tube should be broken so that the diameter of the hole is at least 2 millimeters, which is one-half the inner diameter of the tube. If the hole in the tube is not large enough, a limiting orifice effect results and the actual volume of sample taken cannot be accurately known. This limiting orifice effect could have affected your results in that your actual sample volume may be smaller than you believe.

Kathleen A. Schulte

Kathleen A. Schulte

Enclosure 1
Laboratory Results Sheet

Laboratory of Physical and Chemical Analysis Branch
NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH

INFORMATION ON SAMPLES FOR ANALYSIS

Chemist: [illegible] Date: [illegible]

Lab. No.	Field No.	Type of Sample	Analyses Desired	Remarks	Results (mg)
59189	3	[illegible]	[illegible]	[illegible]	0.8
-190	4	"	[illegible]	"	N.D. *
-191	5	"	[illegible]	trichloroethylene 1,1,1 trichloroethane	1.5 0.4
-192	6	"	[illegible]	[illegible]	0.8
-193	7	"	[illegible]	trichloroethylene 1,1,1 trichloroethane	0.6 1.0
-194	8	"	BLANK		N.D.

All backup sections were analyzed; nothing was found in any of them.

N.D - less than 0.1 mg on section.

approx 10 l vol. for sampling

The holes that were broken in the end of the tubes were too small.

Do you wish unused portion of sample returned to you? Yes [ ] No [X]

[illegible]
(Place sample was collected)

[illegible]
(Name of person submitting sample)

[illegible]

[illegible]
(Affiliation)

TRAINING

Modern technology requires that people throughout the Division and in other Laboratories be equipped with the necessary attitudes, knowledge and skills to deal with quality problems in the analytical laboratory environment. Since most involved have little background in quality control and the statistical tools it uses, it is necessary for those assigned quality control responsibilities to undergo training such as the program outlined below.

This program has three phases:

Phase I - A twelve hour course held by the NIOSH Cincinnati Laboratory for selected NIOSH and State personnel.

First Day, A.M.

I Using frequency distributions in quality control.
Central tendency

II Measuring variability..

III Estimating variability from the average range of test data.

P.M. IV The Normal Curve and Analysis of Control.

V Sampling error evaluation and control.

Second Day.

VI $\bar{X}$ - R Control Charts

VII R Charts for Variability.

Phase II - A self-study program in which users of this manual familiarize themselves and practice the use of tests for significance of difference, described in Section XI of the manual.

Phase III - Attendance at any Quality Control Training Course under NIOSH or other Continuing Education Programs. In addition to the Quality Control Training Sources listed below many local sections of The American Society for Quality Control offer annual courses in elementary, intermediate and advanced Quality Control and statistical methods.

As additional or new personnel are brought into the Quality Control Program care will be taken to ensure that each completes sufficient training to enable continuing support of and participation in the Quality Control Program.

Professional Societies

American Society for Quality Control

American Management Association (AMA)

Colleges and Universities

A list of colleges and universities offering courses in Quality Control, Reliability and Statistics is given in "Procurement Quality Control" available from The American Society For Quality Control, 161 W. Wisconsin Avenue, Milwaukee, Wisconsin 53203.

DOCUMENT CHANGE CONTROL

7.1 Purpose

7.1.1 The purpose of this section is to establish within DLCD a standard procedure for recommending, publishing and distributing changes to Laboratory Analytical Methods, Sampling Data Sheets, Calibration Data Sheets and Analytical Instrument Operating Instructions.

7.1.2 The sign-out, recall system will establish controls to ensure that:

7.1.2.1 Analyses, sampling methods and calibrations are carried out in accordance with the latest technical document issue.

7.1.2.2 An effective communication system between the DLCD Cincinnati Laboratory, the Salt Lake City and State Laboratories and field personnel is developed.

7.1.2.3 A central storage and issue point for technical documents and changes is established.

7.2 Scope This procedure shall define the control of the following:

7.2.1 Laboratory methods.

7.2.2 Releases and changes.

7.2.3 Sampling data sheets.

7.2.4 Calibration instructions.

7.3 Data Review and Check

7.3.1 Technical documents shall contain sufficient data or instructions to enable user personnel to carry out correct analytical, sampling or calibration procedures.

7.3.2 It shall be the responsibility of the Development Laboratory, Physical and Chemical Analysis Branch, DLCD, and the responsible technical committee to ensure that documents contain adequate information to carry out their intended purpose. These groups shall review and check documents for completeness, technical accuracy and for conformance to requirements. We offer the following as information for the guidance of other laboratories who will be responsible for their own document control.

7.4 Document Control

7.4.1 The Development Laboratory will maintain full control on the distribution of documents covered under Section VII. A file control shall be established within the Development Laboratory showing the following minimum information:

Document number

Title

Branch originating the document

Latest issue date

Change number

Signatures of persons receiving the document

7.4.2 Whenever a change is made the Development Laboratory shall issue the new document together with the change notice.

7.4.3 Recipient of new or changed documents must acknowledge receipt by signature.

7.4.4 Obsoleted documents shall be removed from all files and points of use, returned to the Development Laboratory and shall be logged in and destroyed immediately. The Development Laboratory shall have sole authority to destroy obsoleted documents.

7.4.5 All original copies and change notices shall be maintained in the Development Laboratory.

7.5 Document Changes

7.5.1 Requests for changes to methods, sampling data sheets and calibration instructions may be initiated by anyone. the request being made in writing on the Technical Data Change Notice (Annex 1, Section VII). It must be approved by the Chief, Physical and Chemical Analysis Branch before the change is published and distributed.

7.5.2 Changes may be promulgated by the issue of entire new documents, of replacement pages thereto, or, in the case of minor changes, corrections of errata, etc., by pen and ink posting on the original document and this action noted on the change notice.

7.6 The Quality Control Coordinator is responsible for documents being distributed to those concerned and for obtaining the required signatures.

NATIONAL INSTITUTE
FOR
OCCUPATIONAL SAFETY AND HEALTH
DIVISION OF LABORATORIES & CRITERIA DEVELOPMENT

Date_____

TECHNICAL DATA CHANGE NOTICE

TDCN No._____

Document Type ___ Method
___ Sampling data sheet
___ Calibration instruction

No._____

Document Title _____

Requested By: _____

Change From:_____

Change To:_____

Reason for Change:_____

Distribution: No. Copies:	*Costs:	$	Approvals:
	New Equip.		Chief, C & P Anal. Branch
	New Mat'l.		Chief, Eng'r. Lab. Branch
	Scrapped or Obs. Equip.		Other
	Scrapped or Obs. Mat'l.		
	Personnel		
	Other		

* Detail on reverse side.

CONTROL OF INCOMING

REAGENTS AND CHEMICALS

8.1 Purchase Orders

8.1.1 A vendor of analytical materials supplied to laboratories is regarded as a resource to and an extension of the laboratory organization. The standards for quality, therefore, imposed on vendors are the same as those self imposed on laboratories.

8.1.2. The purchase order instructs vendors to mark containers or reagents and chemical and/or packing slips with the following information, when applicable:

Name of material

Vendor's name and address

Vendor's lot number

Quantity

Material specification number and date

This assures that the material is properly identified and the vendor is using the latest specifications.

8.1.3 Copies of all purchase orders for reagents and other analytical materials are sent to the Laboratory Quality Control Coordinator where they are reviewed to assure the latest requirements are correctly specified.

8.1.4 Purchase orders, receiving documents and accompanying certifications are used as part of the receiving control procedure and show information necessary to identify the material being received.

8.2 Control of Incoming Materials

8.2.1 The Laboratory Stores Clerk segregates incoming reagents or analytical materials and prepares receiving reports.

8.2.2 The Stores Clerk checks the package marking (packing slip) against the Receiving and the Quality Control copies of the Purchase Order. If a discrepancy is found, the material is discarded and reordered. If the material is accepted, the material is logged in (See Annex 1 ,- Section VIII) and placed in stores noting,

Identification

Vendor

Date

P. O. number

Assigned log number

The inner container label is stamped with the log number and the shelf life expiration date. (See Annex 2, Section VIII). No reagents, chemicals, standard solutions or other analytical materials shall be used after the expiration of the assigned shelf-life date.

8.2.3 When, in the judgment of the Quality Control Coordinator, it is desirable to check the validity of a certification of a purchased material, he shall request such a check to be made through the chief chemist. Such checks shall be made at random intervals. In the event of rejection, the material is discarded.

8.2.4 It shall be the duty of the Stores Clerk once monthly to survey the inventory to identify materials approaching a shelf-life expiration date within 30 days and to re-order fresh replacement stock.

8.2.5 As supplies are used or requisitioned the amounts are posted against the log number and thus, a running inventory is kept against which replacement orders are made by the Stores Clerk to prevent "stock-outs".

8.2.6 Each receiving report is referenced by log number, to the applicable purchase order, certification, report of analytical results and other related data, and is retained in the Quality Control file.

8.2.7 The logged dispositions are reviewed to establish trends in vendor performance, and to ensure a continuing high quality of analytical materials accepted.

8.2.8 When the Stores Clerk issues materials to the analyst, he (she) checks to be sure that the material is properly identified, shows the log number and has a current shelf-life expiration date. In the case where more than one container is stocked, the oldest is used first.

8.2.9 Since the quality, strength, concentration, or composition of reagents, solutions, or solvents, etc., are nearly always checked against standards or otherwise as a part of the analytical method or procedure, there is no need for any check on these materials prior to placing them in stores, other than that required to validate certification covered in Pars. 8.2.2 and 8.2.3 above.

REAGENT

RECEIVING AND STORES LOG

Log No.	Identification	P.O. No.	Vendor	Date	Disposition Acc. - Rej.

REAGENT SHELF LIFE LIMITS

(Omitted. To be added by Chem. & Phys. Anal. Branch)

PACKING AND SHIPPING

9.1 Because of the fragile and sensitive nature of sample containers shipped from the field or between laboratories, special precautions must be taken for handling, storage, packaging, and shipping to protect the sample integrity of specimens and minimize damage, loss, deterioration, degradation or loss of identification of samples.

9.2 Analytical methods published by NIOSH will include detailed instructions and specifications for handling, storage, packaging and shipping of samples.

9.3 Quality Control Coordinators shall be responsible for monitoring this program and shall instruct all personnel including field industrial hygienists in carrying out these procedures.

INSTRUMENT CALIBRATION AND MAINTENANCE

10.1 Purpose

This section establishes forms (Annexes 1 through 3-X) and the procedure for maintaining the accuracy of instruments and measuring equipment used by Laboratories and Field Industrial Hygienists to collect samples, to conduct tests and analyses for atmospheric contaminents and to conduct certification tests on items of personal safety equipment submitted by manufacturers for approval.

10.2 Scope

10.2.1 The calibration policies and procedures set forth herein shall apply to all instruments and measuring equipment identified in 10.1 including:

Laboratory analytical instruments

Balances and weights

Microscopes

Sampling devices such as detector tube pumps

Rotameters

Manometers, etc.

Vacuum pumps

Dry gas meters

10.2.2 All instruments fall into two general categories:

10.2.2.1 Those which are calibrated prior to each use.

10.2.2.2 Those which are calibrated on a scheduled, periodic basis.

10.3 Records

10.3.1 All equipment to be calibrated under this procedure shall have an assigned record number permanently affixed to the instrument. This may be the capital equipment inventory control number.

10.3.2 A calibration control card (Annex 3 - Section X) is established for each instrument showing:

Identification number

Description including manufacturer, model number and serial number

Location of use or storage

Calibration instruction number reference

Mandatory calibration interval

Date of last calibration

By whom performed (signature)

Due date of next calibration

Values obtained during last calibration

Calibration reports and compensation or correction figures shall be filed with the calibration control card.

10.4 Environmental Controls

All measurement standards and measuring and test equipment shall be calibrated and/or utilized in an area in which the laboratory has provided controls for environmental conditions to the degree necessary to assure measurements of the specified accuracy. The calibration area should be reasonably free of dust, vibration, and radio frequency interferences and should not be physically close to equipment that produces acoustical noise, or vibration, or to areas in which there is environmental testing or use of high power radar work, etc.

Isolation of pressure, mass and acceleration equipment from vibrations is particularly essential. Isolation mounts, seismic masses, etc., should be provided for these.

The laboratory or calibration area should have adequate temperature and humidity controls. A temperature of from 68° F. to 73° F. and a relative humidity of 35%-55% normally provide a suitable environment.

A filtered air supply is a necessity in the calibration area. Dust particles are more than just a nuisance; they can be abrasive, conductive, damaging to instruments and injurious to health. A measure of dust filtration can be provided in the air conditioning system by the washing action of sprays and atomizers, but this may need to be supplemented by electrostatic and/or mechanical filters of the activated charcoal, oilcoated or ribbon type.

Other areas for consideration are:

a. Power. Recommended requirements for electrical power within the laboratory should include voltage regulation of at least 10% (preferably 5%); low values of harmonic distortion; minimum line transients as caused by inter-action of other users on main line to laboratory (separate input power if possible); and a suitable grounding system established to assure equal potentials to ground throughout the laboratory, (or isolation transformers may be used to operate individual equipments.)

b. Lighting. Adequate lighting (suggested values -- 80 to 100 foot candles) must be provided for work bench areas. The lighting may be provided by overhead incandescent or fluorescent lights. Fluorescent lights should be shielded properly to reduce electrical noise.

10.5 Calibration Procedures

10.5.1 Field sampling instruments and equipments.

10.5.1.1 The method of calibration shall be detailed in written instructions which appear in the "Instructions" Section of this manual.

10.5.1.2 The written instructions shall cover each type or group of similar instruments.

10.5.1.3 All primary standards, i.e., those traceable to National Standards shall be calibrated by outside laboratories and no procedures need be written on the method of calibrating them.

10.5.1.4 The internal standard technique is used primarily for emission spectrograph, polarographic, and chromatographic (liquid or vapor phase) procedures. This technique enables the analyst to compensate for electronic and mechanical fluctuations within the instrument. In brief, the internal standard method involves the addition to the sample of known amounts of a substance to which the instrument will respond in a manner similar to the contaminant in the system. The ratio of the internal standard response to the contaminant response determines the concentration of contaminant in the sample. Conditions during analyses will affect the internal standard and the contaminant identically and thereby compensate for any changes. The internal standard should be similar chemically to the contaminant of approximately the same concentration anticipated for the contaminant, and of the purest attainable quality.

10.5.2 All laboratory instruments shall be calibrated and checked by competent personnel. The fact that outside services will schedule and perform these checks does not excuse the laboratory from the responsibility of controlling, monitoring and identifying calibration intervals and seeing that checks are made on time.

10.6 Calibration Identification

10.6.1 Each instrument shall have affixed to it in plain sight a tag bearing the information shown as in Annex 2 - Section X.

10.6.2 Instruments past due for calibration shall be immediately removed from service either physically, or if this is impractical, they must be impounded by tagging, sealing, labelling, or other means.

10.6.3 Field service instruments due for calibration shall be recalled prior to the due date and returned to the laboratory for calibration unless adequate calibration means are made available in the field.

10.6.4 The labelling and recording system described herein extends to calibration services performed by commercial laboratories. Certifications and reports furnished by them shall be filed and made a part of this system.

10.7 Calibration Standards

10.7.1 All measurements or calibrations performed by or for the laboratory in the accomplishment of these requirements shall be traced, directly, or indirectly, through an unbroken chain of properly conducted calibrations (supported by reports or data sheets) to some ultimate or national reference standards maintained by a national organization such as NBS. The ultimate reference standard can also be an independent reproducible standard, i.e., a standard which depends upon accepted values of natural physical constants. A typical example is the cesium beam type of microwave frequency standard.

10.7.2. There must be an up-to-date report for each reference standard (except independent reproducible standards) used in the calibration system (and for any subordinate standards or measuring and test equipment if their accuracy is such as to require supporting data). If calibration services are performed by a commercial laboratory or facility on a contract basis, copies of reports issued by them should be available.

10.7.3 All reports should be kept in a suitable file and should contain the following information:

a. Identification or serial number of standard to which the report pertains.

b. Conditions under which the calibration was performed (temperature, relative humidity, etc.).

c. Accuracy of standard (expressed in percentage or other suitable terms).

d. Deviation or corrections.

e. Report number.

f. Reports for the highest level standards of sources other than NBS or a Government laboratory should bear a statement that comparison has been made with national standards at periodic intervals using proper procedures and qualified personnel.

g. Corrections which must be applied if standard conditions of temperature, gravity, air buoyancy, etc., are not met or differ from those at place of calibration.

Contracts for calibration services should include agreements with the commercial facility for supply of copies of records or traceability of their reference standards.

10.8 Calibration Frequency Schedule

10.8.1 Calibration intervals for complex or sensitive laboratory instruments shall be those recommended by the respective manufacturers unless experience dictates otherwise. These intervals shall be listed on the schedule (Annex 1 - Section X) and are posted to the Calibration Control Card.

10.8.2 Calibration intervals for other equipment shall be established by the engineering laboratory and recorded in a similar manner.

10.8.3 Adherence to the schedule is mandatory. It is operated as follows: as the interval is established the Calibration Control Card is flagged at the appropriate place on the calendar printed at the top of the card using 12 different color flags for months, or using two flags for month and date. These cards are sorted and filed by sequence of date, so that the cards which mature on a given day identify which instruments are due for check.

10.9 Calibration Survey Checklist

The following is a checklist to be used as guidance in evaluation of the ability of state and other laboratories to control calibration procedures for instruments and test equipment.

Omission of one or several of these check-points does not necessarily imply that a given laboratory does not maintain required accuracy and precision in its calibration program, rather, the overall system must be appraised in light of its ability to provide such accuracy and precision.

INSTRUMENT CALIBRATION

CHECKLIST

DATE:_____

Location:

1. Is each piece of measuring or test equipment calibrated on a routine, periodic, basis against appropriate standards traceable to the National Bureau of Standards?

Yes_____No _____Remarks:_____

2. Are schedules for calibration established for each type of instrumentation equipment?

Yes_____No_____Remarks:_____

Have the following factors been considered in designing the schedule:

a. Severity of environment. Yes_____No_____
b. Severity of use. Yes_____No_____
c. Frequency of use. Yes_____No_____
d. Delicacy of the measuring equipment. Yes____No_____
e. Accuracy of measurement required. Yes____No_____
f. Calibration history. Yes_____No_____
g. Importance of the characteristic or condition measured. Yes____No_____
h. Sensitivity of the test methods used on the type of equipment.
Yes____No____Remarks:_____

3. Is calibration performed to: published standard practices, manufacturers' written instructions, or other approved instructions?

Yes_____No_____Remarks:_____

4. Is there positive identification of calibration status of each unit of equipment provided by color code, tag, label, stamp, etc.?

Yes_____No_____ Remarks:_____

5. Does the calibration status identification include the following?

a. Date of calibration. Yes_____No_____
b. Identification of technician performing calibration. Yes____No____
c. Due date for expiration of calibration status. Yes____No____

Remarks:_____

6. Is calibration conducted in an environment controlled to the extent necessary to assure continuation of measurements of the required accuracy?

Yes_____No_____Remarks:_____

7. Does environmental control include controls on:
   a. Temperature Yes_____No_____
   b. Humidity Yes_____No_____
   c. Vibration Yes_____No_____
   d. Dust and/or gaseous contaminants. Yes_____No_____
   e. Cleanliness. Yes_____No_____

   Remarks:_____

8. Are compensation corrective factors applied to calibration results obtained in an environment department from standard conditions?

Yes_____No_____Remarks:_____

9. Are there individual instrument calibration record cards: Yes_____No_____
Do they show:

   a. Description Yes_____No_____
   b. Identification number Yes_____No_____
   c. Calibration interval Yes_____No_____
   d. Reference to calibration instruction Yes_____No_____
   e. Identification of calibrating technician Yes_____No_____

   Remarks:_____

   f. Date of calibration Yes_____No_____
   g. Calibration expiration due date Yes_____No_____
   h. Actual values obtained Yes_____No_____
   I. Location of equipment Yes_____No_____

   Remarks: _____
_____

   Recommendations:_____

Signed_____

_____
Organization

NATIONAL INSTITUTE FOR OCCUPATIONAL
SAFETY AND HEALTH

DIVISION OF LABORATORIES AND CRITERIA DEVELOPMENT

CALIBRATION FREQUENCY SCHEDULE

Type	Minimum Frequency	Instruction Number or Calibration Method
+		

CALIBRATION STATUS TAG

Description:_____

Ident. No._____

Last Calibrated:_____

Calibrated By:_____

Next Cal. Due:_____

NOTE: Use of this instrument beyond the calibration due date is prohibited.

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31												
Jan.	Feb.	Mar.	Apr.	May	Jun.	Jul.	Aug.	Sept.	Oct.	Nov.	Dec.	

INSTRUMENT RECORD CARD

Instr. type_____ Ident. No._____ Instr. Code No._____

Model No._____ Ser. No._____

Date rec'd._____ Date insp._____ O.K.'d by_____

Checking interval_____ App'd. by _____

Location_____ Rec'd. by _____

Calibration Responsibility _____

Calibration Instruction Number

FRONT

Date checked	Checked By	Results of check	Date checked	Checked By	Results of check

REVERSE

STATISTICAL TECHNIQUES

Table of Contents

STATISTICAL TECHNIQUES

INTRODUCTION

This section of the manual attempts to gather various statistical techniques, with appropriate examples of problems, in one ready-reference source in order to aid the analyst in the solution of his problems. Much of the material has been taken from the texts shown on the last page of this manual. The remainder has been gathered from various other sources.

Another purpose of this section is to familiarize the analyst with statistical techniques which are being used or may be used in areas other than the one in which he is engaged.

I CONTROL CHARTS

A. Description and Theory

The control chart provides a tool for distinguishing the pattern of indeterminate (stable) variation from the determinate (assignable cause) variation. This technique displays the test data from a process or method in a form which graphically compares the variability of all test results with the average or expected variability of small groups of data - in effect, a graphical analysis of variance, and a comparison of the "within groups" variability versus the "between group" variability (see Figure XI-1 for the pattern of variation of data).

The data from a series of analytical trials can be plotted with the vertical scale in units of the test result and the horizontal scale in units of time or sequence of analyses. The average or mean value can be calculated and the spread (dispersion or range) can be established (Figures XI-5, 7 and 8).

The determination of appropriate control limits can be based on the capability of the procedure itself as known from past experience, or can be arbitrarily established at any desirable level. Common practice sets the limits at $\pm 3\ \sigma$ on each side of the mean. If the distribution of the basic data exhibits a normal form, the probability of results falling outside of the control limits can be readily calculated.

The control chart is actually a graphical presentation of quality control efficiency. If the procedure is "in control", the results will almost always fall within the established control limits. Further, the chart will disclose trends and cycles from assignable causes which can be corrected promptly. Chances of detecting small changes in the process average are improved when several values for a single control point (an $\bar{x}$ chart) are used. As the sample size increases, the chance that small changes in the average will not be detected is decreased.

The basic procedure of the control chart is to compare "within group" variability to "between group" variability. For a single analyst running a procedure, the "within group" may well represent one day's output and the "between group" represents between days or day-to-day variability. When several analysts or several instruments or laboratories are involved, the selection of the subgroup unit is critical. Assignable causes of variation should show up as "between group" and not "within group" variability. Thus, if the differences between analysts may be assignable causes of variation, their results should not be lumped together in a "within group" subgrouping.

B. Application and Limitations

In order for quality control to provide a means for separating the determinate from indeterminate sources of variation, the analytical method must clearly emphasize those details which should be controlled to minimize variability. A check list would include:

1. Sampling procedures
2. Preservation of the sample
3. Aliquoting methods
4. Dilution techniques
5. Chemical or physical separations and purifications
6. Instrumental procedures
7. Calculation and reporting results

The next step to be considered is the application of control charts for evaluations and control of these unit operations. Decisions relative to the basis for construction of a chart are required:

1. Choose method of measurement
2. Select the objective
   a. Precision (Figure XI-7) or accuracy evaluation (Figure XI-8)
   b. Observe test results, or the range of results
   c. Measurable quality characteristics (Figure XI-1)
3. Select the variable to be measured (from the check list)
4. Basis of subgroup, if used:
   a. Size

      A minimum subgroup size of n = 4 is frequently recommended, however the analyst will usually be dealing with samples of 2. The chance that small changes in the process average remain undetected decreases as the statistical sample size increases.

   b. Frequency of subgroup sampling

      Changes are detected more quickly as the sampling frequency is increased.
5. Control Limits

   Control limits (CL) can be calculated, but judgment must be exercised in determining whether or not the values obtained satisfy criteria established for the method, i.e., does the deviation range fall within limits consistent with the solution or control of the problem. After the mean ($\overline{X}$) of the individual results (X), and the mean of the range ($\overline{R}$) of replicate result differences (R) have been calculated, then CL can be calculated from data established for this purpose (Table XI-6).

Grand Mean $(\overline{\overline{X}}) = \overline{X}/n$

CL's on Mean $= \overline{\overline{X}} \pm A_2 \times \overline{R}$

Range $(\overline{R}) = \Sigma R \div n$

Upper Control Limit (UCL) on Range $= D_4 \overline{R}$ – D_4 and A_2 Factors are furnished on the $\overline{X}$ – R Worksheets for sample sizes 2 and 3·

Where: n – number of subgroups; A_2, D_4 and D_3 are obtained from Table XI-6, R may be calculated directly from the data, or from the standard deviation (σ) using factor d_2. The lower control limit for R is zero when $n \leq 6$.

The calculated CL's include approximately the entire data under "in control" conditions, and therefore, are approximately equal to $\pm$ 3 σ limits which are commonly used but which require more laborious calculations. Warning limits (WL) set at $\pm$ 2 σ limits (95%) of normal distribution serve a very useful function in quality control (see Figure XI-1 and XI-5). The upper warning limit (UWL) for the range can be calculated by:

$$UWL = \overline{R} + 2\sigma_R$$

$$UWL = \overline{R} \pm 2/3 (D_4 \overline{R} - \overline{R}) + \overline{R}$$

Where the subgrouping of size is n = 2, UWL reduces to

$$UWL = 2.51 \overline{R}$$

An example based on actual data follows showing, in detail, how control limits are calculated, step-by-step, following the above outline. The data computed on worksheets is then plotted on $\overline{X}$ & R charts.

Following this example, in section XI-I-D the construction of other types of control charts is examined in detail.

6. Example Application

Many times a laboratory will have much if not all of the information already available to develop and put into operation preliminary control charts. One such example is described below.

a. Program description

An analytical laboratory had been using the commercial Mercury flameless atomic absorption unit based on the work of Hatch and Ott. The instrument was adjusted mechanically for both 100% and 0% transmission. The reagent blank and a 1.0 μgm Hg standard were routinely run with each batch of samples. The data of Table 1 represents all runs on the blank and the reference standard and the few replicate sample determinations made. Most samples were reported based on a single determination. The data was extracted from the laboratory notebook and is given below (unedited).

b. Discussion of data

It would be easy (with the benefit of hindsight) to say that the available data should have been different in some way. In reality, the laboratory had good data on hand upon which to recommend a program for future use.

Almost all analytical control chart techniques are based upon either replicate (usually two) analyses or individual values. Replicate analyses allow one to learn more quickly about the "state of control" of the analyses. A subsequent shift to control charts based upon individual values may be appropriate.

For this discussion, only replicate analysis quality control techniques will be discussed. Standard references should be consulted for other types of control charts.

c. Reagent Blank Control Chart

The data of Table 1 indicates that normal practice for the specific laboratory was to run the blank determination at the beginning and the end of the day's run. Since there are only eight (8) sets of data, any control limits calculated would be very preliminary in nature. Normally, about twenty (20) sets of data are recommended for setting up the "trial control limits."

The calculations involved in setting up the control chart for this analytical situation are worked out on the work sheet following. (Exhibit I)

Since the out-of-control points have occurred in the past, no definitive action to determine their cause may be appropriate. Any future out-of-control sets of data should be studied to determine the "assignable cause" responsible. Once determined, the assignable cause should be removed.

All new data sets should be recorded on the worksheet both in the data table and on the graph (Exhibit II) as they are generated. This process provides "real-time" analysis and feedback for appropriate control actions.

The appropriate actions in response to out-of-control points are analytical or technical actions and are not statistical in nature. The skills and expertise of the analyst or technologist will enable him to determine and resolve the technical problem that caused the statistical "out-of-control" point to appear.

d. Reference Sample Control Chart

The data from Table I indicates that the laboratory made two determinations on the reference sample at the beginning of the run and one at the end of the day's run.

TABLE I

MERCURY QUALITY CONTROL DATA

8-13, Set 1

1.0[1] - 70.0[3]
1.0 - 70.0
B[2] - 97.0
SAMPLES
#5488 - 89.7
1.0 - 70.2
B - 97.1

8-16, Set 2

1.0 - 68.1
1.0 - 68.0
B - 97.5
SAMPLES
#5488 - 90.2

9-7, Set 3

B - 98.2
1.0 - 71.9
1.0 - 69.9
SAMPLES
1.0 - 72.1
B - 98.0

9-9, Set 4

B - 98.9
1.0 - 71.0
1.0 - 72.0
5670A - 97.8
SAMPLES
B - 99.0
1.0 - 74.0
5670B - 98.0
5670C - 98.0

9-13, Set 5

B - 99.5
1.0 - 69.5
1.0 - 72.0
SAMPLES
1.0 - 73.0
B - 99.0

9-23, Set 6

B - 98.9
1.0 - 69.1
1.0 - 69.2
SAMPLES
579A - 96.1
B - 96.0
C - 96.1
1.0 - 70.0
B - 99.0
5720A - 97.0
B - 97.1
C - 97.1

11-1, Set 7

B - 98.5
1.0 - 72.6
1.0 - 73.8
SAMPLES
1.0 - 75.0
B - 99.0

12-3, Set 8

B - 99.0
1.0 - 69.0
1.0 - 68.3
SAMPLES
1.0 - 69.4
B - 97.8

12-16, Set 9

B - 99.5
1.0 - 69.8
1.0 - 68.5
SAMPLES
1.0 - 69.0

12-31, Set 10

B - 100.0
1.0[4] - 84.5
1.0 - 68.9
1.0 - 69.0
SAMPLES
1.0 - 68.0
B - 100.0

NOTES:
1. 1.0 represents the 1.0 µgm Hg reference standard sample
2. B represents the reagent blank determination
3. Reading represents the observed percent transmission
4. Based upon the nonconformity to prior experience, a new stock solution of the 1.0 µg Hg working reference standard was made up and used.

Several approaches could be used to handle the data. A control chart could be set up on the two samples run at the beginning of each day's run. If there is significant drift in the day's run, such action is appropriate. If, on the basis of experience, no drifts occur during a day's run, the number of reference sample determinations could be reduced.

The control chart (Exhibits III and IV) is set up on the basis of the replicate samples at the beginning of the run. A specification limit for individual values can be set up based on the data. This "individual" specification limit can be used to judge acceptability of the single determination at the end of the day's run if it is determined that the basic process is in a state-of-control.

The ideal type of reference sample would be a stable (or stabilized) environmental sample. The reference sample should be subject to all the handling of a routine sample. The sample should contain representative interferences, etc. The level of the constituent being measured may or may not be known. If not known, a value is established at the time that the procedure is known to be well calibrated. At any time later, the determined concentration will be consistent with the value established during calibration.

e. Recycle Sample Control Chart

In many cases, it is not feasible or even possible to prepare a reference sample that meets the "ideal." When the reference sample cannot be an environmental sample, its determination may be made with a precision not achievable on routine environmental samples. It would be unfair and unrealistic to expect "real" samples to check as closely as spikes of pure material in distilled water.

The recycling of environmental samples provides a realistic measure of routine analytical precision. The determinations should be separate independent analyses. Submission of the recycle samples as "blinds" confirms the laboratory's or analyst's capability of reproducing his work independently.

Side-by-side duplicates cannot attest to the capability in performing independent analyses. It may be necessary to run the replicates at different times during the same run rather than on separate days due to sample instability and other problems.

Recycle sample control charts provide information only on routine precision rather than on accuracy since the "true" level of constituent is not known. The Range or R chart of the $\overline{X}$ and R chart previously demonstrated is normally used.

f. Instrumental Control Charts

There are many other areas where control charts can be applied to aid the control of variability. Control charts can be used to monitor; 1) the performance of the analytical instruments themselves, 2) the stability of laboratory calibration set-ups, and 3) the stability of the calibrated field sampling instruments. Such applications are straightforward adaptations of the principles discussed above. Considerable experience and literature is available in related applications. These are usually control charts for individual values rather than for multiple values.

EXHIBIT I

NIOSH – Division of Training

Practice Worksheet

Laboratory Quality Control $\bar{X}$-R Chart

Laboratory _____ Date Aug.-Dec.

Method of Test or Operation Hg / Blank Determination

Reference Value Blank (added) Increment of Measurement % T

Data

No.	X_1	X_2	X_3	$\bar{X}$	R
1	97.0	97.1		97.05	0.1
2	97.5	—			
3	98.2	98.0		98.1	0.2
4	98.9	99.0		98.95	0.1
5	99.5	99.0		99.25	0.5
6	98.9	99.0		98.95	0.1
7	98.5	99.0		98.75	0.5
8	99.0	97.8		98.4	1.2
9	99.5	—			
10	100.0	100.0		100.0	0
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Totals $\Sigma \bar{X}$ 789.45 ΣR 2.7

X_i = observed value R = largest – smallest
n = sets of values CL = control limit
Σ = summation WL = warning limit
U = upper L = lower
D_4 = 3.268 for n' = 2; 2.574 for n' = 3
A_2 = 1.880 for n' = 2; 1.023 for n' = 3
n' = number of values in the set

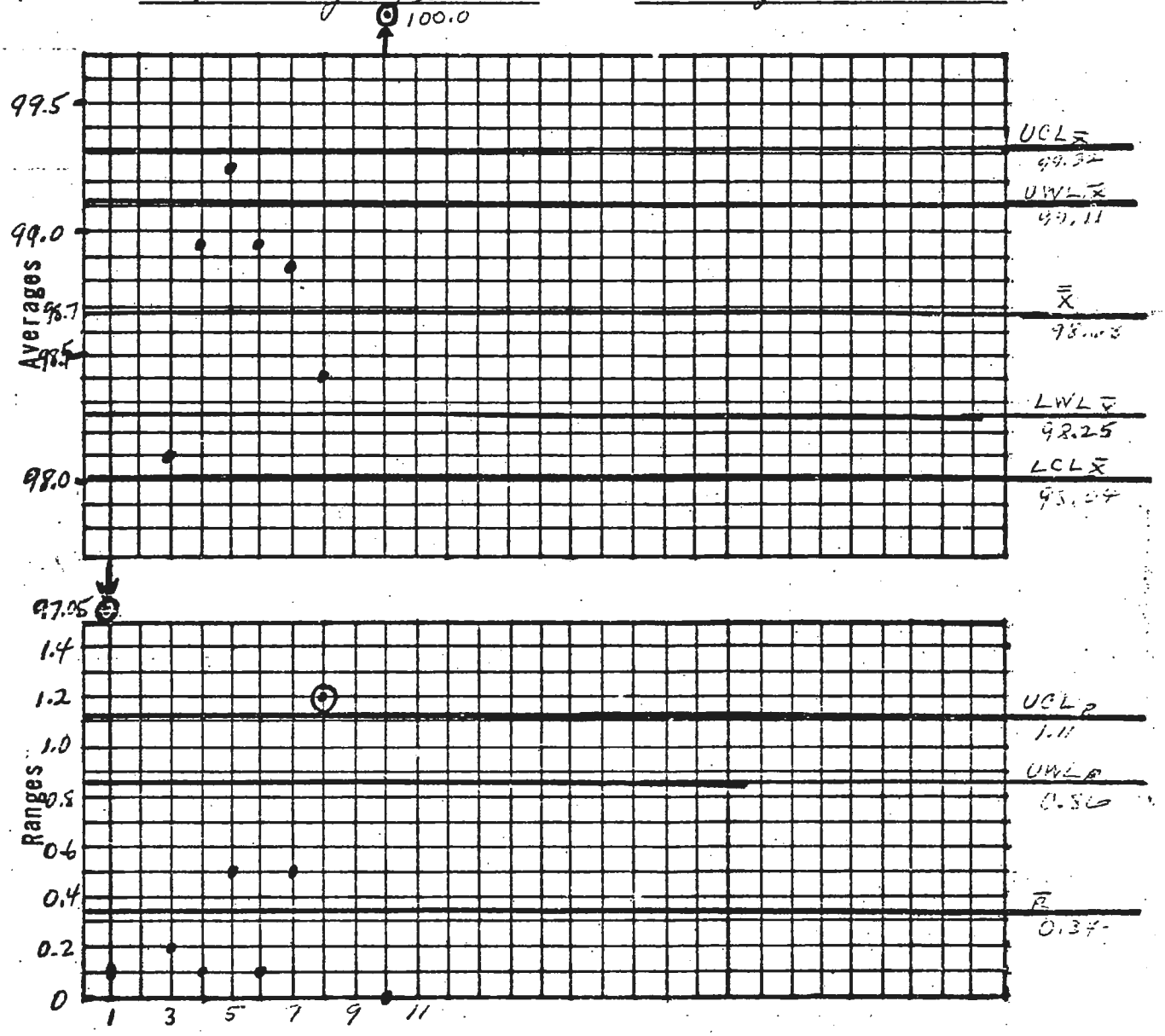
Calculations

1. $\bar{R} = \Sigma R \div n$
   0.34 = 2.7 ÷ 8
2. $UCL_R = D_4 \times \bar{R}$
   1.11 = 3.268 × 0.34
3. $UWL_R = 2/3(D_4\bar{R} - \bar{R}) + \bar{R}$
   0.86 = 2/3(1.11 − .34) + .34
   0.52
4. $\bar{\bar{X}} = \Sigma \bar{X} \div n$
   98.68 = 789.45 ÷ 8
5. $CL_{\bar{X}} = A_2 \times \bar{R}$
   0.64 = 1.88 × 0.34
6. $WL_{\bar{X}} = 2/3 \times CL_{\bar{X}}$
   0.43 = 2/3 × 0.64
7. $UCL_{\bar{X}} = \bar{\bar{X}} + CL_{\bar{X}}$
   99.32 = 98.68 + 0.64
8. $UWL_{\bar{X}} = \bar{\bar{X}} + WL_{\bar{X}}$
   99.11 = 98.68 + 0.43
9. $LWL_{\bar{X}} = \bar{\bar{X}} - WL_{\bar{X}}$
   98.25 = 98.68 − 0.43
10. $LCL_{\bar{X}} = \bar{\bar{X}} - CL_{\bar{X}}$
    98.04 = 98.68 − 0.64

EXHIBIT II

NIOSH – Division of Training
Practice Worksheet
Laboratory Quality Control $\bar{X}$-R Chart

Operation Mercury – Blank Date Aug –



Directions:	Value		
1. Draw $\bar{R}$ line	0.34	6. Draw UCL$_{\bar{X}}$ line	99.32
2. Draw UCL$_R$ line	1.11	7. Draw UWL$_{\bar{X}}$ line	99.11
3. Draw UWL$_R$ line	0.86	8. Draw LWL$_{\bar{X}}$ line	98.25
4. Plot R's as generated		9. Draw LCL$_{\bar{X}}$ line	98.04
5. Draw $\bar{\bar{X}}$ line	98.68	10. Plot $\bar{X}$'s as generated	

EXHIBIT III
NIOSH – Division of Training

Practice Worksheet
Laboratory Quality Control $\bar{X}$-R Chart

Laboratory _____ Date Aug – Dec

Method of Test or Operation Hg Reference Std 1.0 µg/ml

Reference Value 1.0 µg Increment of Measurement 1/10

Data

No.	X_1	X_2	X_3	$\bar{X}_{1+2}$	R_{1+2}
1	70.0	70.0	70.2	70.00	0.0
2	68.1	68.0		68.05	0.1
3	71.9	69.9	72.1	70.90	2.0
4	71.0	72.0		71.50	1.0
5	69.5	72.0	73.0	70.75	2.5
6	69.1	69.2	70.0	69.15	0.1
7	72.6	73.8	75.0	73.20	1.2
8	69.0	68.3	69.4	68.65	0.7
9	69.8	68.5	69.0	69.15	1.3
10	68.9	69.0	68.0	68.95	0.1
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Totals $\Sigma \bar{X}$ 700.50 ΣR 9.0

X_i = observed value R = largest – smallest
n = sets of values CL = control limit
Σ = summation WL = warning limit
U = upper L = lower
D_4 = 3.268 for n' = 2; 2.574 for n' = 3
A_2 = 1.880 for n' = 2; 1.023 for n' = 3
n' = number of values in the set

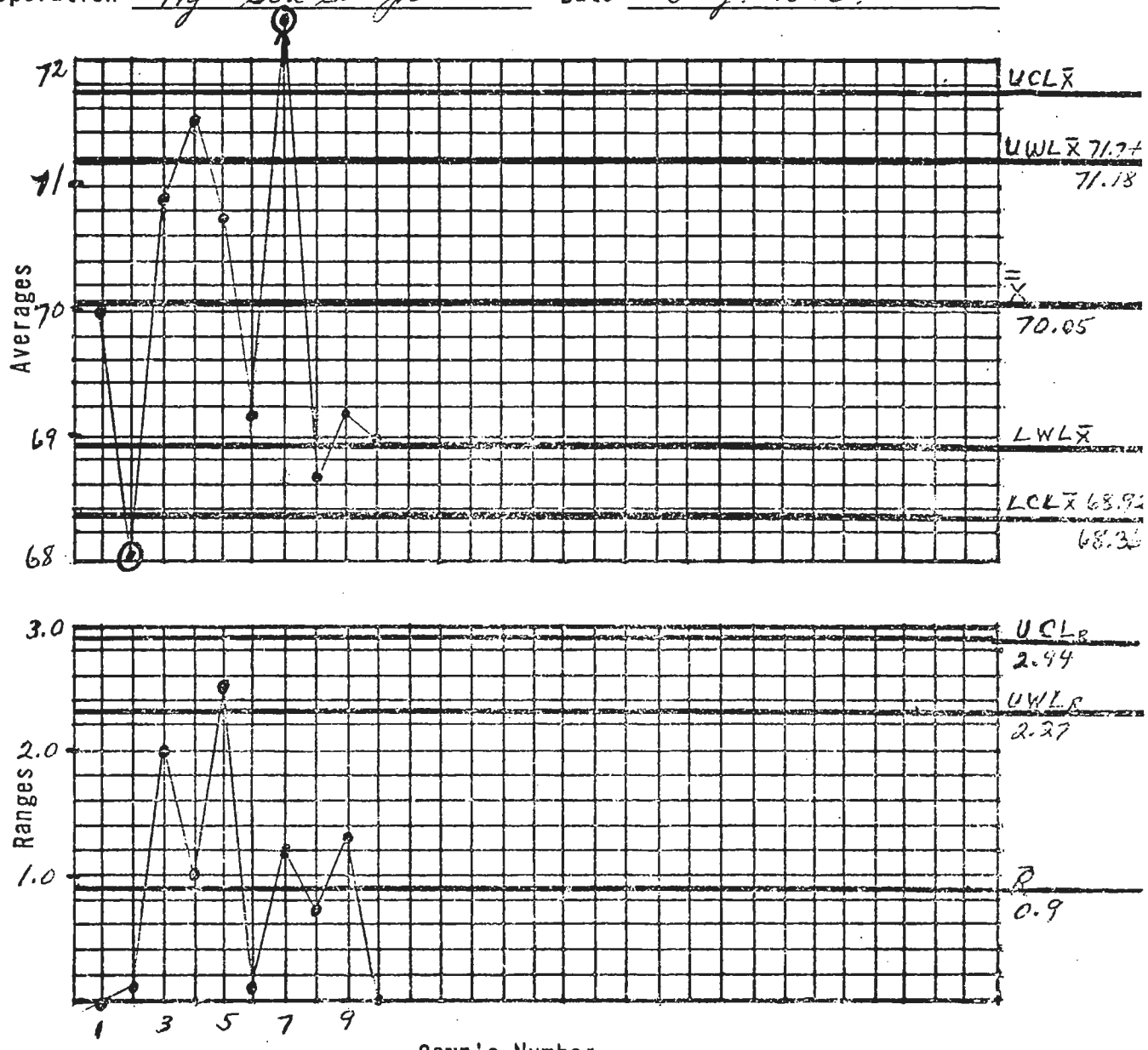
Calculations

1. $\bar{R} = \Sigma R \div n$
   $0.9 = 9.0 \div 10$
2. $UCL_R = D_4 \times \bar{R}$
   $2.94 = 3.268 \times 0.9$
3. $UWL_R = 2/3(D_4\bar{R} - \bar{R}) + \bar{R}$
   $2.27 = 2/3(2.94 - 0.9) + 0.9$
4. $\bar{\bar{X}} = \Sigma \bar{X} \div n$
   $70.05 = 700.50 \div 10$
5. $CL_{\bar{X}} = A_2 \times \bar{R}$
   $1.69 = 1.880 \times 0.9$
6. $WL_{\bar{X}} = 2/3 \times CL_{\bar{X}}$
   $1.13 = 2/3 \times 1.69$
7. $UCL_{\bar{X}} = \bar{\bar{X}} + CL_{\bar{X}}$
   $71.74 = 70.05 + 1.69$
8. $UWL_{\bar{X}} = \bar{\bar{X}} + WL_{\bar{X}}$
   $71.18 = 70.05 + 1.13$
9. $LWL_{\bar{X}} = \bar{\bar{X}} - WL_{\bar{X}}$
   $68.92 = 70.05 - 1.13$
10. $LCL_{\bar{X}} = \bar{\bar{X}} - CL_{\bar{X}}$
    $68.36 = 70.05 - 1.69$

EXHIBIT IV

NIOSH – Division of Training
Practice Worksheet
Laboratory Quality Control $\bar{X}$–R Chart

Operation Hg – Std Sample Date Aug. - Dec.



Directions:	Value		
1. Draw $\bar{R}$ line	0.9	6. Draw UCL$_{\bar{X}}$ line	71.74
2. Draw UCL$_R$ line	2.94	7. Draw UWL$_{\bar{X}}$ line	71.18
3. Draw UWL$_R$ line	2.27	8. Draw LWL$_{\bar{X}}$ line	68.92
4. Plot R's as generated		9. Draw LCL$_{\bar{X}}$ line	68.36
5. Draw $\bar{\bar{X}}$ line	70.05	10. Plot $\bar{X}$'s as generated	

C. Construction of Control Charts

1. Precision Control Charts

The use of range (R) in place of sample standard deviation (σ) is justified for limited sets of data $n \leq 10$ since R is approximately as efficient as σ for use in estimating σ ' and is easier to calculate. The average range ($\overline{R}$) can be calculated from accumulated results, or from a known or selected σ ($d_2 \sigma$). LCL = 0 when $n \leq 6$. (LCL = lower control limit).

The steps employed in the construction of a precision control chart for an automatic analyzer illustrate the technique (Table XI-8).

a. Calculate R for each set of side-by-side duplicate analyses of identical aliquots.

b. Calculate $\overline{R}$ from the sum or R values divided by the number (n) of sets of duplicates.

c. Calculate the upper control limit (UCL) for the range:

$$UCL = D_4 \overline{R}$$

Since the analyses are in duplicates, $D_4 = 3.27$ (from Table XI-6).

d. Calculate the upper warning limit (UWL):

$$UWL = \overline{R} + 2\sigma_R = \overline{R} + 2/3 (D_4 \overline{R}) = 2.51 \overline{R}$$ (D_4 from Table XI-6)

which corresponds to the 95% confidence limits.

e. Chart $\overline{R}$, UWL and UCL on an appropriate scale which will permit addition of new results obtained as shown in Figure XI-7 and Table XI-8.

f. Plot results (R) and take action on out-of-control points.

D. Accuracy Control Charts - Mean or Nominal Value Basis

$\overline{X}$ charts simplify and render more exact the calculation of Control Limits as the distribution of data which conforms to the normal curve can be completely specified

by $\overline{X}$ and σ. Stepwise construction of an accuracy control chart for the automatic analyzer based on duplicate sets of results obtained from consecutive analysis of knowns serves as an example (Table XI-9).

a. Calculate $\overline{X}$ for each duplicate set

b. Group the $\overline{X}$ values into a consistent reference scale (in groups by orders of magnitude for the full range of known concentrations).

c. Calculate the UCL and the lower control limit (LCL) by the equation:

$CL = \pm A_2 \overline{R}$ (A_2 from Table XI-6).

d. Calculate the upper and lower Warning Limit (WL) by the equation:

$$WL = \pm 2/3\ A_2 \overline{R}$$

e. Chart CL's and WL's as deviations from the standard. Thus, the standard value for this chart is set at zero as shown in Figure XI-8 ("order" related to consecutive, or chronological order of the analyses) and Table XI-9.

f. Plot the difference between the nominal value and $\overline{X}$ and take action on points which fall outside of the control limits.

D. 2. Control Charts for Individuals

In many instances a rational basis for subgrouping may not be available, or the analysis may be so infrequent as to require action on the basis of individual results. In such cases X charts of individual values are employed. However, the Control Limits must come from some subgrouping to obtain a measure of "within group" variability. This alternative has the advantage of displaying each result with respect to tolerance, or specification limits (Figures XI-1, 5 and 6.) The disadvantages must be recognized when considering this approach.

a. The chart does not respond to changes in the average.

b. Changes in dispersion are not detected unless an R chart is included, based on a subgrouping.

c. The distribution of results must be approximately normal for the control limits to be valid.

D. 3. Moving Averages and Ranges

The $\overline{X}$ control chart is more efficient than an X chart for disclosing moderate changes in the average as the subgroup size increases. A logical compromise between the X and $\overline{X}$ approach would be application of the moving average. For a given series of analyses, the moving average is plotted.

Such a set of data is shown in Table XI-7. The moving range serves well as a measure of acceptable variation when no rational basis for subgrouping is available or when results are infrequent or expensive to gather.

The techniques described earlier are not applicable to computation of moving average or moving range control limits.

E. Other Control Charts for Variables

Although the Standard $\overline{X}$ and R control chart for variables is the most common, it does not always do the best job. Several examples follow where other charts are more applicable.

1. Variable Subgroup Size

The standard $\overline{X}$ and R chart is applicable for a constant size subgroup on n = 2, 3, 4, 5. In some cases such a situation does not exist. Control limit values must be calculated for each sample size. Plotting is done in the usual manner with the control limits drawn in for each subgroup depending on its size.

2. R or σ Charts

In some situations the dispersion is equal over a range of assay values. In this case, a control chart for either range or standard deviation where no average, $\overline{X}$, is appropriate. When the dispersion is a function of concentration, control

limits can be expressed in terms of a percentage of the mean. In practice such control limits would be given as in the example below.

$\pm$ 5 units/liter for 0-100 units/liter concentration

$\pm$ 5% for $>$ 100 units/liter concentration

An alternative procedure involves transformation of the data. For example, logarithms would be the appropriate transformation, when the standard deviation is proportional to the mean.

3. $\bar{X}$ and σ Charts

If the subgroup size exceeds 10, the Range Chart becomes inefficient. The use of a σ chart would then be appropriate. Where the cost of obtaining the test data is high, the increase in efficiency using σ rather than R may be worthwhile.

II USING THE RANGE $\bar{R}$ TO ESTIMATE THE STANDARD DEVIATION σ

Instead of computing the standard deviation σ by calculating the square root of the average of the squared deviations from the mean, we may estimate it from the average range, $\bar{R}$ using Table XI-1.

If we have 10 samples of 2 each, of analyses of toluene on charcoal corrected to 10 L, the results being as follows:

Sample Pair No.	Range R Between the Values in the Pair in mg/L
1	.18
2	.04
3	.20
4	.14
5	.13
6	.03
7	.11
8	.18
9	.07
10	.13
Total	1.21 $\bar{R}$ = .121

Procedure	Example
(1) Compute the average Range $\bar{R}$.	(1) $\frac{1.21}{10} = .121$
(2) Find d_m for samples of 2 in Table XI-1	(2) $d_{m_2} = 1.128$
(3) Compute estimate of σ	(3) $\sigma = \frac{.121}{1.128} = .1072$

The need often arises for determining whether or not differences in results based on experimental data are statistically significant. The following examples are representative of the more usual problems which arise. The form of solution depends upon the kind and amount of background information available.

III TEST FOR DIFFERENCE BETWEEN SAMPLE MEAN ($\bar{X}'$) AND MEAN OF POPULATION ($\bar{X}$)

from which the sample is believed to have been drawn when the population standard deviation (σ') is known[1].

Example:

A large number of samples of urine was checked for mercury which indicated an average content of 5.15 g mercury/liter of urine, and a standard deviation of .25. Later, a second sample was checked from the same individuals with the following results:

Specimen No.	Hg/Liter
1	5.02
2	4.87
3	4.95
4	4.88
5	5.01
6	4.93
7	4.91
8	5.09
9	4.96
10	4.89
11	5.06
12	4.85
Total	59.42
Average	4.95

The question posed is: is the second sample significantly different in average than the first sample taken?

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Solution: The difference between past performance (5.15 μg/Hg/Liter) and the average of the sample from the new lot (4.95 μg/Hg/Liter) is the key figure. This difference is 5.15 - 4.95 = 0.20. To interpret this difference, use is made of the fact that the natural pattern of the averages coming from a controlled environment is well-known and can be predicted from the past variability (σ'). The observed difference is compared with the expected pattern as a means of determining how unusual the observed difference is by finding the value of the ratio.

$$t = \frac{\bar{X} - \bar{X}'}{\frac{\sigma'}{\sqrt{n}}}$$

Substituting the proper values,

$$t = \frac{4.95 - 5.15}{0.25 / \sqrt{12}} = 2.75$$

To determine how significant this value is, it is necessary to consult a table of t values (XI-2). When the value of σ' is well established from any past observations, the table of t values is entered on the lowest line, = DF - ∞. Find among the values of t given, the one nearest to 2.75 (only plus values are given). The value falls between 2.576 and 3.291 which are in columns with probabilities of .01 and .001 respectively. Interpolation is usally not necessary, and in this case, it is obvious that the probability of having so great a difference by chance alone is considerably less than 0.01.

Conclusion:

The difference is classified as signifcant since odds of 100 to 1 are regarded as too great to be attributed to sampling variation alone. The alternative is to believe that a real change has occurred in the environment.

IV TEST FOR DIFFERENCE BETWEEN SAMPLE MEAN ($\bar{X}$) AND POPULATION MEAN ($\bar{X}'$)

when the population standard deviation σ') is unknown and must be estimated from the sample.[2]

Records indicated that the average free silica found in a certain location over a period of a year was 49.95%. No record of individual measurements was kept. After a change was made in plant operations, sixty-one new samples were taken and analysis showed an average percentage of 54.62% and a standard deviation of 5.30%.

The question arises: Is the average percentage of free silica significantly different under the new operating conditions than before?

	Procedure		Example
(1)	Estimate the standard deviation computing $\hat{\sigma}' = \sigma \sqrt{\frac{n}{n-1}}$	(1)	$\hat{\sigma}' = 5.3 \sqrt{1.008} = 5.34$
(2)	Compute the ratio: $t = \frac{\bar{X} - \bar{X}'}{\hat{\sigma}'/\sqrt{n}}$	(2)	$t = \frac{54.62 - 49.95}{5.34 / \sqrt{61}} = 6.83$
(3)	Enter the table of t values at DF = 60 (n-1)	(3)	$t_{60} = < .005 \therefore 6.83 >$ any value given
(4)	if $\frac{X - X'}{\hat{\sigma}' / \sqrt{n}} > t$ decide that the average of the new samples is different from that of the standard; otherwise, that there is no reason to believe that they differ.	(4)	Conclude that the last samples taken are significantly higher in free silica, since a chance difference in average percentage 54.62 - 49.95 = 4.67 is so unlikely.

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V X^2 FOR DIFFERENCE BETWEEN SAMPLE VARIABILITY (σ') WITH A KNOWN POPULATION VARIABILITY.[3]

A decrease in the uniformity of test samples coming from the same source may be fully as important as a shift in the average value. If the test results have been close, such a loss of precision may cause a serious number of questionable analyses. On the other hand, detection of increased uniformity and the reasons for it may pave the way to a permanent improvement in quality.

Example: For the urine specimens tabulated in III, the sample σ, if computed, is found to be 0.036 μg/Hg/Liter. The previous variability, recorded over a period, was $\sigma' =$ 0.25 μg/Hg/Liter. Does the low value of 0.036 indicate that the new sample is significantly more uniform?

Procedure		Example	
(1)	Compute $X^2 = n\left(\frac{\sigma}{\sigma'}\right)^2$	(1)	$X^2 = 12\left(\frac{.036}{.25}\right)^2 = 0.25$
(2)	Look up the value nearest .25 for n - 1 = 11 degrees of freedom in the X^2 table (Table XI-4).	(2)	Since the value falls to the left of all those tabulated, we conclude that the probability is well above .99.
(3)	For X^2, a very low P (below 0.01) is interpreted as meaning that the sample σ is significantly large. A high P (above 0.99) is interpreted as meaning that the sample σ is significantly small.	(3)	We conclude the sample σ is significantly small. Values in the new sample were significantly more uniform in mercury content than the previous sample.

Note: Values of X^2 are given for DF up to 30. For larger samples, use can be made of the fact that the distribution of t is approaching the normal curve. A test with

$$t = \frac{(\sigma - \sigma')\sqrt{2n}}{\sigma'}$$

may be made, finding the probabilities of the table of t values on line DF = ∞

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V-a ALTERNATE METHOD FOR TESTING FOR DIFFERENCE BETWEEN SAMPLE VARIABILITY AND A KNOWN POPULATION VARIABILITY.

	Procedure		Example
(1)	Compute: $G = \frac{\sigma}{\sigma'}$	(1)	$\frac{\sigma}{\sigma'} = \frac{.036}{.25} = .1440$
(2)	Look up - G.95 in Table XI-5	(2)	For N = 12 G.95 = 1.28
(3)	Compare - $1.28 > .1440$	(3)	We conclude there is a significant difference.

VI t TEST FOR DIFFERENCE BETWEEN TWO SAMPLE MEANS ($\bar{X}_1$ and $\bar{X}_2$) BASED ON INDEPENDENT RANDOM SAMPLES,

when σ' is known and common to both sets of data.[4]

A battery manufacturer has two plants. Eight blood samples are drawn from similar production areas in each plant with results shown below. The population standard deviation was estimated from experience to be .22 ug Pb/100 g blood.

Plant A μg Pb/100 g blood	Plant B μg Pb/100 g blood
12.53	12.53
12.37	13.22
12.48	13.01
12.77	12.97
12.52	12.96
12.81	13.03
12.76	12.82
12.52	13.43
Total 100.76	Total 103.97
$\bar{X}_1$ = 12.60	$\bar{X}_2$ = 13.00

Procedure	Example
(1) There is no prior knowledge of the population value corresponding to the measure being tested. The difference under study is between two quantities, each of which has some sampling error. The error contributed by each of the samples is inversely proportional to the sample size. Adapt the test ratio of III to show this:	(1) $t = \dfrac{\bar{X}_1 - \bar{X}_2}{\sigma' \sqrt{(n_1 + n_2) / n_1 n_2}}$
(2) Compute: $t = \dfrac{\bar{X}_1 - \bar{X}_2}{\sigma' \sqrt{\dfrac{(n_1 + n_2)}{n_1 n_2}}}$	(2) $t = \dfrac{12.60 - 13.00}{0.22 \sqrt{(8 + 8) / 64}} = -3.64$

[4]Adapted by permission from QUALITY CONTROL HANDBOOK, edited by J. M. Juran, 2 Ed. Copyright® 1962 by McGraw-Hill, Inc. Used with permission of McGraw-Hill Book Co.

(3) Look up for degrees of freedom = ∞ (for any specified value) in Table XI-2.

(4) If $t \geq t$ df ∞ decide that $\overline{X}_1$ and $\overline{X}_2$ differ with regard to their average lead levels.

(3) $t = 3.64$ is larger than the right hand value; therefore, the probability is less than .001 for a two-tailed test.

(4) Conclude that the difference in lead content is significant.

VII t TEST FOR DIFFERENCE BETWEEN TWO SAMPLE MEANS ($\bar{X}_1$ and $\bar{X}_2$) WHERE σ' IS UNKNOWN BUT BELIEVED TO BE THE SAME FOR THE TWO POPULATIONS.[5]

The same samples of toluene on charcoal were analyzed by two different laboratories with the following results:

	Lab A	Lab B
No. of samples analyzed	10	10
Avg. mg/1 = $\bar{X}$ mg/l	1.609	1.530
Variability mg σ	.0844	.1104

The question to be answered: Can it be stated confidently that the two laboratories' analyses results are significantly different? The value 1.609 - 1.530 is the difference under consideration.

	Procedure	Example
(1)	Compute $t = \dfrac{\bar{X}_1 - \bar{X}_2}{\sqrt{(\sigma_2^2 + \sigma_2^2) / (n - 1)}}$	(1) $t = \dfrac{1.609 - 1.530}{\sqrt{\frac{.0192}{9}}} = 2.858$
(2)	Compute DF = 2 (n - 1)	(2) DF - 2 (9) = 18
(3)	Look up the value 2.858 in the t table	(3) The probability is very small -- less than .025 of finding by chance a difference of .106 between the two laboratories.

[5]Adapted by permission from QUALITY CONTROL HANDBOOK, edited by J. M. Juran, 2 Ed. Copyright® 1962 by McGraw-Hill, Inc. Used with permission of McGraw-Hill Book Co.

VIII F TEST FOR DIFFERENCE IN VARIABILITY (σ_1^2 and σ_2^2) IN TWO SAMPLES.[6]

Using the data from the preceding test, determine whether the Laboratory results differ in variability or are the results of Laboratory A more consistent than those of Laboratory B?

Procedure	Example
(1) Compute: $F = \frac{\sigma_1^2}{\sigma_2^2}$	(1) $F = \frac{.021}{.0071} = 1.7042$
(2) Look up $F_1 - \frac{.05}{2}$ for DF n - 1 for numerator and DF n - 1 for denominator in Table XI-5a	(2) $n_A - 1 = 9$ $n_B - 1 = 9$ F.975 (9,9) = 3.18
(3) If $F >$ F.975 (9, 9) or $F < \frac{1}{\text{F.975 (9, 9)}}$ decide that the two sets of results are different in their variability; otherwise assume they are not different.	(3) F.975 (9, 9) = 3.18 $\frac{1}{\text{F.975 (9, 9)}} = .3144$ Since F (1.7042) is not larger then 3.18 nor smaller than .3144 there is no reason to believe the two sets of test results differ significantly in variability.

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IX TESTS OF NORMALITY [7]

Many statistical tests are based upon the assumption that the population from which a sample is drawn is normally distributed. It is often wise, if possible, to test this assumption. Various procedures are available for making this test.

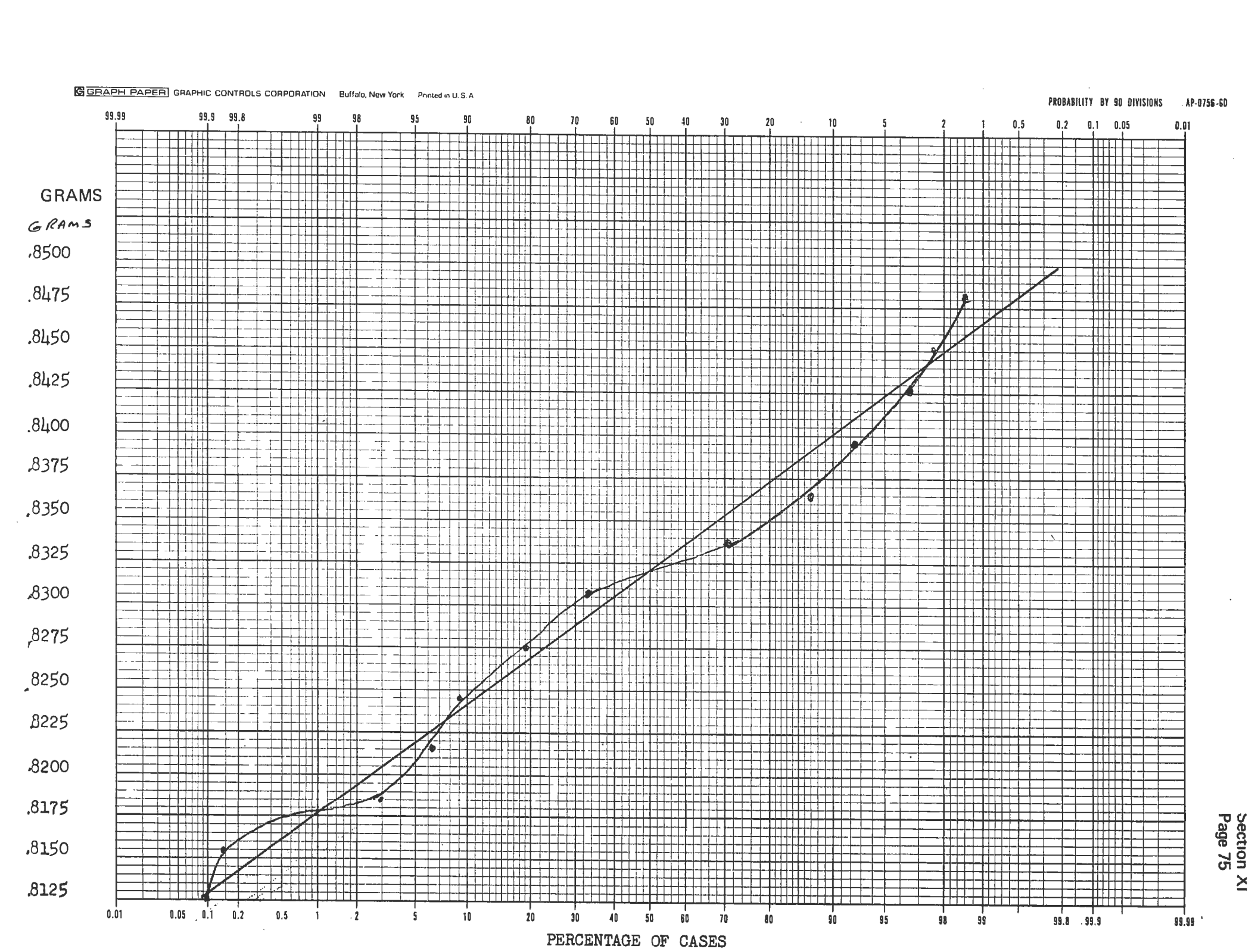
One method is to construct a histogram, if the sample is large enough, and then to plot a normal curve having the same mean and standard deviation with the histogram to see how well the normal curve fits.

This is an imprecise method at best and, unless there is an extremely good fit of a normal curve laid over the resulting histogram or polygon, the cumulated distribution should be plotted on normal probability paper before proceeding. As an example, the following table gives the frequency distribution of the results of a series of 145 similar tests:

Grams	Observed Frequency	Percentage	Cumulative
.8485	2	1.37%	1.37
.8455	1	.68	2.05
.8425	2	1.37	3.42
.8395	6	4.13	7.55
.8365	7	4.82	12.37
.8335	23	15.86	28.23
.8305	55	37.93	66.16
.8275	21	14.48	80.64
.8245	14	9.65	90.29
.8215	5	3.44	93.73
.8185	4	2.75	96.48
.8155	3	2.06	98.54
.8125	2	1.37	99.91
Total	145	99.91	99.91

[7] Adapted from Table 4, QUALITY CONTROL AND INDUSTRIAL STATISTICS, A. J. Duncan, Revised Edition. Used with permission of Richard D. Irwin, Inc.

This data has been plotted on the following page. Now, having looked at the fit, we decide how good it is. The graph does not really tell whether the departure from fit is significant. The most accurate way of testing for normality is to use the X^2 test for normality of data. However, the calculations are tedious and time consuming for desk calculator computation. Standard X^2 computer programs are commonly available. However, judgment must be used to weigh the cost of getting an accurate determination against the value of the information.


GRAPH PAPER GRAPHIC CONTROLS CORPORATION Buffalo, New York Printed in U.S.A.
PROBABILITY BY 90 DIVISIONS AP-0756-GD
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GRAMS
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0.01 0.05 0.1 0.2 0.5 1 2 5 10 20 30 40 50 60 70 80 90 95 98 99 99.8 99.9 99.99
PERCENTAGE OF CASES

X A SHORT CUT TO THE ANALYSIS OF VARIANCE[2]

Tukey's Gap and Straggler Test offers a test which involves the following simple calculations:

The results of analyses for mercury in urine of aliquots of the same urine samples by three different laboratories are as follows:

Sample No.	Lab A	mg/l Lab B	Lab C
1	.126	.100	.09
2	.062	.048	.04
3	.050	.033	.03
4	.060	.045	.05
5	.102	.090	.06
6	.158	.178	.11
7	.046	.038	.03
8	.038	.045	.03
9	.042	.043	.02
10	.034	.033	.03
Σ =	.718	.653	.490
R =	.124	.145	.090

The question to be answered is: Is there a significant difference between Laboratories at the 1% significance level?

	Procedure		Example			
(1)	Compute the sum and range of each column of data.	(1)		Lab A	B	C
			Column sum	.718	.653	.490
			Column R	.124	.145	.090
(2)	Compute the range of sums.	(2)	.718 - .490 = .228			
(3)	Compute the sum of ranges.	(3)	.124 + .145 + .090 = .359			

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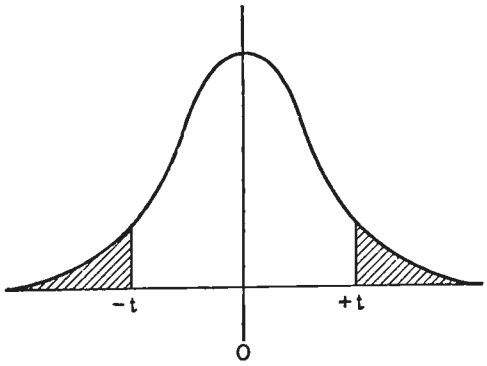
(4)	Multiply the sum of ranges by a critical value obtained from Table XI-3.	(4)	1.55 (.359) = .5564
(5)	If the range of sums (2) is greater than the product found in item (4), the question under consideration is significant at the stated level associated with the critical value. Otherwise there is no significant difference.	(5)	Since the range of sums (.228) is less than .5564, there is not a significant difference between laboratory analytical results.

TABLE XI-1

FACTORS FOR ESTIMATING THE STANDARD DEVIATION FROM THE RANGE

Size of Sample	$\frac{1}{d_m}$
2	.8865
3	.5907
4	.4857
5	.4299
6	.3946
7	.3698
8	.3512
9	.3367
10	.3249
12	.3069
16	.2831



Distribution of t*

Values of t corresponding to certain selected probabilities (*i.e.*, tail areas under the curve). To illustrate: the probability is 0.05 that a sample with 20 degrees of freedom would have $t = 2.086$ or larger.

DF	Probability							
	0.80	0.40	0.20	0.10	0.05	0.02	0.01	0.001
1	0.325	1.376	3.078	6.314	12.706	31.821	63.657	636.619
2	0.289	1.061	1.886	2.920	4.303	6.965	9.925	31.598
3	0.277	0.978	1.638	2.353	3.182	4.541	5.841	12.941
4	0.271	0.941	1.533	2.132	2.776	3.747	4.604	8.610
5	0.267	0.920	1.476	2.015	2.571	3.365	4.032	6.859
6	0.265	0.906	1.440	1.943	2.447	3.143	3.707	5.959
7	0.263	0.896	1.415	1.895	2.365	2.998	3.499	5.405
8	0.262	0.889	1.397	1.860	2.306	2.896	3.355	5.041
9	0.261	0.883	1.383	1.833	2.262	2.821	3.250	4.781
10	0.260	0.879	1.372	1.812	2.228	2.764	3.169	4.587
11	0.260	0.876	1.363	1.796	2.201	2.718	3.106	4.437
12	0.259	0.873	1.356	1.782	2.179	2.681	3.055	4.318
13	0.259	0.870	1.350	1.771	2.160	2.650	3.012	4.221
14	0.258	0.868	1.345	1.761	2.145	2.624	2.977	4.140
15	0.258	0.866	1.341	1.753	2.131	2.602	2.947	4.073
16	0.258	0.865	1.337	1.746	2.120	2.583	2.921	4.015
17	0.257	0.863	1.333	1.740	2.110	2.567	2.898	3.965
18	0.257	0.862	1.330	1.734	2.101	2.552	2.878	3.922
19	0.257	0.861	1.328	1.729	2.093	2.539	2.861	3.883
20	0.257	0.860	1.325	1.725	2.086	2.528	2.845	3.850
21	0.257	0.859	1.323	1.721	2.080	2.518	2.831	3.819
22	0.256	0.858	1.321	1.717	2.074	2.508	2.819	3.792
23	0.256	0.858	1.319	1.714	2.069	2.500	2.807	3.767
24	0.256	0.857	1.318	1.711	2.064	2.492	2.797	3.745
25	0.256	0.856	1.316	1.708	2.060	2.485	2.787	3.725
26	0.256	0.856	1.315	1.706	2.056	2.479	2.779	3.707
27	0.256	0.855	1.314	1.703	2.052	2.473	2.771	3.690
28	0.256	0.855	1.313	1.701	2.048	2.467	2.763	3.674
29	0.256	0.854	1.311	1.699	2.045	2.462	2.756	3.659
30	0.256	0.854	1.310	1.697	2.042	2.457	2.750	3.646
40	0.255	0.851	1.303	1.684	2.021	2.423	2.704	3.551
60	0.254	0.848	1.296	1.671	2.000	2.390	2.660	3.460
120	0.254	0.845	1.289	1.658	1.980	2.358	2.617	3.373
∞	0.253	0.842	1.282	1.645	1.960	2.326	2.576	3.291

* Reproduced in abridged form from Table III of Fisher and Yates, "Statistical Tables for Biological, Agricultural, and Medical Research," published by Oliver & Boyd, Ltd., Edinburgh, by permission of the authors and publishers.

Critical Factors for One-way (Balanced) Divisions into Groups*

The four entries are, respectively, for:

Range with 5% risk Gap with 5% risk
Range with 1% risk Gap with 1% risk

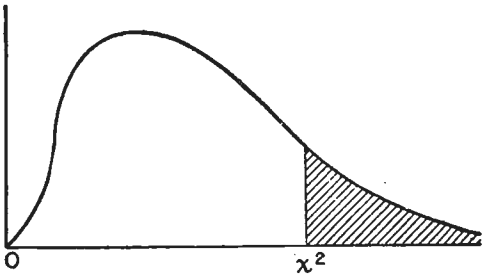
and are to be multiplied by the sum of the ranges within groups. (Risks are on per experiment basis.)

Number of groups = number of ranges summed	Number in group = number per range																	
	2		3		4		5		6		7		8		9		10	
2	3.43	3.43	1.91	1.91	1.63	1.63	1.53	1.53	1.50	1.50	1.49	1.49	1.49	1.49	1.50	1.50	1.52	1.52
	7.92	7.92	3.14	3.14	2.47	2.47	2.24	2.24	2.14	2.14	2.10	2.10	2.08	2.08	2.09	2.09	2.09	2.09
3	2.37	1.76	1.44	1.14	1.25	1.02	1.19	0.98	1.18	0.96	1.17	0.96	1.17	0.97	1.18	0.98	1.20	0.99
	4.42	3.25	2.14	1.73	1.74	1.47	1.60	1.37	1.55	1.32	1.53	1.33	1.52	1.33	1.53	1.34	1.55	1.35
4	1.98	1.18	1.13	0.81	1.01	0.74	0.94	0.72	0.92	0.71	0.92	0.71	0.94	0.72	0.96	0.73	0.97	0.74
	2.96	1.96	1.57	1.19	1.33	1.04	1.24	0.98	1.21	0.96	1.21	0.96	1.21	0.97	1.22	0.98	1.23	0.99
5	1.40	0.88	0.94	0.63	0.85	0.58	0.81	0.56	0.80	0.56	0.80	0.56	0.81	0.57	0.82	0.58	0.84	0.59
	2.06	1.39	1.25	0.91	1.08	0.80	1.02	0.77	0.99	0.76	0.99	0.76	0.99	0.77	1.00	0.77	1.01	0.78
6	1.16	0.70	0.81	0.52	0.75	0.48	0.69	0.47	0.69	0.46	0.69	0.47	0.70	0.47	0.71	0.48	0.72	0.49
	1.69	1.07	1.04	0.73	0.94	0.66	0.86	0.63	0.85	0.62	0.85	0.63	0.85	0.63	0.85	0.64	0.86	0.65
7	1.00	0.58	0.70	0.44	0.63	0.40	0.61	0.40	0.61	0.40	0.61	0.40	0.62	0.41	0.63	0.41	0.63	0.42
	1.39	0.87	0.89	0.61	0.78	0.55	0.75	0.54	0.74	0.53	0.74	0.53	0.74	0.54	0.75	0.55	0.76	0.55
8	0.87	0.50	0.62	0.38	0.57	0.35	0.55	0.34	0.55	0.34	0.55	0.35	0.55	0.35	0.56	0.36	0.57	0.37
	1.20	0.74	0.78	0.53	0.69	0.48	0.66	0.47	0.65	0.46	0.65	0.46	0.66	0.47	0.66	0.48	0.67	0.48
9	0.78	0.44	0.56	0.33	0.51	0.31	0.50	0.30	0.49	0.30	0.50	0.31	0.50	0.31	0.51	0.31	0.52	0.32
	1.03	0.63	0.71	0.46	0.62	0.44	0.59	0.43	0.59	0.42	0.59	0.42	0.59	0.42	0.60	0.43	0.61	0.43
10	0.70	0.39	0.51	0.30	0.46	0.28	0.45	0.27	0.45	0.27	0.45	0.28	0.46	0.28	0.47	0.28	0.47	0.29
	0.91	0.56	0.62	0.41	0.57	0.38	0.54	0.37	0.53	0.36	0.54	0.37	0.54	0.37	0.55	0.38	0.55	0.38

* Reproduced with permission from Table 1 of John W. Tukey, "Quick and Dirty Methods in Statistics. II. Simple Analyses for Standard Designs," Quality Control Conference Papers, Fifth Annual Convention, American Society for Quality Control, May 23 and 24, 1951, p. 194.

Distribution of χ^2 *

Values of χ^2 corresponding to certain selected probabilities (*i.e.*, tail areas under the curve). To illustrate: the probability is 0.05 that a sample with 20 degrees of freedom, taken from a normal distribution, would have $\chi^2 = 31.410$ or larger.



DF	Probability										
	0.99	0.98	0.95	0.90	0.80	0.20	0.10	0.05	0.02	0.01	0.001
1	0.0^3157	0.0^3628	0.00393	0.0158	0.0642	1.642	2.706	3.841	5.412	6.635	10.827
2	0.0201	0.0404	0.103	0.211	0.446	3.219	4.605	5.991	7.824	9.210	13.815
3	0.115	0.185	0.352	0.584	1.005	4.642	6.251	7.815	9.837	11.341	16.268
4	0.297	0.429	0.711	1.064	1.649	5.989	7.779	9.488	11.668	13.277	18.465
5	0.554	0.752	1.145	1.610	2.343	7.289	9.236	11.070	13.388	15.086	20.517
6	0.872	1.134	1.635	2.204	3.070	8.558	10.645	12.592	15.033	16.812	22.457
7	1.239	1.564	2.167	2.833	3.822	9.803	12.017	14.067	16.622	18.475	24.322
8	1.646	2.032	2.733	3.490	4.594	11.030	13.362	15.507	18.168	20.090	26.125
9	2.088	2.532	3.325	4.168	5.380	12.242	14.684	16.919	19.679	21.666	27.877
10	2.558	3.059	3.940	4.865	6.179	13.442	15.987	18.307	21.161	23.209	29.588
11	3.053	3.609	4.575	5.578	6.989	14.631	17.275	19.675	22.618	24.725	31.264
12	3.571	4.178	5.226	6.304	7.807	15.812	18.549	21.026	24.054	26.217	32.909
13	4.107	4.765	5.892	7.042	8.634	16.985	19.812	22.362	25.472	27.688	34.528
14	4.660	5.368	6.571	7.790	9.467	18.151	21.064	23.685	26.873	29.141	36.123
15	5.229	5.985	7.261	8.547	10.307	19.311	22.307	24.996	28.259	30.578	37.697
16	5.812	6.614	7.962	9.312	11.152	20.465	23.542	26.296	29.633	32.000	39.252
17	6.408	7.255	8.672	10.085	12.002	21.615	24.769	27.587	30.995	33.409	40.790
18	7.015	7.906	9.390	10.865	12.857	22.760	25.989	28.869	32.346	34.805	42.312
19	7.633	8.567	10.117	11.651	13.716	23.900	27.204	30.144	33.687	36.191	43.820
20	8.260	9.237	10.851	12.443	14.578	25.038	28.412	31.410	35.020	37.566	45.315
21	8.897	9.915	11.591	13.240	15.445	26.171	29.615	32.671	36.343	38.932	46.797
22	9.542	10.600	12.338	14.041	16.314	27.301	30.813	33.924	37.659	40.289	48.268
23	10.196	11.293	13.091	14.848	17.187	28.429	32.007	35.172	38.968	41.638	49.728
24	10.856	11.992	13.848	15.659	18.062	29.553	33.196	36.415	40.270	42.980	51.179
25	11.524	12.697	14.611	16.473	18.940	30.675	34.382	37.652	41.566	44.314	52.620
26	12.198	13.409	15.379	17.292	19.820	31.795	35.563	38.885	42.856	45.642	54.052
27	12.879	14.125	16.151	18.114	20.703	32.912	36.741	40.113	44.140	46.963	55.476
28	13.565	14.847	16.928	18.939	21.588	34.027	37.916	41.337	45.419	48.278	56.893
29	14.256	15.574	17.708	19.768	22.475	35.139	39.087	42.557	46.693	49.588	58.302
30	14.953	16.306	18.493	20.599	23.364	36.250	40.256	43.773	47.962	50.892	59.703

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Table XI-5

Significant Ratios for σ/σ' = G Normal Population

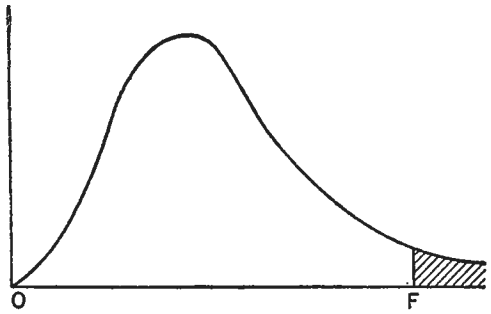
No. of Cases, N, in Sample	Probability of As Great or a Greater Value of σ/σ'				
	.99	.95	.05	.01	.001
2	.009	.044	1.386	1.821	2.327
3	.082	.185+	1.413	1.752	2.146
4	.170	.297	1.398	1.684	2.017
5	.244	.377	1.378	1.630	1.922
6	.304	.437	1.358	1.586	1.849
7	.353	.483	1.341	1.550	1.791
8	.394	.520	1.326	1.520	1.744
9	.428	.551	1.313	1.494	1.704
10	.457	.577	1.301	1.472	1.670
11	.482	.598	1.290	1.453	1.640
12	.504	.617	1.280	1.435+	1.614
13	.524	.634	1.272	1.420	1.591
14	.542	.649	1.264	1.406	1.570
15	.557	.662	1.257	1.394	1.552
16	.572	.674	1.250	1.382	1.535-
17	.585-	.684	1.244	1.372	1.520
18	.597	.694	1.238	1.362	1.505+
19	.608	.703	1.233	1.353	1.492
20	.618	.711	1.228	1.345+	1.480
21	.627	.719	1.223	1.337	1.469
22	.636	.726	1.219	1.330	1.458
23	.644	.732	1.214	1.324	1.449
24	.652	.739	1.211	1.317	1.439
25	.659	.744	1.207	1.311	1.431
26	.666	.750	1.203	1.306	1.423
27	.672	.755-	1.200	1.300	1.414
28	.678	.759	1.197	1.295+	1.408
29	.684	.764	1.194	1.290	1.401
30	.689	.768	1.191	1.286	1.394
31	.695-	.772	1.188	1.281	1.388
35	.713	.787	1.178	1.266	1.365+
40	.732	.801	1.168	1.249	1.342
45	.748	.814	1.159	1.236	1.323
50	.761	.824	1.152	1.224	1.307
55	.772	.832	1.145+	1.214	1.292
60	.782	.840	1.140	1.205+	1.280
65	.791	.847	1.135-	1.198	1.269
70	.799	.853	1.130	1.191	1.260
75	.806	.858	1.126	1.184	1.251
80	.812	.863	1.122	1.179	1.243
85	.818	.867	1.119	1.174	1.236
90	.823	.871	1.116	1.169	1.229
95	.828	.874	1.113	1.164	1.223
100	.832	.878	1.110	1.160	1.218
200	.882	.915-	1.079	1.114	1.154
300	.904	.931	1.065+	1.094	1.126
400	.917	.940	1.057	1.081	1.109
500	.926	.947	1.051	1.073	1.098
1000	.948	.963	1.036	1.052	1.069

+ and - .005's denote which direction to round if only 2 places are

Distribution of F*

5 Per Cent (Roman Type) and 1 Per Cent (Boldface Type)

Values of F corresponding to two selected probabilities (*i.e.*, tail areas under the curve). To illustrate: the probability is 0.05 that the ratio of two mean squares obtained with 20 and 10 degrees of freedom in numerator and denominator, respectively, would yield $F = 2.77$ or larger.



DF_2	DF_1 degrees of freedom for greater mean square (placed in the numerator)																							
	1	2	3	4	5	6	7	8	9	10	11	12	14	16	20	24	30	40	50	75	100	200	500	∞
1	161	200	216	225	230	234	237	239	241	242	243	244	245	246	248	249	250	251	252	253	253	254	254	254
	4,052	**4,999**	**5,403**	**5,625**	**5,764**	**5,859**	**5,928**	**5,981**	**6,022**	**6,056**	**6,082**	**6,106**	**6,142**	**6,169**	**6,208**	**6,234**	**6,258**	**6,286**	**6,302**	**6,323**	**6,334**	**6,352**	**6,361**	**6,366**
2	18.51	19.00	19.16	19.25	19.30	19.33	19.36	19.37	19.38	19.39	19.40	19.41	19.42	19.43	19.44	19.45	19.46	19.47	19.47	19.48	19.49	19.49	19.50	19.50
	98.49	**99.00**	**99.17**	**99.25**	**99.30**	**99.33**	**99.34**	**99.36**	**99.38**	**99.40**	**99.41**	**99.42**	**99.43**	**99.44**	**99.45**	**99.46**	**99.47**	**99.48**	**99.48**	**99.49**	**99.49**	**99.49**	**99.50**	**99.50**
3	10.13	9.55	9.28	9.12	9.01	8.94	8.88	8.84	8.81	8.78	8.76	8.74	8.71	8.69	8.66	8.64	8.62	8.60	8.58	8.57	8.56	8.54	8.54	8.53
	34.12	**30.82**	**29.46**	**28.71**	**28.24**	**27.91**	**27.67**	**27.49**	**27.34**	**27.23**	**27.13**	**27.05**	**26.92**	**26.83**	**26.69**	**26.60**	**26.50**	**26.41**	**26.35**	**26.27**	**26.23**	**26.18**	**26.14**	**26.12**
4	7.71	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00	5.96	5.93	5.91	5.87	5.84	5.80	5.77	5.74	5.71	5.70	5.68	5.66	5.65	5.64	5.63
	21.20	**18.00**	**16.69**	**15.98**	**15.52**	**15.21**	**14.98**	**14.80**	**14.66**	**14.54**	**14.45**	**14.37**	**14.24**	**14.15**	**14.02**	**13.93**	**13.83**	**13.74**	**13.69**	**13.61**	**13.57**	**13.52**	**13.48**	**13.46**
5	6.61	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.78	4.74	4.70	4.68	4.64	4.60	4.56	4.53	4.50	4.46	4.44	4.42	4.40	4.38	4.37	4.36
	16.26	**13.27**	**12.06**	**11.39**	**10.97**	**10.67**	**10.45**	**10.27**	**10.15**	**10.05**	**9.96**	**9.89**	**9.77**	**9.68**	**9.55**	**9.47**	**9.38**	**9.29**	**9.24**	**9.17**	**9.13**	**9.07**	**9.04**	**9.02**
6	5.99	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10	4.06	4.03	4.00	3.96	3.92	3.87	3.84	3.81	3.77	3.75	3.72	3.71	3.69	3.68	3.67
	13.74	**10.92**	**9.78**	**9.15**	**8.75**	**8.47**	**8.26**	**8.10**	**7.98**	**7.87**	**7.79**	**7.72**	**7.60**	**7.52**	**7.39**	**7.31**	**7.23**	**7.14**	**7.09**	**7.02**	**6.99**	**6.94**	**6.90**	**6.88**
7	5.59	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68	3.63	3.60	3.57	3.52	3.49	3.44	3.41	3.38	3.34	3.32	3.29	3.28	3.25	3.24	3.23
	12.25	**9.55**	**8.45**	**7.85**	**7.46**	**7.19**	**7.00**	**6.84**	**6.71**	**6.62**	**6.54**	**6.47**	**6.35**	**6.27**	**6.15**	**6.07**	**5.98**	**5.90**	**5.85**	**5.78**	**5.75**	**5.70**	**5.67**	**5.65**
8	5.32	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39	3.34	3.31	3.28	3.23	3.20	3.15	3.12	3.08	3.05	3.03	3.00	2.98	2.96	2.94	2.93
	11.26	**8.65**	**7.59**	**7.01**	**6.63**	**6.37**	**6.19**	**6.03**	**5.91**	**5.82**	**5.74**	**5.67**	**5.56**	**5.48**	**5.36**	**5.28**	**5.20**	**5.11**	**5.06**	**5.00**	**4.96**	**4.91**	**4.88**	**4.86**
9	5.12	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18	3.13	3.10	3.07	3.02	2.98	2.93	2.90	2.86	2.82	2.80	2.77	2.76	2.73	2.72	2.71
	10.56	**8.02**	**6.99**	**6.42**	**6.06**	**5.80**	**5.62**	**5.47**	**5.35**	**5.26**	**5.18**	**5.11**	**5.00**	**4.92**	**4.80**	**4.73**	**4.64**	**4.56**	**4.51**	**4.45**	**4.41**	**4.36**	**4.33**	**4.31**
10	4.96	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.97	2.94	2.91	2.86	2.82	2.77	2.74	2.70	2.67	2.64	2.61	2.59	2.56	2.55	2.54
	10.04	**7.56**	**6.55**	**5.99**	**5.64**	**5.39**	**5.21**	**5.06**	**4.95**	**4.85**	**4.78**	**4.71**	**4.60**	**4.52**	**4.41**	**4.33**	**4.25**	**4.17**	**4.12**	**4.05**	**4.01**	**3.96**	**3.93**	**3.91**

* This table is reproduced from Table 10.7 in "Statistical Methods," 4th ed., 1946, with the permission of Prof. George W. Snedecor and the publishers, The Iowa State University Press, Ames, Iowa. Fifth edition available, 1956.

DF_2	DF_1 degrees of freedom for greater mean square (placed in the numerator)																							
	1	2	3	4	5	6	7	8	9	10	11	12	14	16	20	24	30	40	50	75	100	200	500	∞
11	4.84	3.98	3.59	3.36	3.20	3.09	3.01	2.95	2.90	2.86	2.82	2.79	2.74	2.70	2.65	2.61	2.57	2.53	2.50	2.47	2.45	2.42	2.41	2.40
	9.65	**7.20**	**6.22**	**5.67**	**5.32**	**5.07**	**4.88**	**4.74**	**4.63**	**4.54**	**4.46**	**4.40**	**4.29**	**4.21**	**4.10**	**4.02**	**3.94**	**3.86**	**3.80**	**3.74**	**3.70**	**3.66**	**3.62**	**3.60**
12	4.75	3.88	3.49	3.26	3.11	3.00	2.92	2.85	2.80	2.76	2.72	2.69	2.64	2.60	2.54	2.50	2.46	2.42	2.40	2.36	2.35	2.32	2.31	2.30
	9.33	**6.93**	**5.95**	**5.41**	**5.06**	**4.82**	**4.65**	**4.50**	**4.39**	**4.30**	**4.22**	**4.16**	**4.05**	**3.98**	**3.86**	**3.78**	**3.70**	**3.61**	**3.56**	**3.49**	**3.46**	**3.41**	**3.38**	**3.36**
13	4.67	3.80	3.41	3.18	3.02	2.92	2.84	2.77	2.72	2.67	2.63	2.60	2.55	2.51	2.46	2.42	2.38	2.34	2.32	2.28	2.26	2.24	2.22	2.21
	9.07	**6.70**	**5.74**	**5.20**	**4.86**	**4.62**	**4.44**	**4.30**	**4.19**	**4.10**	**4.02**	**3.96**	**3.85**	**3.78**	**3.67**	**3.59**	**3.51**	**3.42**	**3.37**	**3.30**	**3.27**	**3.21**	**3.18**	**3.16**
14	4.60	3.74	3.34	3.11	2.96	2.85	2.77	2.70	2.65	2.60	2.56	2.53	2.48	2.44	2.39	2.35	2.31	2.27	2.24	2.21	2.19	2.16	2.14	2.13
	8.86	**6.51**	**5.56**	**5.03**	**4.69**	**4.46**	**4.28**	**4.14**	**4.03**	**3.94**	**3.86**	**3.80**	**3.70**	**3.62**	**3.51**	**3.43**	**3.34**	**3.26**	**3.21**	**3.14**	**3.11**	**3.06**	**3.02**	**3.00**
15	4.54	3.68	3.29	3.06	2.90	2.79	2.70	2.64	2.59	2.55	2.51	2.48	2.43	2.39	2.33	2.29	2.25	2.21	2.18	2.15	2.12	2.10	2.08	2.07
	8.68	**6.36**	**5.42**	**4.89**	**4.56**	**4.32**	**4.14**	**4.00**	**3.89**	**3.80**	**3.73**	**3.67**	**3.56**	**3.48**	**3.36**	**3.29**	**3.20**	**3.12**	**3.07**	**3.00**	**2.97**	**2.92**	**2.89**	**2.87**
16	4.49	3.63	3.24	3.01	2.85	2.74	2.66	2.59	2.54	2.49	2.45	2.42	2.37	2.33	2.28	2.24	2.20	2.16	2.13	2.09	2.07	2.04	2.02	2.01
	8.53	**6.23**	**5.29**	**4.77**	**4.44**	**4.20**	**4.03**	**3.89**	**3.78**	**3.69**	**3.61**	**3.55**	**3.45**	**3.37**	**3.25**	**3.18**	**3.10**	**3.01**	**2.96**	**2.89**	**2.86**	**2.80**	**2.77**	**2.75**
17	4.45	3.59	3.20	2.96	2.81	2.70	2.62	2.55	2.50	2.45	2.41	2.38	2.33	2.29	2.23	2.19	2.15	2.11	2.08	2.04	2.02	1.99	1.97	1.96
	8.40	**6.11**	**5.18**	**4.67**	**4.34**	**4.10**	**3.93**	**3.79**	**3.68**	**3.59**	**3.52**	**3.45**	**3.35**	**3.27**	**3.16**	**3.08**	**3.00**	**2.92**	**2.86**	**2.79**	**2.76**	**2.70**	**2.67**	**2.65**
18	4.41	3.55	3.16	2.93	2.77	2.66	2.58	2.51	2.46	2.41	2.37	2.34	2.29	2.25	2.19	2.15	2.11	2.07	2.04	2.00	1.98	1.95	1.93	1.92
	8.28	**6.01**	**5.09**	**4.58**	**4.25**	**4.01**	**3.85**	**3.71**	**3.60**	**3.51**	**3.44**	**3.37**	**3.27**	**3.19**	**3.07**	**3.00**	**2.91**	**2.83**	**2.78**	**2.71**	**2.68**	**2.62**	**2.59**	**2.57**
19	4.38	3.52	3.13	2.90	2.74	2.63	2.55	2.48	2.43	2.38	2.34	2.31	2.26	2.21	2.15	2.11	2.07	2.02	2.00	1.96	1.94	1.91	1.90	1.88
	8.18	**5.93**	**5.01**	**4.50**	**4.17**	**3.94**	**3.77**	**3.63**	**3.52**	**3.43**	**3.36**	**3.30**	**3.19**	**3.12**	**3.00**	**2.92**	**2.84**	**2.76**	**2.70**	**2.63**	**2.60**	**2.54**	**2.51**	**2.49**
20	4.35	3.49	3.10	2.87	2.71	2.60	2.52	2.45	2.40	2.35	2.31	2.28	2.23	2.18	2.12	2.08	2.04	1.99	1.96	1.92	1.90	1.87	1.85	1.84
	8.10	**5.85**	**4.94**	**4.43**	**4.10**	**3.87**	**3.71**	**3.56**	**3.45**	**3.37**	**3.30**	**3.23**	**3.13**	**3.05**	**2.94**	**2.86**	**2.77**	**2.69**	**2.63**	**2.56**	**2.53**	**2.47**	**2.44**	**2.42**
21	4.32	3.47	3.07	2.84	2.68	2.57	2.49	2.42	2.37	2.32	2.28	2.25	2.20	2.15	2.09	2.05	2.00	1.96	1.93	1.89	1.87	1.84	1.82	1.81
	8.02	**5.78**	**4.87**	**4.37**	**4.04**	**3.81**	**3.65**	**3.51**	**3.40**	**3.31**	**3.24**	**3.17**	**3.07**	**2.99**	**2.88**	**2.80**	**2.72**	**2.63**	**2.58**	**2.51**	**2.47**	**2.42**	**2.38**	**2.36**
22	4.30	3.44	3.05	2.82	2.66	2.55	2.47	2.40	2.35	2.30	2.26	2.23	2.18	2.13	2.07	2.03	1.98	1.93	1.91	1.87	1.84	1.81	1.80	1.78
	7.94	**5.72**	**4.82**	**4.31**	**3.99**	**3.76**	**3.59**	**3.45**	**3.35**	**3.26**	**3.18**	**3.12**	**3.02**	**2.94**	**2.83**	**2.75**	**2.67**	**2.58**	**2.53**	**2.46**	**2.42**	**2.37**	**2.33**	**2.31**
23	4.28	3.42	3.03	2.80	2.64	2.53	2.45	2.38	2.32	2.28	2.24	2.20	2.14	2.10	2.04	2.00	1.96	1.91	1.88	1.84	1.82	1.79	1.77	1.76
	7.88	**5.66**	**4.76**	**4.26**	**3.94**	**3.71**	**3.54**	**3.41**	**3.30**	**3.21**	**3.14**	**3.07**	**2.97**	**2.89**	**2.78**	**2.70**	**2.62**	**2.53**	**2.48**	**2.41**	**2.37**	**2.32**	**2.28**	**2.26**
24	4.26	3.40	3.01	2.78	2.62	2.51	2.43	2.36	2.30	2.26	2.22	2.18	2.13	2.09	2.02	1.98	1.94	1.89	1.86	1.82	1.80	1.76	1.74	1.73
	7.82	**5.61**	**4.72**	**4.22**	**3.90**	**3.67**	**3.50**	**3.36**	**3.25**	**3.17**	**3.09**	**3.03**	**2.93**	**2.85**	**2.74**	**2.66**	**2.58**	**2.49**	**2.44**	**2.36**	**2.33**	**2.27**	**2.23**	**2.21**
25	4.24	3.38	2.99	2.76	2.60	2.49	2.41	2.34	2.28	2.24	2.20	2.16	2.11	2.06	2.00	1.96	1.92	1.87	1.84	1.80	1.77	1.74	1.72	1.71
	7.77	**5.57**	**4.68**	**4.18**	**3.86**	**3.63**	**3.46**	**3.32**	**3.21**	**3.13**	**3.05**	**2.99**	**2.89**	**2.81**	**2.70**	**2.62**	**2.54**	**2.45**	**2.40**	**2.32**	**2.29**	**2.23**	**2.19**	**2.17**

DISTRIBUTION OF F (*Continued*)

5 Per Cent (Roman Type) and 1 Per Cent (Boldface Type) Points for the Distribution of F

DF_2	DF_1 degrees of freedom for greater mean square (placed in the numerator)																							
	1	2	3	4	5	6	7	8	9	10	11	12	14	16	20	24	30	40	50	75	100	200	500	∞
26	4.22	3.37	2.98	2.74	2.59	2.47	2.39	2.32	2.27	2.22	2.18	2.15	2.10	2.05	1.99	1.95	1.90	1.85	1.82	1.78	1.76	1.72	1.70	1.69
	7.72	**5.53**	**4.64**	**4.14**	**3.82**	**3.59**	**3.42**	**3.29**	**3.17**	**3.09**	**3.02**	**2.96**	**2.86**	**2.77**	**2.66**	**2.58**	**2.50**	**2.41**	**2.36**	**2.28**	**2.25**	**2.19**	**2.15**	**2.13**
27	4.21	3.35	2.96	2.73	2.57	2.46	2.37	2.30	2.25	2.20	2.16	2.13	2.08	2.03	1.97	1.93	1.88	1.84	1.80	1.76	1.74	1.71	1.68	1.67
	7.68	**5.49**	**4.60**	**4.11**	**3.79**	**3.56**	**3.39**	**3.26**	**3.14**	**3.06**	**2.98**	**2.93**	**2.83**	**2.74**	**2.63**	**2.55**	**2.47**	**2.38**	**2.33**	**2.25**	**2.21**	**2.16**	**2.12**	**2.10**
28	4.20	3.34	2.95	2.71	2.56	2.44	2.36	2.29	2.24	2.19	2.15	2.12	2.06	2.02	1.96	1.91	1.87	1.81	1.78	1.75	1.72	1.69	1.67	1.65
	7.64	**5.45**	**4.57**	**4.07**	**3.76**	**3.53**	**3.36**	**3.23**	**3.11**	**3.03**	**2.95**	**2.90**	**2.80**	**2.71**	**2.60**	**2.52**	**2.44**	**2.35**	**2.30**	**2.22**	**2.18**	**2.13**	**2.09**	**2.06**
29	4.18	3.33	2.93	2.70	2.54	2.43	2.35	2.28	2.22	2.18	2.14	2.10	2.05	2.00	1.94	1.90	1.85	1.80	1.77	1.73	1.71	1.68	1.65	1.64
	7.60	**5.42**	**4.54**	**4.04**	**3.73**	**3.50**	**3.33**	**3.20**	**3.08**	**3.00**	**2.92**	**2.87**	**2.77**	**2.68**	**2.57**	**2.49**	**2.41**	**2.32**	**2.27**	**2.19**	**2.15**	**2.10**	**2.06**	**2.03**
30	4.17	3.32	2.92	2.69	2.53	2.42	2.34	2.27	2.21	2.16	2.12	2.09	2.04	1.99	1.93	1.89	1.84	1.79	1.76	1.72	1.69	1.66	1.64	1.62
	7.56	**5.39**	**4.51**	**4.02**	**3.70**	**3.47**	**3.30**	**3.17**	**3.06**	**2.98**	**2.90**	**2.84**	**2.74**	**2.66**	**2.55**	**2.47**	**2.38**	**2.29**	**2.24**	**2.16**	**2.13**	**2.07**	**2.03**	**2.01**
32	4.15	3.30	2.90	2.67	2.51	2.40	2.32	2.25	2.19	2.14	2.10	2.07	2.02	1.97	1.91	1.86	1.82	1.76	1.74	1.69	1.67	1.64	1.61	1.59
	7.50	**5.34**	**4.46**	**3.97**	**3.66**	**3.42**	**3.25**	**3.12**	**3.01**	**2.94**	**2.86**	**2.80**	**2.70**	**2.62**	**2.51**	**2.42**	**2.34**	**2.25**	**2.20**	**2.12**	**2.08**	**2.02**	**1.98**	**1.96**
34	4.13	3.28	2.88	2.65	2.49	2.38	2.30	2.23	2.17	2.12	2.08	2.05	2.00	1.95	1.89	1.84	1.80	1.74	1.71	1.67	1.64	1.61	1.59	1.57
	7.44	**5.29**	**4.42**	**3.93**	**3.61**	**3.38**	**3.21**	**3.08**	**2.97**	**2.89**	**2.82**	**2.76**	**2.66**	**2.58**	**2.47**	**2.38**	**2.30**	**2.21**	**2.15**	**2.08**	**2.04**	**1.98**	**1.94**	**1.91**
36	4.11	3.26	2.86	2.63	2.48	2.36	2.28	2.21	2.15	2.10	2.06	2.03	1.98	1.93	1.87	1.82	1.78	1.72	1.69	1.65	1.62	1.59	1.56	1.55
	7.39	**5.25**	**4.38**	**3.89**	**3.58**	**3.35**	**3.18**	**3.04**	**2.94**	**2.86**	**2.78**	**2.72**	**2.62**	**2.54**	**2.43**	**2.35**	**2.26**	**2.17**	**2.12**	**2.04**	**2.00**	**1.94**	**1.90**	**1.87**
38	4.10	3.25	2.85	2.62	2.46	2.35	2.26	2.19	2.14	2.09	2.05	2.02	1.96	1.92	1.85	1.80	1.76	1.71	1.67	1.63	1.60	1.57	1.54	1.53
	7.35	**5.21**	**4.34**	**3.86**	**3.54**	**3.32**	**3.15**	**3.02**	**2.91**	**2.82**	**2.75**	**2.69**	**2.59**	**2.51**	**2.40**	**2.32**	**2.22**	**2.14**	**2.08**	**2.00**	**1.97**	**1.90**	**1.86**	**1.84**
40	4.08	3.23	2.84	2.61	2.45	2.34	2.25	2.18	2.12	2.07	2.04	2.00	1.95	1.90	1.84	1.79	1.74	1.69	1.66	1.61	1.59	1.55	1.53	1.51
	7.31	**5.18**	**4.31**	**3.83**	**3.51**	**3.29**	**3.12**	**2.99**	**2.88**	**2.80**	**2.73**	**2.66**	**2.56**	**2.49**	**2.37**	**2.29**	**2.20**	**2.11**	**2.05**	**1.97**	**1.94**	**1.88**	**1.84**	**1.81**
42	4.07	3.22	2.83	2.59	2.44	2.32	2.24	2.17	2.11	2.06	2.02	1.99	1.94	1.89	1.82	1.78	1.73	1.68	1.64	1.60	1.57	1.54	1.51	1.49
	7.27	**5.15**	**4.29**	**3.80**	**3.49**	**3.26**	**3.10**	**2.96**	**2.86**	**2.77**	**2.70**	**2.64**	**2.54**	**2.46**	**2.35**	**2.26**	**2.17**	**2.08**	**2.02**	**1.94**	**1.91**	**1.85**	**1.80**	**1.78**
44	4.06	3.21	2.82	2.58	2.43	2.31	2.23	2.16	2.10	2.05	2.01	1.98	1.92	1.88	1.81	1.76	1.72	1.66	1.63	1.58	1.56	1.52	1.50	1.48
	7.24	**5.12**	**4.26**	**3.78**	**3.46**	**3.24**	**3.07**	**2.94**	**2.84**	**2.75**	**2.68**	**2.62**	**2.52**	**2.44**	**2.32**	**2.24**	**2.15**	**2.06**	**2.00**	**1.92**	**1.88**	**1.82**	**1.78**	**1.75**
46	4.05	3.20	2.81	2.57	2.42	2.30	2.22	2.14	2.09	2.04	2.00	1.97	1.91	1.87	1.80	1.75	1.71	1.65	1.62	1.57	1.54	1.51	1.48	1.46
	7.21	**5.10**	**4.24**	**3.76**	**3.44**	**3.22**	**3.05**	**2.92**	**2.82**	**2.73**	**2.66**	**2.60**	**2.50**	**2.42**	**2.30**	**2.22**	**2.13**	**2.04**	**1.98**	**1.90**	**1.86**	**1.80**	**1.76**	**1.72**
48	4.04	3.19	2.80	2.56	2.41	2.30	2.21	2.14	2.08	2.03	1.99	1.96	1.90	1.86	1.79	1.74	1.70	1.64	1.61	1.56	1.53	1.50	1.47	1.45
	7.19	**5.08**	**4.22**	**3.74**	**3.42**	**3.20**	**3.04**	**2.90**	**2.80**	**2.71**	**2.64**	**2.58**	**2.48**	**2.40**	**2.28**	**2.20**	**2.11**	**2.02**	**1.96**	**1.88**	**1.84**	**1.78**	**1.73**	**1.70**

DF_2	DF_1 degrees of freedom for greater mean square (placed in the numerator)																							
	1	2	3	4	5	6	7	8	9	10	11	12	14	16	20	24	30	40	50	75	100	200	500	∞
50	4.03	3.18	2.79	2.56	2.40	2.29	2.20	2.13	2.07	2.02	1.98	1.95	1.90	1.85	1.78	1.74	1.69	1.63	1.60	1.55	1.52	1.48	1.46	1.44
	7.17	**5.06**	**4.20**	**3.72**	**3.41**	**3.18**	**3.02**	**2.88**	**2.78**	**2.70**	**2.62**	**2.56**	**2.46**	**2.39**	**2.26**	**2.18**	**2.10**	**2.00**	**1.94**	**1.86**	**1.82**	**1.76**	**1.71**	**1.68**
55	4.02	3.17	2.78	2.54	2.38	2.27	2.18	2.11	2.05	2.00	1.97	1.93	1.88	1.83	1.76	1.72	1.67	1.61	1.58	1.52	1.50	1.46	1.43	1.41
	7.12	**5.01**	**4.16**	**3.68**	**3.37**	**3.15**	**2.98**	**2.85**	**2.75**	**2.66**	**2.59**	**2.53**	**2.43**	**2.35**	**2.23**	**2.15**	**2.06**	**1.96**	**1.90**	**1.82**	**1.78**	**1.71**	**1.66**	**1.64**
60	4.00	3.15	2.76	2.52	2.37	2.25	2.17	2.10	2.04	1.99	1.95	1.92	1.86	1.81	1.75	1.70	1.65	1.59	1.56	1.50	1.48	1.44	1.41	1.39
	7.08	**4.98**	**4.13**	**3.65**	**3.34**	**3.12**	**2.95**	**2.82**	**2.72**	**2.63**	**2.56**	**2.50**	**2.40**	**2.32**	**2.20**	**2.12**	**2.03**	**1.93**	**1.87**	**1.79**	**1.74**	**1.68**	**1.63**	**1.60**
65	3.99	3.14	2.75	2.51	2.36	2.24	2.15	2.08	2.02	1.98	1.94	1.90	1.85	1.80	1.73	1.68	1.63	1.57	1.54	1.49	1.46	1.42	1.39	1.37
	7.04	**4.95**	**4.10**	**3.62**	**3.31**	**3.09**	**2.93**	**2.79**	**2.70**	**2.61**	**2.54**	**2.47**	**2.37**	**2.30**	**2.18**	**2.09**	**2.00**	**1.90**	**1.84**	**1.76**	**1.71**	**1.64**	**1.60**	**1.56**
70	3.98	3.13	2.74	2.50	2.35	2.23	2.14	2.07	2.01	1.97	1.93	1.89	1.84	1.79	1.72	1.67	1.62	1.56	1.53	1.47	1.45	1.40	1.37	1.35
	7.01	**4.92**	**4.08**	**3.60**	**3.29**	**3.07**	**2.91**	**2.77**	**2.67**	**2.59**	**2.51**	**2.45**	**2.35**	**2.28**	**2.15**	**2.07**	**1.98**	**1.88**	**1.82**	**1.74**	**1.69**	**1.62**	**1.56**	**1.53**
80	3.96	3.11	2.72	2.48	2.33	2.21	2.12	2.05	1.99	1.95	1.91	1.88	1.82	1.77	1.70	1.65	1.60	1.54	1.51	1.45	1.42	1.38	1.35	1.32
	6.96	**4.88**	**4.04**	**3.56**	**3.25**	**3.04**	**2.87**	**2.74**	**2.64**	**2.55**	**2.48**	**2.41**	**2.32**	**2.24**	**2.11**	**2.03**	**1.94**	**1.84**	**1.78**	**1.70**	**1.65**	**1.57**	**1.52**	**1.49**
100	3.94	3.09	2.70	2.46	2.30	2.19	2.10	2.03	1.97	1.92	1.88	1.85	1.79	1.75	1.68	1.63	1.57	1.51	1.48	1.42	1.39	1.34	1.30	1.28
	6.90	**4.82**	**3.98**	**3.51**	**3.20**	**2.99**	**2.82**	**2.69**	**2.59**	**2.51**	**2.43**	**2.36**	**2.26**	**2.19**	**2.06**	**1.98**	**1.89**	**1.79**	**1.73**	**1.64**	**1.59**	**1.51**	**1.46**	**1.43**
125	3.92	3.07	2.68	2.44	2.29	2.17	2.08	2.01	1.95	1.90	1.86	1.83	1.77	1.72	1.65	1.60	1.55	1.49	1.45	1.39	1.36	1.31	1.27	1.25
	6.84	**4.78**	**3.94**	**3.47**	**3.17**	**2.95**	**2.79**	**2.65**	**2.56**	**2.47**	**2.40**	**2.33**	**2.23**	**2.15**	**2.03**	**1.94**	**1.85**	**1.75**	**1.68**	**1.59**	**1.54**	**1.46**	**1.40**	**1.37**
150	3.91	3.06	2.67	2.43	2.27	2.16	2.07	2.00	1.94	1.89	1.85	1.82	1.76	1.71	1.64	1.59	1.54	1.47	1.44	1.37	1.34	1.29	1.25	1.22
	6.81	**4.75**	**3.91**	**3.44**	**3.14**	**2.92**	**2.76**	**2.62**	**2.53**	**2.44**	**2.37**	**2.30**	**2.20**	**2.12**	**2.00**	**1.91**	**1.83**	**1.72**	**1.66**	**1.56**	**1.51**	**1.43**	**1.37**	**1.33**
200	3.89	3.04	2.65	2.41	2.26	2.14	2.05	1.98	1.92	1.87	1.83	1.80	1.74	1.69	1.62	1.57	1.52	1.45	1.42	1.35	1.32	1.26	1.22	1.19
	6.76	**4.71**	**3.88**	**3.41**	**3.11**	**2.90**	**2.73**	**2.60**	**2.50**	**2.41**	**2.34**	**2.28**	**2.17**	**2.09**	**1.97**	**1.88**	**1.79**	**1.69**	**1.62**	**1.53**	**1.48**	**1.39**	**1.33**	**1.28**
400	3.86	3.02	2.62	2.39	2.23	2.12	2.03	1.96	1.90	1.85	1.81	1.78	1.72	1.67	1.60	1.54	1.49	1.42	1.38	1.32	1.28	1.22	1.16	1.13
	6.70	**4.66**	**3.83**	**3.36**	**3.06**	**2.85**	**2.69**	**2.55**	**2.46**	**2.37**	**2.29**	**2.23**	**2.12**	**2.04**	**1.92**	**1.84**	**1.74**	**1.64**	**1.57**	**1.47**	**1.42**	**1.32**	**1.24**	**1.19**
1000	3.85	3.00	2.61	2.38	2.22	2.10	2.02	1.95	1.89	1.84	1.80	1.76	1.70	1.65	1.58	1.53	1.47	1.41	1.36	1.30	1.26	1.19	1.13	1.08
	6.66	**4.62**	**3.80**	**3.34**	**3.04**	**2.82**	**2.66**	**2.53**	**2.43**	**2.34**	**2.26**	**2.20**	**2.09**	**2.01**	**1.89**	**1.81**	**1.71**	**1.61**	**1.54**	**1.44**	**1.38**	**1.28**	**1.19**	**1.11**
∞	3.84	2.99	2.60	2.37	2.21	2.09	2.01	1.94	1.88	1.83	1.79	1.75	1.69	1.64	1.57	1.52	1.46	1.40	1.35	1.28	1.24	1.17	1.11	1.00
	6.64	**4.60**	**3.78**	**3.32**	**3.02**	**2.80**	**2.64**	**2.51**	**2.41**	**2.32**	**2.24**	**2.18**	**2.07**	**1.99**	**1.87**	**1.79**	**1.69**	**1.59**	**1.52**	**1.41**	**1.36**	**1.25**	**1.15**	**1.00**

Table XI-6

FACTORS FOR COMPUTING CONTROL CHART LINES (1)

Observations in Subgroup (n)	Factor A_2	Factor d_2	Factor D_4	Factor D_3
2	1.880	1.13	3.267	0
3	1.023	1.60	2.575	0
4	0.729	2.06	2.282	0
5	0.577	2.33	2.115	0
6	0.483	2.53	2.004	0
7	0.419	2.70	1.924	0.08
8	0.373	2.85	1.864	0.14

Table XI-7

MOVING AVERAGE AND RANGE TABLE (n = 2)

SAMPLE NO.	ASSAY VALUE	SAMPLE NOS. INCLUDED	MOVING AVERAGE	MOVING RANGE
1	17.09	-	-	-
2	17,35	1 - 2	17.24	-0.25
3	17.40	2 - 3	17.38	-0.05
4	17.23	3 - 4	17.32	-0.17
5	17.09	4 - 5	17.15	-0.14
6	16.14	5 - 6	17.02	-0.15
7	16.68	6 - 7	16.81	-0.26
8	17.11	7 - 8	16.90	-0.43
9	18.47	8 - 9	17.79	-1.36
10	17.08	9 - 10	17.78	-1.39
11	17.08	10 - 11	17.08	-0.00
12	16.23	11 - 12	17.00	-0.16
13	18.03	12 - 13	17.43	-1.11
14	16.81	13 - 14	17.42	-1.22
15	17.18	14 - 15	16.98	-0.34
16	17.34	15 - 16	17.25	-0.13
17	16.71	16 - 17	17.03	-0.73
18	17.28	17 - 18	17.00	-0.57
19	16.54	18 - 19	16.91	-0.74
20	17.30	19 - 20	16.92	-0.76

Table XI-8

PRECISION (DUPLICATES) DATA

DATE	DATA			R
9/69	# 8	25.1	24.9	0.2
	#16	25.0	24.5	0.5
	#24	10.9	10.6	0.3
10/69	# 7	12.6	12.4	0.2
	#16	26.9	26.2	0.7
	#24	4.7	5.1	0.4
2/70	# 6	9.2	8.9	0.3
	#12	13.2	13.1	0.1
	#16	16.2	16.3	0.1
	#22	8.8	8.8	0.0
4/70	# 6	14.9	14.9	0.0
	#12	17.2	18.1	0.9
	#18	21.9	22.2	0.3
5/70	# 6	34.8	32.6	2.2
	#12	37.8	37.4	0.4
6/70	# 6	40.8	39.8	1.0
	#10	46.0	43.5	2.5
	#17	40.8	41.2	0.4
	#24	38.1	36.1	2.0
7/70	# 6	12.2	12.5	0.3
	#12	25.4	26.9	1.5
	#18	20.4	19.8	0.6

$\overline{R} = 14.9/22 = 0.68$

UCL = 3.27 X 0.68 = 2.2

UWL = 2.51 X 0.68 = 1.7

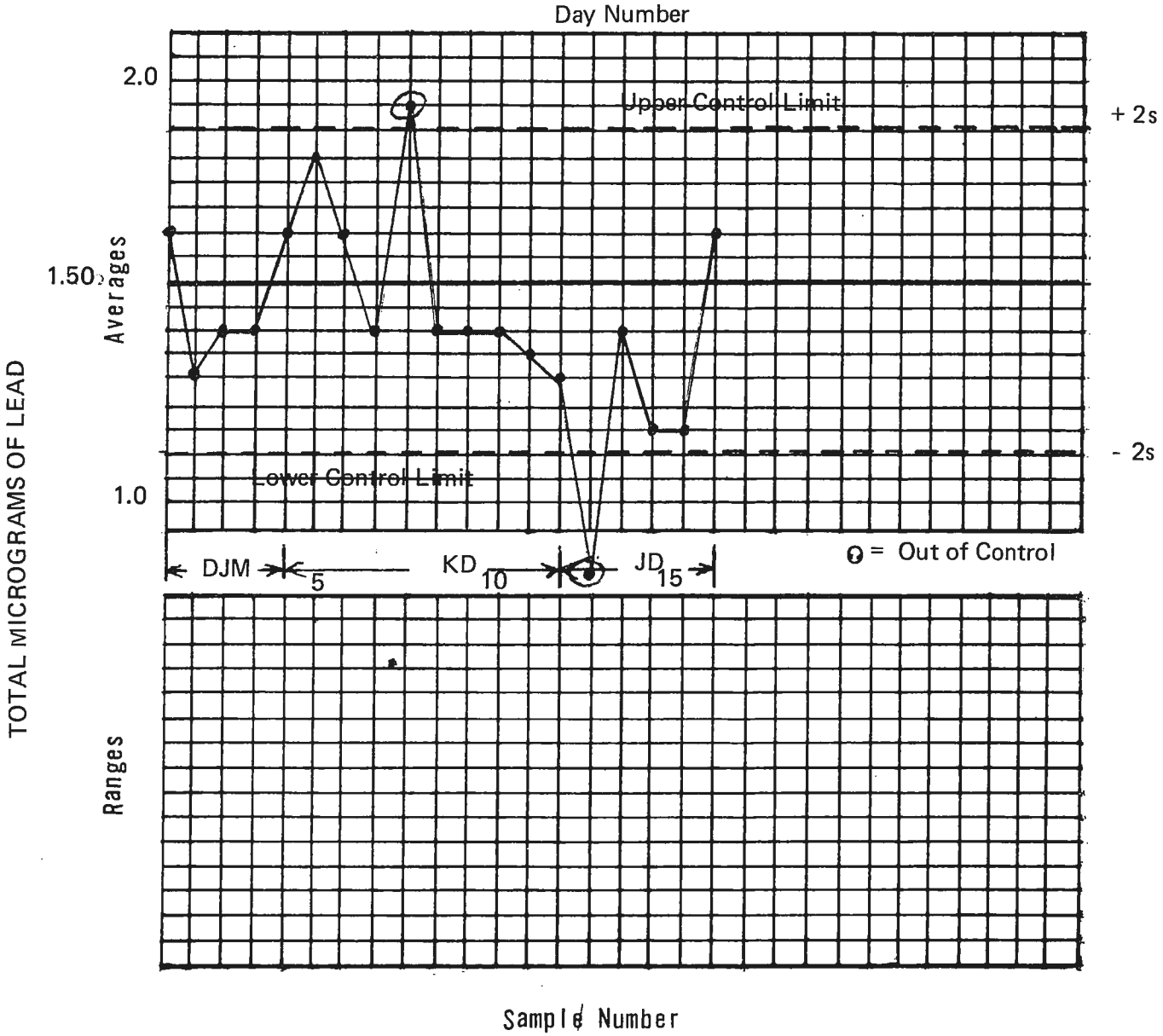
Table XI-9

ACCURACY DATA

Date	Calibrating Range	Nominal (N)	Values	$\overline{X}$	N-$\overline{X}$
9/69	10 - 400 ppm	100 ppm	22.9, 21.5/ 22.7, 22.3	22.2 22.5	-0.7 -0.4
	1.7 - 69.7 scale	22.9			
10/69	10 - 400	100	21.6, 21.3/	21.5	0.0
	1.5 - 67.6	21.5			
2/70	10 - 400	100	23.6, 24.1	23.9	-0.6
	1.4 - 62.5	24.5			
4/70	10 - 400	100	25.8, 26.5/	26.2	+0.2
	1.6 - 59.4	26.0	26.0, 26.7	26.4	+0.4
5/70	10 - 150	100	72.2, 70.2/	71.2	+1.2
	6.3 - 83.0	70.0			
6/70	10 - 150	100	71.0, 70.8/ 71.0, 71.3	71.1 71.2	+0.1 +0.2
	6.6 - 85.0	71.0			
7/70	10 - 150	60	14.9, 14.7/ 15.1, 14.4	14.8 14.8	-0.2 -0.2
	1.8 - 33.5	15.0			

FIGURE XI-1
NIOSH – Division of Training
Practice Worksheet
Laboratory Quality Control $\bar{X}$–R Chart

Operation Lead in Blood Control Chart Date _____



Directions:

	Value		Value
1. Draw $\bar{R}$ line	_____	6. Draw $UCL_{\bar{X}}$ line	_____
2. Draw UCL_R line	_____	7. Draw $UWL_{\bar{X}}$ line	_____
3. Draw UWL_R line	_____	8. Draw $LWL_{\bar{X}}$ line	_____
4. Plot R's as generated		9. Draw $LCL_{\bar{X}}$ line	_____
5. Draw $\bar{\bar{X}}$ line	_____	10. Plot $\bar{X}$'s as generated	

NIOSH – Division of Training

Practice Worksheet

Laboratory Quality Control $\bar{X}$–R Chart

Laboratory _____ Date _____

Method of Test or Operation _____

Reference Value _____ Increment of Measurement _____

Data

No.	X_1	X_2	X_3	$\bar{X}$	R
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Totals $\Sigma \bar{X}$ _____ ΣR _____

X_i = observed value R = largest – smallest

n = sets of values CL = control limit

Σ = summation WL = warning limit

U = upper L = lower

D_4 = 3.268 for n = 2; 2.574 for n = 3

A_2 = 1.880 for n = 2; 10023 for n = 3

Calculations

1. $\bar{R} = \Sigma R \div n$

   ___ = ___ ÷ ___

2. $UCL_R = D_4 \times \bar{R}$

   ___ = ___ × ___

3. $UWL_R = 2/3(D_4\bar{R} - \bar{R}) + \bar{R}$

   ___ = 2/3(___ – ___) + ___

4. $\bar{\bar{X}} = \Sigma \bar{X} \div n$

   ___ = ___ ÷ ___

5. $CL_{\bar{X}} = A_2 \times \bar{R}$

   ___ = ___ × ___

6. $WL_{\bar{X}} = 2/3 \times CL_{\bar{X}}$

   ___ = 2/3 × ___

7. $UCL_{\bar{X}} = \bar{\bar{X}} + CL_{\bar{X}}$

   ___ = ___ + ___

8. $UWL_{\bar{X}} = \bar{\bar{X}} + WL_{\bar{X}}$

   ___ = ___ + ___

9. $LWL_{\bar{X}} = \bar{\bar{X}} - WL_{\bar{X}}$

   ___ = ___ – ___

10. $LCL_{\bar{X}} = \bar{\bar{X}} - CL_{\bar{X}}$

    ___ = ___ – ___

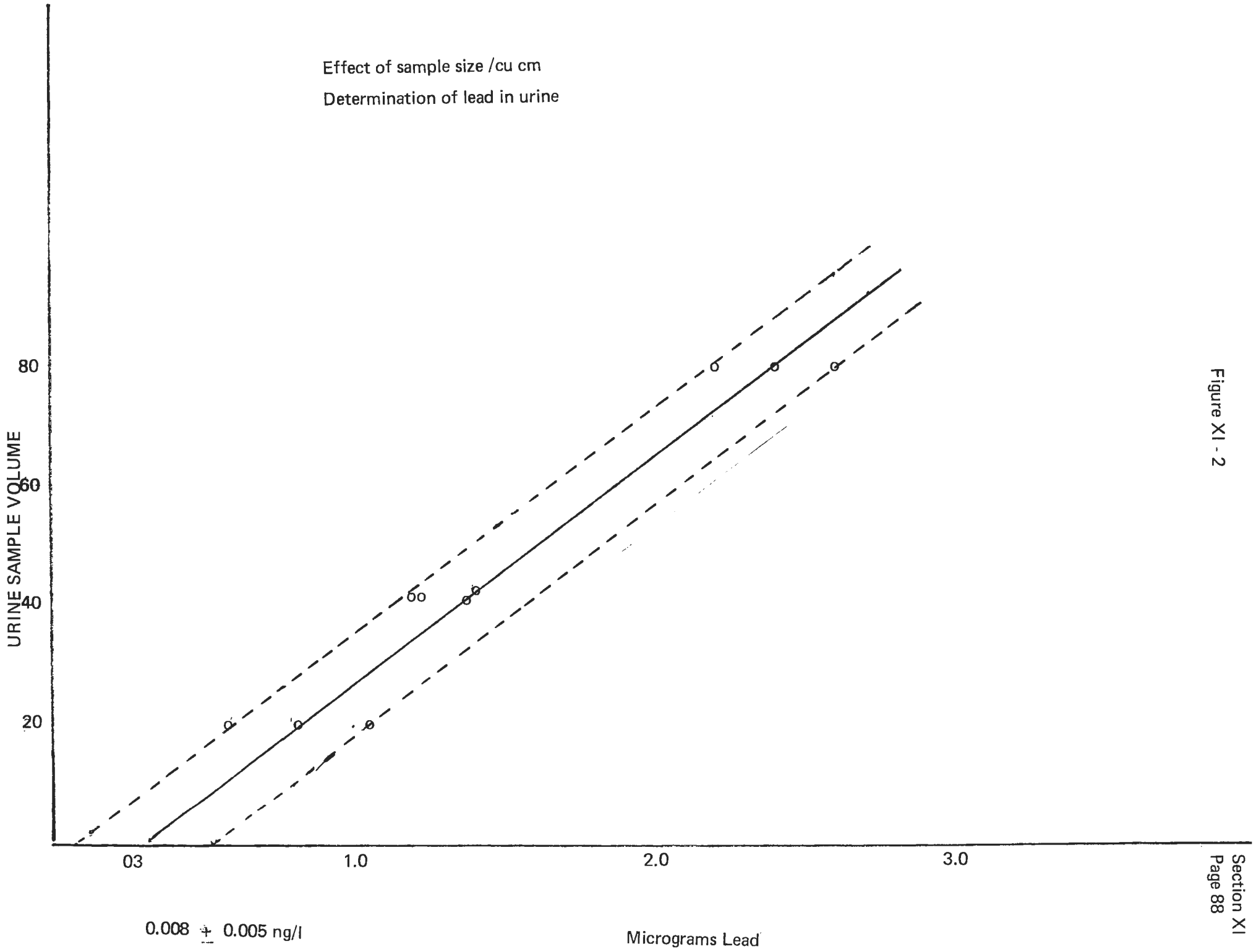

Effect of sample size /cu cm
Determination of lead in urine
URINE SAMPLE VOLUME
80
60
40
20
03
1.0
2.0
3.0
0.008 ± 0.005 ng/l
Micrograms Lead

Figure XI - 2

FIGURE XI-3

Graussian or Normal Curve of Frequencies

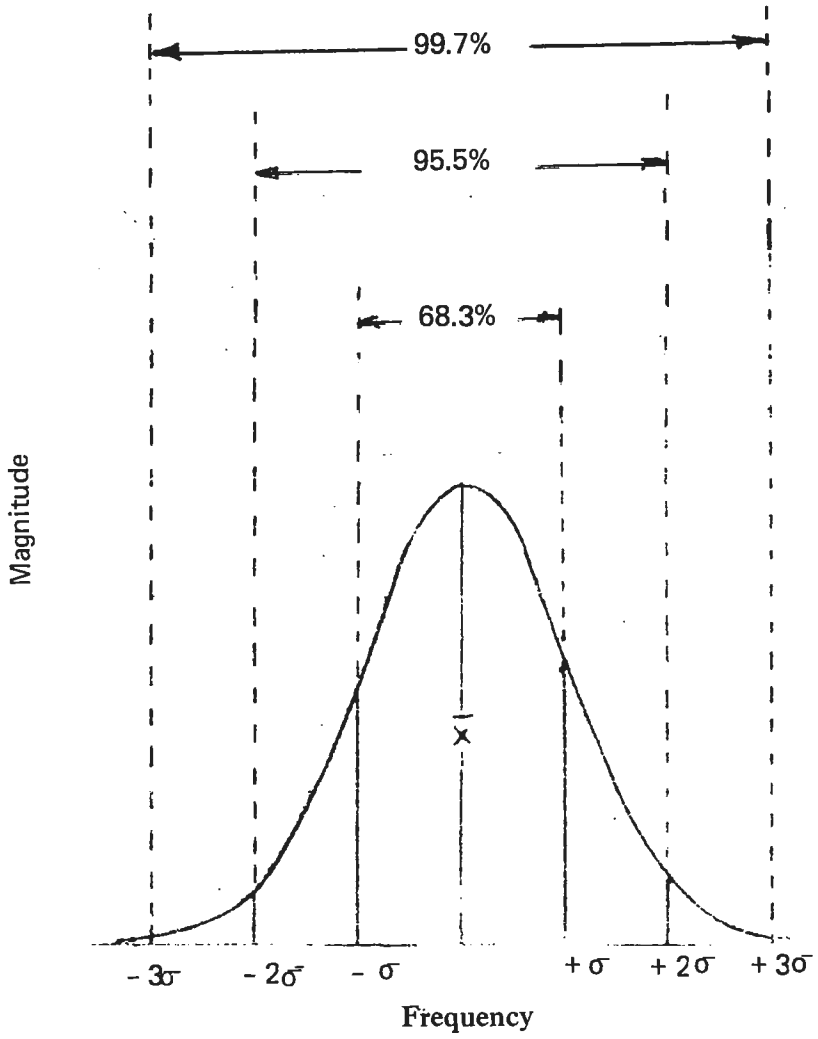


FIGURE XI-4

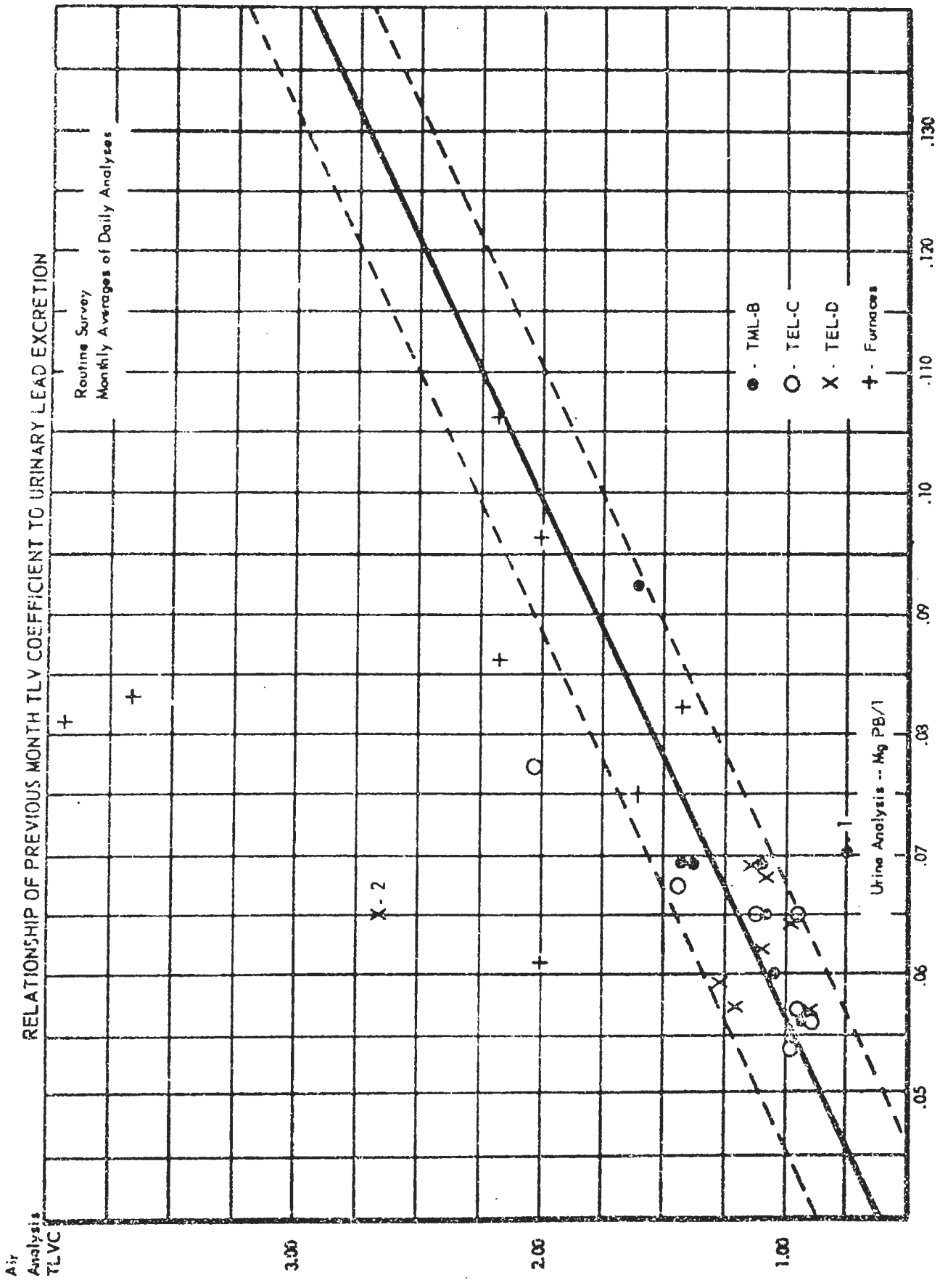
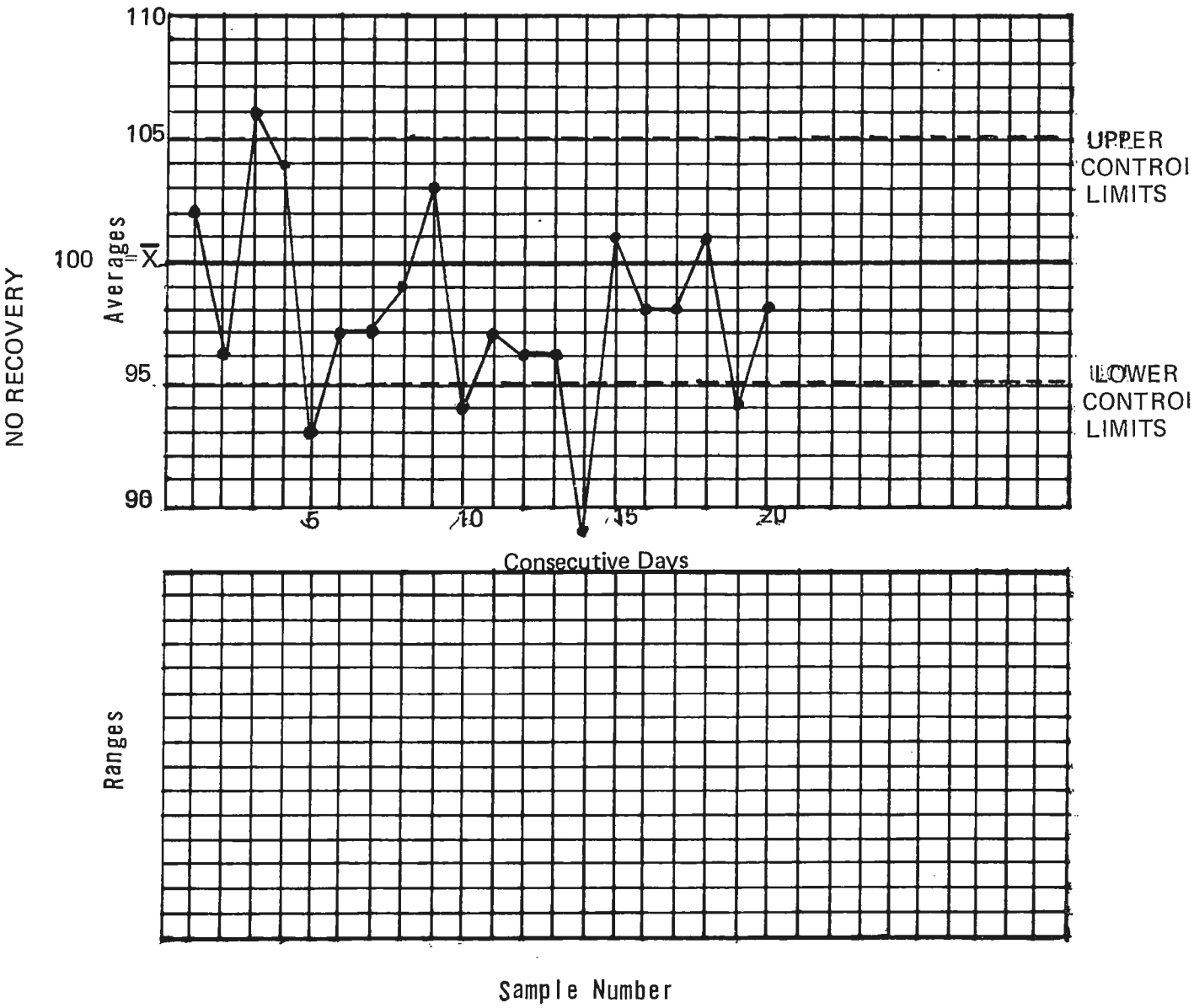

RELATIONSHIP OF PREVIOUS MONTH TLV COEFFICIENT TO URINARY LEAD EXCRETION
Routine Survey
Monthly Averages of Daily Analyses
Air Analysis TLVC
3.00
2.00
1.00
.05
.06
.07
.08
.09
.10
.110
.120
.130
Urine Analysis -- Mg PB/1
● - TML-B
O - TEL-C
X - TEL-D
+ - Furnaces
X-2

FIGURE XI-5
NIOSH – Division of Training
Practice Worksheet
Laboratory Quality Control $\bar{X}$–R Chart

Operation Recovery of Lead from Blood Date _____



Directions: Value

1. Draw $\bar{R}$ line _____
2. Draw UCL_R line _____
3. Draw UWL_R line _____
4. Plot R's as generated
5. Draw $\bar{\bar{X}}$ line _____
6. Draw $UCL_{\bar{X}}$ line _____
7. Draw $UWL_{\bar{X}}$ line _____
8. Draw $LWL_{\bar{X}}$ line _____
9. Draw $LCL_{\bar{X}}$ line _____
10. Plot $\bar{X}$'s as generated

NIOSH – Division of Training

Practice Worksheet

Laboratory Quality Control $\bar{X}$-R Chart

Laboratory _____ Date _____

Method of Test or Operation _____

Reference Value _____ Increment of Measurement _____

Data

No.	X_1	X_2	X_3	$\bar{X}$	R
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Totals $\Sigma \bar{X}$ _____ ΣR _____

X_i = observed value R = largest – smallest

n = sets of values CL = control limit

Σ = summation WL = warning limit

U = upper L = lower

D_4 = 3.268 for n = 2; 2.574 for n = 3

A_2 = 1.880 for n = 2; 10023 for n = 3

Calculations

1. $\bar{R} = \Sigma R \div n$

   $\_\_ = \_\_\_ \div \_\_$

2. $UCL_R = D_4 \times \bar{R}$

   $\_\_\_ = \_\_ \times \_\_$

3. $UWL_R = 2/3(D_4\bar{R} - \bar{R}) + \bar{R}$

   $\_\_\_ = 2/3(\_\_\_ - \_) + \_\_$

4. $\bar{\bar{X}} = \Sigma \bar{X} \div n$

   $\_\_ = \_\_\_ \div \_\_$

5. $CL_{\bar{X}} = A_2 \times \bar{R}$

   $\_\_\_ = \_\_ \times \_\_$

6. $WL_{\bar{X}} = 2/3 \times CL_{\bar{X}}$

   $\_\_\_ = 2/3 \times \_\_\_$

7. $UCL_{\bar{X}} = \bar{\bar{X}} + CL_{\bar{X}}$

   $\_\_\_ = \_\_ + \_\_\_$

8. $UWL_{\bar{X}} = \bar{\bar{X}} + WL_{\bar{X}}$

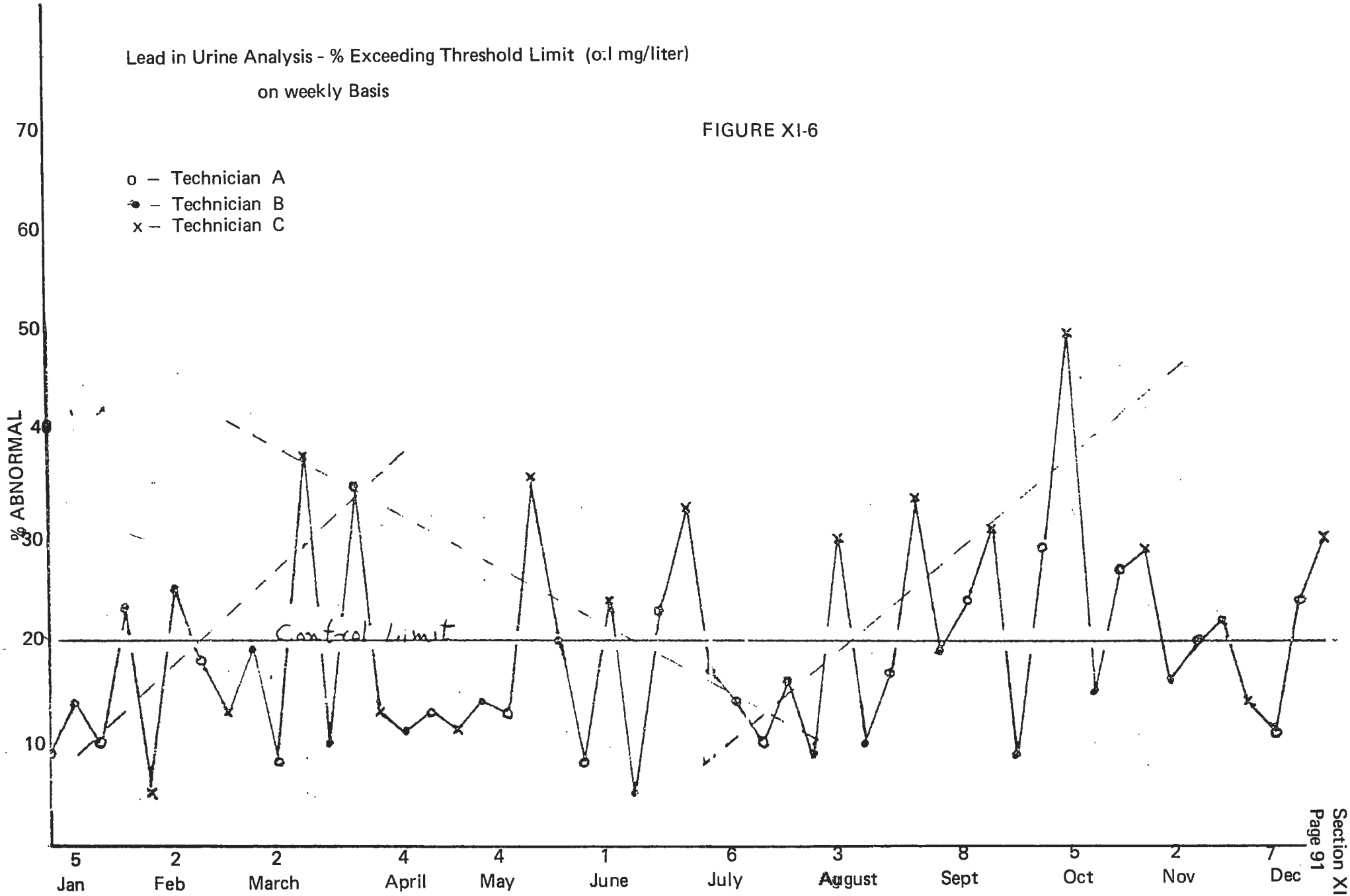
   $\_\_\_ = \_\_ + \_\_\_$

9. $LWL_{\bar{X}} = \bar{\bar{X}} - WL_{\bar{X}}$

   $\_\_\_ = \_\_ - \_\_\_$

10. $LCL_{\bar{X}} = \bar{\bar{X}} - CL_{\bar{X}}$

    $\_\_\_ = \_\_ - \_\_\_$


Lead in Urine Analysis - % Exceeding Threshold Limit (o:l mg/liter)
on weekly Basis
FIGURE XI-6
o – Technician A
• – Technician B
x – Technician C
% ABNORMAL
70
60
50
40
30
20
10
Control Limit
5 Jan
2 Feb
2 March
4 April
4 May
1 June
6 July
3 August
8 Sept
5 Oct
2 Nov
7 Dec

NIOSH – Division of Training
Practice Worksheet
Laboratory Quality Control $\bar{X}$–R Chart

Operation _____ Date _____

FIG XI-8

ACCURACY CONTROL CHART

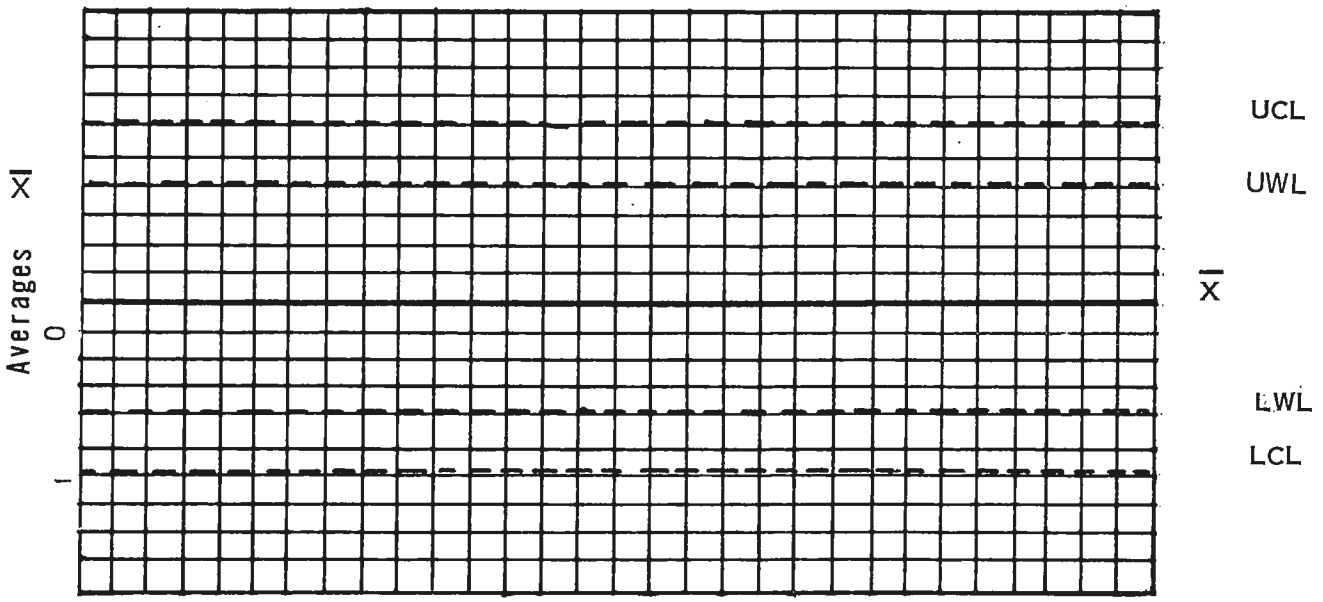
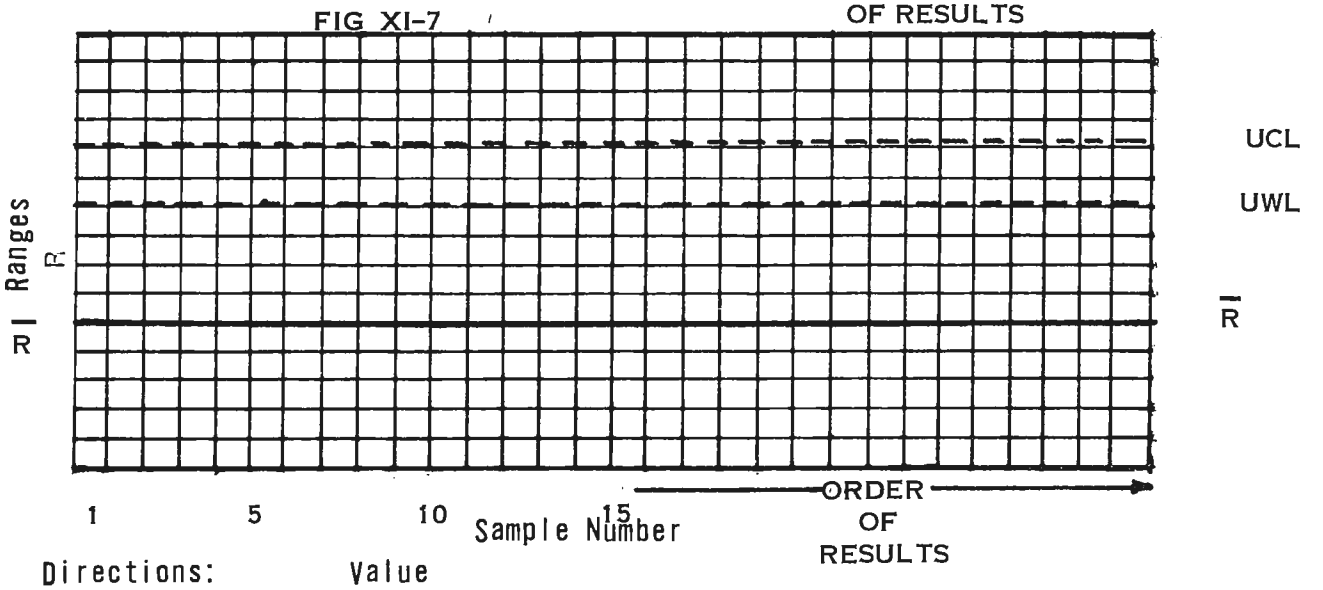


FIG XI-7

PRECISION CONTROL CHART



Directions: Value

1. Draw $\bar{R}$ line _____
2. Draw UCL_R line _____
3. Draw UWL_R line _____
4. Plot R's as generated
5. Draw $\bar{\bar{X}}$ line _____
6. Draw $UCL_{\bar{X}}$ line _____
7. Draw $UWL_{\bar{X}}$ line _____
8. Draw $LWL_{\bar{X}}$ line _____
9. Draw $LCL_{\bar{X}}$ line _____
10. Plot $\bar{X}$'s as generated

NIOSH – Division of Training

Practice Worksheet

Laboratory Quality Control $\bar{X}$–R Chart

Laboratory _____ Date _____

Method of Test or Operation _____

Reference Value _____ Increment of Measurement _____

Data

No.	X_1	X_2	X_3	$\bar{X}$	R
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Totals $\Sigma \bar{X}$ _____ ΣR _____

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   $\_\_ = \_\_\_ \div \_\_$

2. $UCL_R = D_4 \times \bar{R}$

   $\_\_\_ = \_\_ \times \_\_$

3. $UWL_R = 2/3(D_4\bar{R} - \bar{R}) + \bar{R}$

   $\_\_\_ = 2/3(\_\_\_ - \_) + \_\_$

4. $\bar{\bar{X}} = \Sigma \bar{X} \div n$

   $\_\_ = \_\_\_ \div \_\_$

5. $CL_{\bar{X}} = A_2 \times \bar{R}$

   $\_\_\_ = \_\_ \times \_\_$

6. $WL_{\bar{X}} = 2/3 \times CL_{\bar{X}}$

   $\_\_\_ = 2/3 \times \_\_\_$

7. $UCL_{\bar{X}} = \bar{\bar{X}} + CL_{\bar{X}}$

   $\_\_\_ = \_\_ + \_\_\_$

8. $UWL_{\bar{X}} = \bar{\bar{X}} + WL_{\bar{X}}$

   $\_\_\_ = \_\_ + \_\_\_$

9. $LWL_{\bar{X}} = \bar{\bar{X}} - WL_{\bar{X}}$

   $\_\_\_ = \_\_ - \_\_\_$

10. $LCL_{\bar{X}} = \bar{\bar{X}} - CL_{\bar{X}}$

    $\_\_\_ = \_\_ - \_\_\_$

GLOSSARY OF TERMS

ANALYSIS OF VARIANCE -

A technique of statistical analysis by which the importance of variation between components of sets of data is estimated.

BLANK ANALYSIS -

A laboratory technique employing control charts to determine accuracy and precision of a method by recording graphically in succession the results of methods used on controlled blank samples.

CENTRAL TENDENCY -

The tendency of values in a set of data to group around a center value. The measures of central tendency are: the mean, the mode and the median. (q.v.)

CHI SQUARE TEST -

1. A test for differences between two variabilities based on the Chi Square distribution χ^2.

2. The χ^2 test may also be used to estimate whether or not a set of values has a normal distribution.

CONTROL CHART - (also Shewhart Control Chart)

A graphic record of values, usually averages or ranges, or both, of sets of data, recorded successively.

CONTROL LIMIT -

Limits on a control chart are established statistically at $\pm$ 3 standard deviations from the mean. Plotted values falling outside the upper or lower control limit are said to be "out of control".

DEGREES OF FREEDOM -

A factor dependant on the number of values free from constraints imposed on a set of values or a number of sets of values interdependant. For example - in the statistic $\sigma = \sqrt{\frac{\Sigma(X - \bar{X})^2}{n-1}}$ we imposed one constraint - that the n deviations (d) must equal o ($\Sigma d = o$).

DETERMINATE (assignable) CAUSES -

Those causes of error which contribute to variation and can be (at least theoretically) detected and eliminated from the procedure.

F TEST -

A test for variability based on the F sampling distribution. A means of testing for differences between variances.

INDETERMINATE (random) CAUSES -

Those causes of error which do not have assignable causes and appear to follow only the laws of chance.

INTERNAL STANDARD -

See Section X, Par. 10.5.1.4.

MEAN - ARITHMETIC AVERAGE

$$\bar{X} = \frac{\sum X}{n}$$

The mean $\bar{X}$ is equal to the sum of units ($\sum X$) divided by the number of units (n).

MEASUREMENT STANDARDS -

Standards traceable to primary standards maintained in the laboratory which are traceable to National Standards.

MEDIAN -

The value in a set of data having the greatest number of occurrences of that value.

NORMAL CURVE -

A bell-shaped curve embracing 3 standard deviations on each side of the mean. Also called the Gaussian Curve. This distribution of values around the mean is found to occur naturally in many cases when data is generated for some purpose.

POPULATION -

The universe from which a set of sample values is drawn.

PRIMARY STANDARDS -

See measurement standards.

PROFICIENCY TESTS -

A laboratory technique using control charts or other means to determine the proficiency of the method and the analyst by recording the results of methods used on reference samples.

RANDOM SAMPLES -

Samples taken so that all units in the lot, population or universe have an equal chance of being selected in the sample.

RANGE -

In a set of data, the difference between the largest and the smallest values.

RECYCLE SAMPLE ANALYSIS -

A laboratory technique in which the analytical method is replicated on the sample or on aliquots thereof and the results recorded on precision (R) charts.

REPLICATION -

Independent testing or analysis of the same sample or samples by same technicians, or by different technicians.

RUGGEDNESS -

A method is said to be "rugged" when its precision and accuracy are stable despite the inevitable slight deviations in procedure encountered in routine work.

SAMPLE -

1. A random selection of units from a lot, population or universe.

2. An environmental or biological specimen collected by a field industrial hygienist and submitted to a laboratory for analysis.

SAMPLE SIZE -

1. Number of units in a sample, often prescribed by a sampling plan.

2. The physical size or volume of a sample submitted, ie., ft^3 of air sampled or ml of urine, etc.

SAMPLING ERROR -

The estimated error inherent in a sampling plan by which a small sample does not exactly represent the quality or other characteristics of the lot or the population.

SIGNIFICANT DIFFERENCES -

Differences in a set of results from some theoretical or target value by more than can be attributed to chance alone.

σ STANDARD DEVIATION -

The square root of the average of the squared deviations from the mean.

$$\sigma = \sqrt{\sum_{n=1}(X - \bar{X})^2}$$

STATISTICAL QUALITY CONTROL -

All those measures employed including administrative, investigative, management, research, and statistical techniques to ensure the attainment of stated quality objectives at an economic cost.

STUDENT'S t DISTRIBUTION -

(see t Test) A sampling distribution developed by W. S. Gossett under the pseudonym "Student".

t TEST -

A test for the significance of differences in sets of data based on the "t" distribution; a means of estimating the characteristics of a population from a small sample with assurance of accuracy at a chosen level. Also known as "Student's" t distribution. (Q.V.)

VARIABILITY -

A term encompassing the several measures of variation, including the range, standard deviation, variance, etc.

VARIANCE - σ^2 -

The square of the standard deviation. The arithmetic mean of the squares of the differences of individual values from their mean.

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We feel this manual to be a pioneering effort in its field. We, therefore, plan to revise it as user experience makes it advisable or necessary. Your comments, criticisms and suggestions are invited to make it a more useful instrument for all concerned. Please use this sheet and/or supplemental pages. Please mail them when completed to: Mr. John Bryant, Acting Deputy Director DLCD, NIOSH, 1014 Broadway, Cincinnati, Ohio 45202.

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