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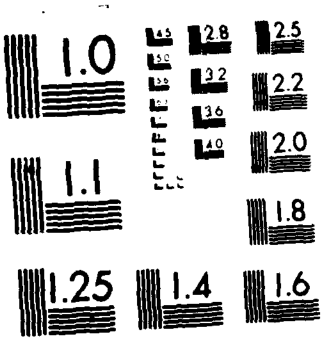

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NATIONAL BUREAU OF STANDARDS 1963-A

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QUALITY ASSURANCE GUIDE

FEBRUARY 1983

USAF Occupational and Environmental Health Laboratory
Aerospace Medical Division (AFSC)
Brooks Air Force Base,Texas 78235

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This guide on quality assurance provides instructions and other information pertinent to USAF OEHL's pursuit of producing high quality analytical work. It identifies the various performance evaluation programs in which the USAF OEHL participates. Also included are areas on quality control data handling, reagents, glassware and glassware cleaning.

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QUALITY ASSURANCE GUIDE

FEBRUARY 1983

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TABLE OF CONTENTS

List of Illustrations

Figures

Tables

USAF Occupational and Environmental Health Laboratory
Analytical Services Division
Quality Assurance

INTRODUCTION

Because of the decision making effects, based upon qualitative and quantitative data provided by the USAF Occupational and Environmental Health Laboratory Analytical Services Division (USAF OEHL/SA), it is essential that the data accurately describe the samples being analyzed. Faulty data may lead to a decision that would have been better made without data.

The overall quality of the work performed by SA is dependent upon many factors. These factors include, but are not limited to: quality of glassware, quality of reagents, quality and limitations of analytical equipment, analyst abilities and limitations, timeliness in processing samples, maintaining samples at the correct temperature between collection and analysis, analysis of a sufficient number of duplicate and reference samples to insure quality control, and keeping current with the procedures and methodology outlined by the various regulatory agencies.

Responsibility for quality assurance belongs to everyone involved with sample processing and reporting. The Quality Assurance Function has the responsibility for insuring that all analytical data generated in SA are as accurate as possible. This is accomplished by following acceptable procedures in analyzing the samples and establishing acceptable precision and accuracy limits. The precision and accuracy are evaluated by using split, spiked, reference, and standard samples. Laboratory reports are also evaluated for completeness before leaving the Laboratory.

QUALITY ASSURANCE WORK CENTER FUNCTIONS

The Quality Assurance Chief (QAC) works under the supervision of the Analytical Services Division Chief (Laboratory Director) (Figure 1). The QAC manages the overall quality assurance program and keeps the Division Chief informed as to progress and problem areas.

Goals of the Quality Assurance program are: to insure that samples are analyzed as timely as possible, to provide accurate results and maintain certification/accreditation by the appropriate federal, state and professional organizations.

Quality Assurance (QA) personnel are responsible for preparing internal reference samples and processing reference samples for analysis from external sources. The goal is to have approximately 10 percent of the work load as quality control samples. This 10 percent rate applies to the total analytical work load. In addition, approximately 10 percent of all samples received are to have duplicate analyses performed on them. The samples for replicate analyses are determined by the work center leader.

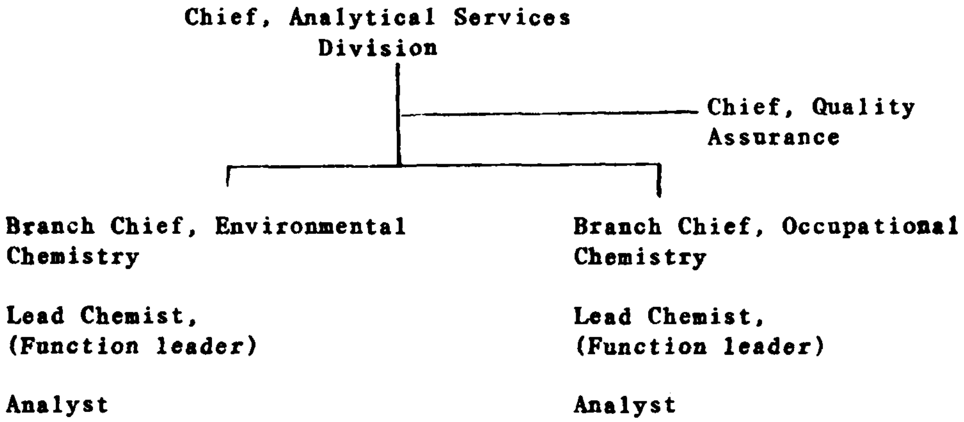

Figure 1. Organizational Chart, Analytical Services Division

QA personnel will utilize appropriate statistical procedures and control charts for evaluating laboratory results and providing feedback to the analysts. The QA personnel are also responsible for processing all samples, forms and associated information received for certification, approval or accreditation purposes from the Environmental Protection Agency (EPA), state agencies, NIOSH and CDC. They track the samples until analyzed, fill out the data sheets and mail to the evaluating agency. QA personnel are also to work with the analysts in assuring that the appropriate grade of chemicals, reagents and solvents are being employed in the laboratory.

Included in the evaluation samples received from external sources are: NIOSH Proficiency Analytical Testing (PAT) samples, interlaboratory correlation studies, Air Force Aviators Breathing Oxygen (ABO) correlation studies, EPA Water Supply Performance Evaluation Samples, EPA Water Pollution Performance Evaluation Samples, Center for Disease Control (CDC) Proficiency Testing Samples, Research Triangle Institute asbestos samples and performance samples from states.

Calibration of equipment will be accomplished by the user, according to manufacturer's instructions. QA personnel will provide log books, as needed, for the analyst to record results of the calibration checks, and will review the calibration notes.

ANALTYCIAL METHODS FILE

All analytical methods should be available in one central file. In addition, each Branch should maintain a file of methods for that Branch. Also, each function leader and analyst should have a file of the methods covering their areas of responsibility. Any and all deviations from the prescribed methods must be approved by the Division Chief and annotated, as appropriate, in each file.

The following items should be addressed during the preparation and typing of methods.

1. Scope and Application
2. Summary of Method
3. Range and Sensitivity
4. Interferences
5. Safety
6. Apparatus
7. Reagents
8. Calibration
9. Quality Control
10. Sample Storage
11. Daily Performance Tests
12. Sample Extraction and Analysis
13. Qualitative Identification
14. Quantitative Determination
15. References

The majority of the methods in use for Occupational Chemistry are from the "NIOSH Manual of Analytical Methods." The Environmental Chemistry Branch will be using methods found in the latest edition of "Standard Methods/Water and Wastewater" for most of their analyses. Additional methods that may be employed will be those recognized by the EPA or other recognized valid methods.

SAMPLE PROCESSING

The Sample Control Function is under the supervision of Quality Assurance. Just as critical as accurate analysis is analyzing samples within acceptable time frames if the analytical results are to be reliable. If the samples are processed in and delivered to the work centers efficiently, the actual in-house time for a sample can be reduced to a minimum.

Samples are delivered to the laboratory by various means. Once delivered, sample control personnel open the shipping container and remove the samples and paperwork. They are checked for breakage and leakage as well as accurate identification on the accompanying forms. If any problems are identified, the customer (Base) is notified by telephone or message so that corrections can be made. If everything is correct, the samples and forms are assigned matching OEHL control numbers. Next, the sample information is entered into a computer and progress is tracked by computer until the sample is analyzed and the results reported to the customer. The properly identified samples are delivered to the appropriate work center, refrigerator or freezer until analysis can be started.

During analysis of the samples, approximately 10 percent should be analyzed in duplicate for the analyst to evaluate precision of the method and instrument during the analytical procedure. Additionally, samples will be split and spiked by the Quality Assurance section. Each and every sample run should have a minimum of one duplicate evaluation per analyte.

For routine sample analyses, standards in combination with control charts should be sufficient to determine that the analyses are in or out of control. The charts are prepared by evaluating the analytical performance on synthetic controls over time. To evaluate interferences, due to sample matrices, the recovery of analyte spikes on split samples will also be employed.

Nonroutine sample analyses (performed infrequently) require special attention to document quality control. These are the analyses that are performed on a one-time basis or at least so infrequently that the compilation of extensive method data is unlikely. Samples in this category will be quality controlled by splitting and spiking portions with known amounts of the analyte of interest. A sufficient number of samples from a group (50 percent if more than 5 samples, 100 percent for 5 or fewer samples) should be split and spiked. All samples, including the spikes, should be processed through the entire analytical procedure. The percent recovery of the spike is calculated as follows:

$$\% \text{ recovery} = \frac{\text{Analyte Conc. (Spiked)} - \text{Analyte Conc. (unspiked)}}{\text{Conc. of Analyte added}} \times 100$$

Samples are also sent out to laboratories under contract for analysis. As a means of evaluating sample integrity during shipment and the contract laboratory's performance, spiked and synthetic samples will be included in shipments. The contract laboratories will also be required to furnish data on their quality control programs.

ANALYSIS OF QUALITY CONTROL DATA

The computer program used in analyzing quality control data generated at the USAF OEHL was developed by one of our contract laboratories, UBTL Division, University of Utah Research Institute, Salt Lake City, Utah. Warning limits are set at ±2 standard deviations and control limits are set at ±3 standard deviations for the control charts. All quality control samples are analyzed at least in duplicate. The following information is from the UBTL Analytical Chemistry Quality Control Manual (July 16, 1981).

Quality control charts are generated for each analyte through the analysis of quality control sample results. Each result is divided by the theoretical value to standardize results so that data from all concentrations can be directly compared. The ith sample result is then defined by the following parameters:

$$Xi = \frac{\text{Calculated Mean}}{\text{Theoretical Value}}$$

$$Ri = \frac{\text{Range of Values}}{\text{Theoretical Value}}$$

n_i = number of replicate determinations

When a control data set of N sample results has been accumulated, the following statistics are calculated:

Mean Percent Recovery

$$\bar{X} = \frac{\sum_{i=1}^{N} n_i X_i}{\sum_{i=1}^{N} n_i}$$

Standard Deviation Among Samples

$$\sigma = \sqrt{\frac{\sum_{i=1}^{N} \frac{(n_i X_i)^2}{n_i} - \bar{X}^2 \sum_{i=1}^{N} n_i}{N-1}}$$

Standard Deviation Among Replicates

$$\sigma' = \frac{\sum_{i=1}^{N} \frac{n_i R_i}{d_{2i}}}{\sum_{i=1}^{N} n_i}$$

Where d_{2i} is given in Table 1 and is used to convert ranges to standard deviations for sample size n_i.

Table 1

FACTORS FOR QUALITY CONTROL LIMITS

n	d_2	D_1	D_2	D'_1	D'_2
2	1.128	0	3.69	0	2.83
3	1.693	0	4.36	0	3.47
4	2.059	0	4.70	.30	3.82
5	2.326	0	4.92	.60	4.05
6	2.534	0	5.08	.84	4.23
7	2.704	.20	5.20	1.04	4.37
8	2.847	.39	5.31	1.21	4.49
9	2.970	.55	5.39	1.35	4.59
10	3.078	.69	5.47	1.48	4.67

d_2, D_1 and D_2 are taken directly from tables in the reference below.

D'_1, D'_2 correspond to two standard deviation limits for ranges and are calculated from a table of the studentized range.

These statistics are then used to determine accuracy and precision quality control limits. For each new quality control sample with n_j replicates and results X_j and R_j, quality control limits are:

	Lower	Upper
Accuracy Control Limits on X_j	$1.00 - \frac{3\sigma}{\sqrt{n_j}}$	$1.00 + \frac{3\sigma}{\sqrt{n_j}}$
Accuracy Warning Limits on X_j	$1.00 - \frac{2\sigma}{\sqrt{n_j}}$	$1.00 + \frac{2\sigma}{\sqrt{n_j}}$
Precision Control Limits on R_j	$D_1\sigma'$	$D_2\sigma'$
Precision Warning Limits on R_j	$D'_1\sigma'$	$D'_2\sigma'$

D_1, D_2, D'_1, D'_2, are given in Table 1.

The control data set is updated after evaluation of 20 successive quality control samples and includes data on the 50 most recent results. Any value X_i or R_i which is out of control is not used in the subsequent determination of new limits. Figures 2 and 3 show sample plots of the accuracy and precision control charts.

Reference

Grant, E., and R. Levenworth, Statistical Quality Control, New York, McGraw Hill, 1974.

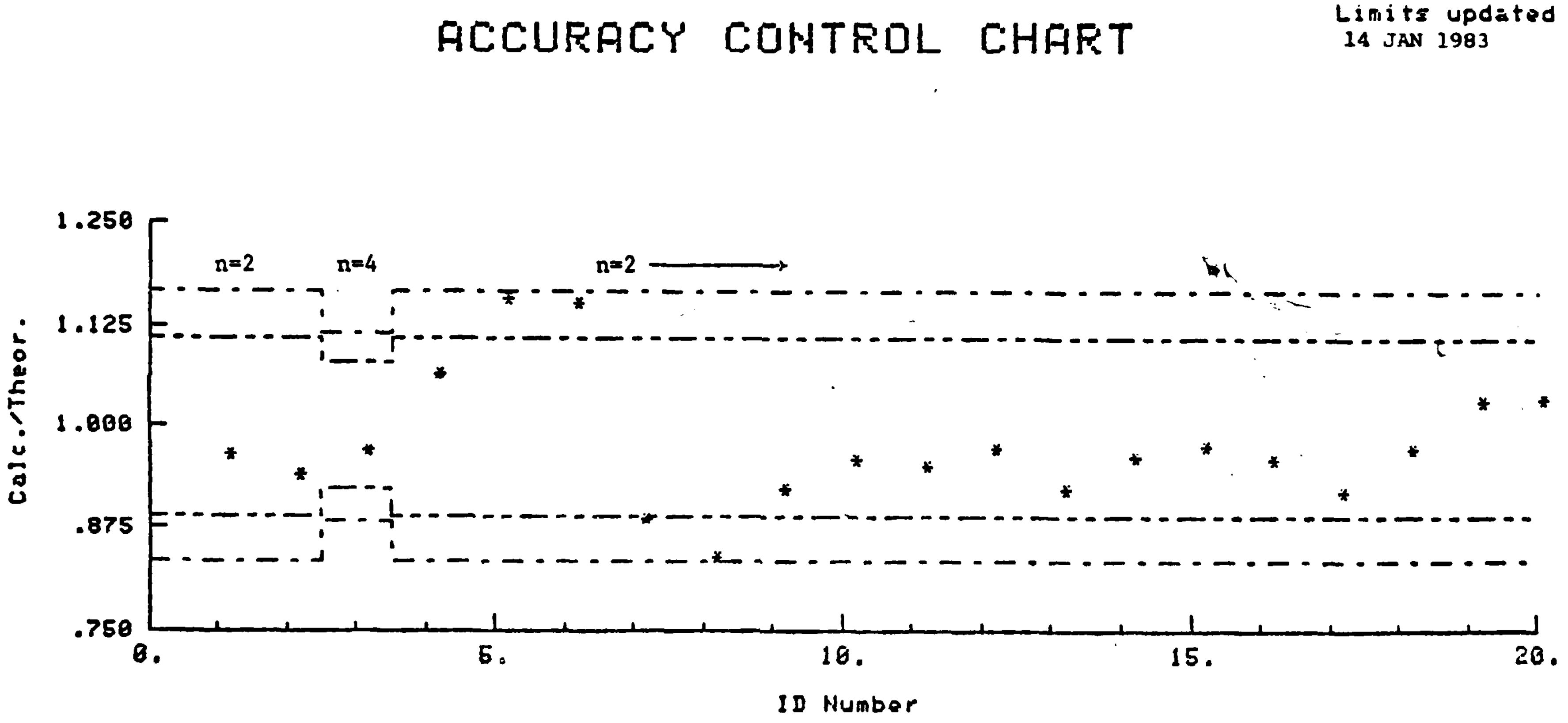

Figure 2. Accuracy Control Chart

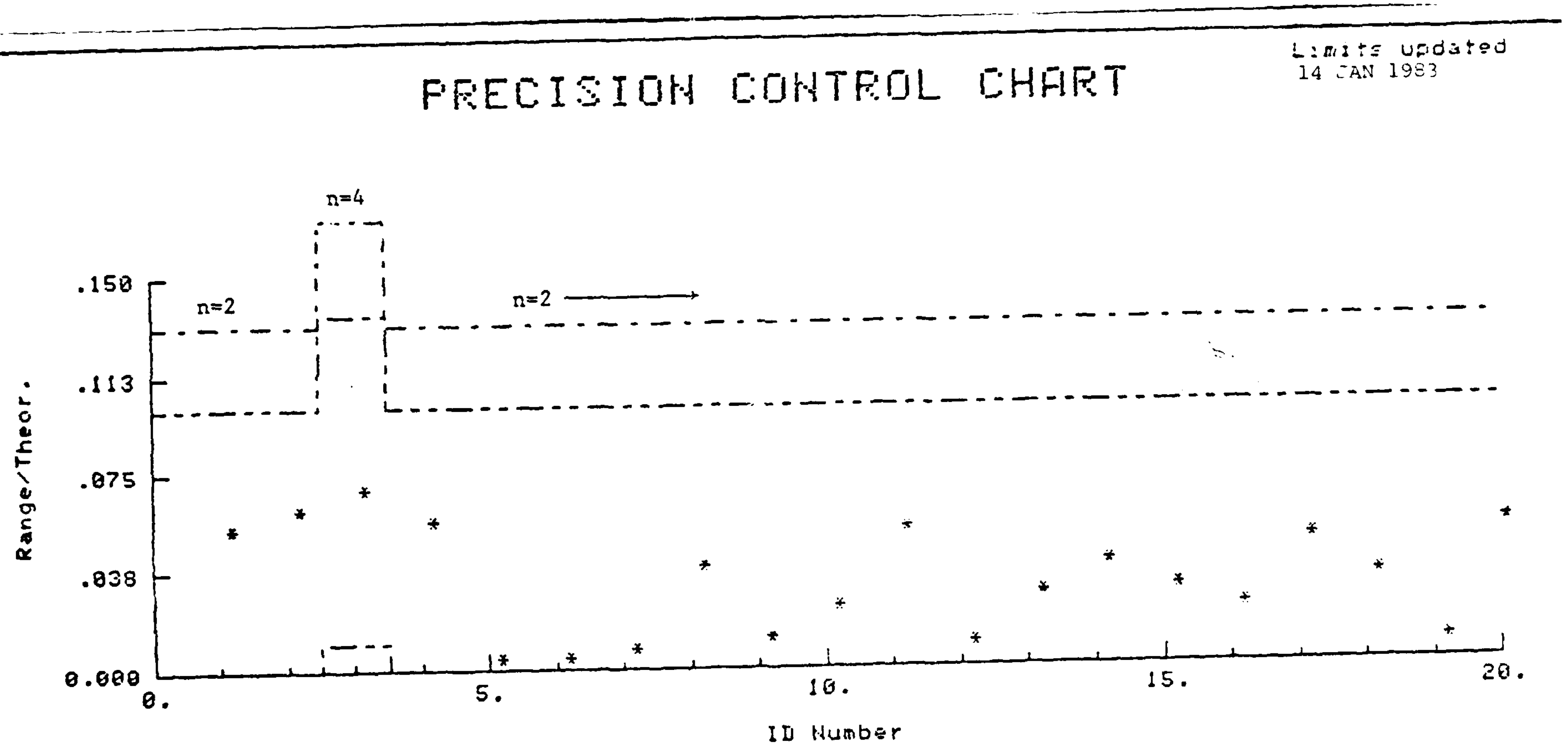

Figure 3. Precision Control Chart

When problems occur such as results falling outside the ±2 standard deviations warning limits for more than an occasional sample, or outside the ±3 standard deviations control limits for any sample, the analyst and quality control personnel will proceed as follows:

1. Check all sample data, including a recheck on all calculations.

2. Evaluate instrument performance by reviewing all charts and making another run if necessary.

3. Review the preparation of standards and prepare more if necessary. Also, prepare a new calibration curve and compare the new with the old if new standards are required.

4. Check blank samples to determine possible interferences.

5. Evaluate the integrity of the original QC samples by preparing and analyzing new ones.

6. If necessary reanalyze all samples, standards and QC controls, go through the entire procedure, including preparation, digesting, extraction, etc., as appropriate.

7. If for some unknown reason (unusual matrix, etc.) the QC results are still out of control after doing everything possible to determine the problem, discuss the problem with the requesting customer.

The acceptance or rejection of sample data is also based upon replicate sample analyses. The value of the range divided by the average for each sample replicated is used to make this determination. Based upon established criteria, UBTL uses the following as maximum acceptable range/average values:

Analysis	Range/Average Limit
AA (other than aspiration AA)	0.25
UV/Vis (colorimetric	0.05
Microscopy	0.50
Gravimetric	0.05
LC	0.20
All other analyses	0.10

These limits apply to all replicate values which are at least 10 times the limit of detection. We will use these same guidelines until such time that we might determine other limits to be more appropriate.

CALIBRATION AND MAINTENANCE OF EQUIPMENT

Routine calibration and performance checks will be performed each time an instrument is used. These checks are to be accomplished by following manufacturer's guidelines for instrument calibration and analyzing known concentrations of standards. Records are to be kept by each analyst (log book/folder) for calibration and performance checks. These will be reviewed by QA personnel.

Additional calibration and preventive maintenance checks will be performed by the Electronics Systems Branch (USAF OEHL/SUE) personnel, Maintenance Mgt Function (USAFSAM/TSNM) or through service contracts with equipment suppliers. If an instrument fails to operate properly during calibration or performance checks by the analyst and Branch Chief, a service representative will be called to correct the problem. Records of the corrective measures will be kept in the log book/folder for calibration and performance checks.

CHEMICAL REAGENTS, SOLVENTS, AND GASES

According to EPA's "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," 1979, the following are synonymous terms:

"Analytical Reagent Grade," "Reagent Grade" and "ACS Analytical Reagent Grade." In general, reagents graded as one of these will be satisfactory for inorganic analyses.

EPA recommends that all standards and reagents used in metals analyses be of spectroquality. Some of the organic analyses will require pesticide-quality reagents if low-level work is required. So, the grade or quality of reagents required will be dictated by the analyses being performed and the levels of detection needed.

Compressed gases used within the laboratory may range from commercial grade oxygen and nitrogen for determining total organic carbon to prepurified dry grade for GC work. Other special requirements may be needed for the compressed gases for particular analyses, such as oxygen traps, or having to be ultra-pure.

Primary standards are available through commercial chemical suppliers and should be used to prepare working standards. Some primary standards are also available through the National Bureau of Standards.

All reagent bottles should be marked as to the date they were opened and initialed by the individual who opened them. All working standards will be labeled as to concentration, date prepared, what it is and initialed by the individual preparing them. Always compare a new standard with the old by running an analysis to determine that the correct concentration was prepared.

GLASSWARE

Much of the following information on glassware was taken from the EPA "Handbook for Analytical Quality Control in Water and Wastewater Laboratories."

Laboratory vessels serve three functions: storage of reagents, measurement of solution volumes, and confinement of reactions. For special purposes, vessels made from materials such as porcelain, nickel, iron, aluminum, platinum, stainless steel, and plastic may be employed to advantage. Glass, however, is the most widely used material of construction. There are many grades and types of glassware from which to choose, ranging from student grade to others possessing specific properties such as super strength, low boron content, and resistance to thermal shock or alkali. Soft glass containers are not recommended for general use, especially for storage of reagents, because of the possibility of dissolving of the glass (or of some of the constituents of the glass). The mainstay of the modern analytical laboratory is a highly resistant borosilicate glass, such as that manufactured by Corning Glass Works under the name Pyrex^R or by Kimble Glass Co. as Kimax^R. This glassware is satisfactory for all analyses included in reference 1.

Depending on the particular manufacturer, various trade names are used for specific brands possessing special properties such as resistance to heat, shock, and alkalies. Examples of some of these special brands follow:

a. Kimax or Pyrex glass is a relatively inert all-purpose borosilicate glass.

b. Vycor^R glass is a silica glass (96 percent) made to withstand continuous temperatures up to 900°C and can be down-shocked in ice water without breakage.

c. Corning^R glass is claimed to be 50 times more resistant to alkalies than conventional ware and practically boron-free (maximum 0.2 percent).

d. Ray-Sorb^R or Low-Actinic^R glass is used when the reagents or materials are light sensitive.

e. Corex^R labware is harder than conventional borosilicates and, therefore, better able to resist clouding and scratching.

The use of plastic vessels, containers, and other apparatus made of Teflon^R, polyethylene, polystyrene, and polypropylene has increased markedly over recent years. Some of these materials such as Teflon, are quite expensive; however, Teflon stopcock plugs have practically replaced glass plugs in burets, separatory funnels, etc., because lubrication to avoid sticking or "freezing" is not required. Polypropylene, a methylpentene polymer, is available as laboratory bottles, graduates, beakers, and even volumetric flasks. It is crystal clear, shatterproof, autoclavable, and chemically resistant.

The following are some points to consider in choosing glassware or plastic ware:

a. The special types of glass listed above, other than Pyrex or Kimax, generally are not required to perform the analyses given in "Methods for Chemical Analysis of Water and Wastes"(1).

b. Unless instructed otherwise, borosilicate or polyethylene bottles may be used for the storage of reagents and standard solutions.

c. Dilute metal solutions are prone to plate out on container walls over long periods of storage. Thus, dilute metal standard solutions must be prepared fresh at the time of analysis.

d. For some operations, disposable glassware is entirely satisfactory. One example is the use of disposable test tubes as sample containers for use with the Technicon Automatic Sampler.

e. Plastic bottles of polyethylene and Teflon have been found satisfactory for the shipment of water samples. Strong mineral acids (such as sulfuric acid) and organic solvents will readily attack polyethylene and are to be avoided.

f. Borosilicate glassware is not completely inert, particularly to alkalies; therefore, standard solutions of silica, boron, and the alkali metals are usually stored in polyethylene bottles.

For additional information, the reader is referred to the catalogs of the various glass and plastic manufacturers. These catalogs contain a wealth of information such as specific properties, uses, and sizes.

Volumetric

By common usage, accurately calibrated glassware for precise measurements of volume has become known as volumetric glassware. This group includes volumetric flasks, volumetric pipets, and accurately calibrated burets. Less accurate types of glassware including graduated cylinders and serological and measuring pipets also have specific uses in the analytical laboratory when exact volumes are not required.

The precision of volumetric work depends in part upon the accuracy with which volumes of solutions can be measured. There are certain sources of error that must be carefully considered. The volumetric apparatus must be read correctly; that is, the bottom of the meniscus should be tangent to the calibration mark. There are other sources of error, however, such as changes in temperature, which result in changes in the actual capacity of glass apparatus and in the volume of the solutions. The capacity of an ordinary glass flask of 1000-mL volume increases 0.025 mL/deg with rise in temperature, but if the flask is made of borosilicate glass, the increase is much less. One thousand mL of water, or of most 0.1 N solutions, increases in volume by approximately 0.20 mL/deg increase at room temperature. Thus, solutions must be measured at the temperature at which the apparatus was calibrated.

This temperature (usually 20°C) will be indicated on all volumetric ware. There may also be errors of calibration of the apparatus; that is, the volume marked on the apparatus may not be the true volume. Such errors can be eliminated only by recalibrating the apparatus or by replacing it.

Volumetric apparatus is calibrated to contain or to deliver a definite volume of liquid. This will be indicated on the apparatus with the letters "TC" (to contain) or "TD" (to deliver). Volumetric flasks are calibrated to contain a given volume and are available in various shapes and sizes.

Volumetric pipets are calibrated to deliver a fixed volume. The usual capacities are 1 through 100 mL although micropipets are also available. Micropipets are most useful in furnace work and are available in sizes ranging from 1 to 100 µL.

In emptying volumetric pipets, they should be held in a vertical position and the outflow should be unrestricted. The tip of the pipet is kept in contact with the wall of the receiving vessel for a second or two after the free flow has stopped. The liquid remaining in the tip is not removed; <u>this is most important</u>.

Measuring and serological pipets should also be held in a vertical position for dispensing liquids; however, the tip of the pipet is only touched to the wet surface of the receiving vessel after the outflow has ceased. For those pipets where the small amount of liquid remaining in the tip is to be blown out and added, indication is made by a frosted band near the top.

Burets are used to deliver definite volumes. The more common types are usually of 25- or 50- mL capacity, graduated to tenths of a milliliter, and are provided with stopcocks. For precise analytical methods in microchemistry, microburets are also used. Microburets generally are of 5- or 10-mL capacity, graduated in divisions of hundredths of a milliliter. Automatic burets with reservoirs are also available ranging in capacity from 10 to 100 mL. Reservoir capacity ranges from 100 to 4,000 mL.

General rules in regard to the manipulation of a buret are as follows: Do not attempt to dry a buret that has been cleaned for use, but rinse it two or three times with a small volume of the solution with which it is to be filled. Do not allow alkaline solutions to stand in a buret because the glass will be attacked, and the stopcock, unless made of Teflon, will tend to freeze. A 50-mL buret should not be emptied faster than 0.7 ml/sec, otherwise too much liquid will adhere to the walls and as the solution drains down, the meniscus will gradually rise, giving a high false reading. <u>It should be emphasized that improper use or reading of burets can result in serious calculation errors</u>.

Glassware used for delivering liquids, must be absolutely clean so that the film of liquid never breaks at any point. Careful attention must be paid to this fact or the required amount of solution will not be delivered. The various cleaning agents and their use are described later.

Reference 2 contains a description of federal specifications for volumetric glassware. The National Bureau of Standards (NBS) no longer accepts stock quantitites of volumetric apparatus from manufacturers or dealers for certification and return for future sale to consumers. This certification service is still available, but apparatus will be tested only when submitted by the ultimate user, and then only after an agreement has been reached with the Bureau concerning the work to be done.

Consequently, the various glass manufacturers have discontinued the listing of NBS-certified ware. In its place, catalog listings of volumetric glass apparatus that meet the federal specifications are designated as class A and all such glassware is permanently marked with a large "A." These NBS specifications are listed in Table 2. The glassware in question includes the usual burets, volumetric flasks, and volumetric pipets.

In addition to the "A" marking found on calibrated glassware and the temperature at which the calibration was made, other markings also appear. These include the type of glass, such as Pyrex, Corex, or Kimax; the stock number of the particular item; and the capacity of the vessel. If the vessel contains a ground-glass connection, this will also be included along with the TD or TC symbol. An example of the markings usually found on volumetric ware is shown in Figure 4. Class A glassware need not be recalibrated before use. However, should it become necessary to calibrate a particular piece of glassware, directions may be found in text (4) on quantitative analysis.

Table 2

TOLERANCES FOR VOLUMETRIC GLASSWARE[1]

Type of Glassware	Capacity[2] (mL)	Limit of Error (mL)
Graduated flask	25	0.03
	50	0.05
	100	0.08
	200	0.10
	250	0.11
	300	0.12
	500	0.15
	1,000	0.30
	2,000	0.50
Transfer pipet	2	0.006
	5	0.01
	10	0.02
	25	0.025
	30	0.03
	50	0.05
	100	0.08
	200	0.10
Buret[3]	5	0.01
	10	0.02
	30	0.03
	50	0.05
	100	0.10

[1] Abridged from reference 3.
[2] Less than and including.
[3] Limits of error are of total or partial capacity.

NOTE: Customary practice is to test the capacity at five intervals.

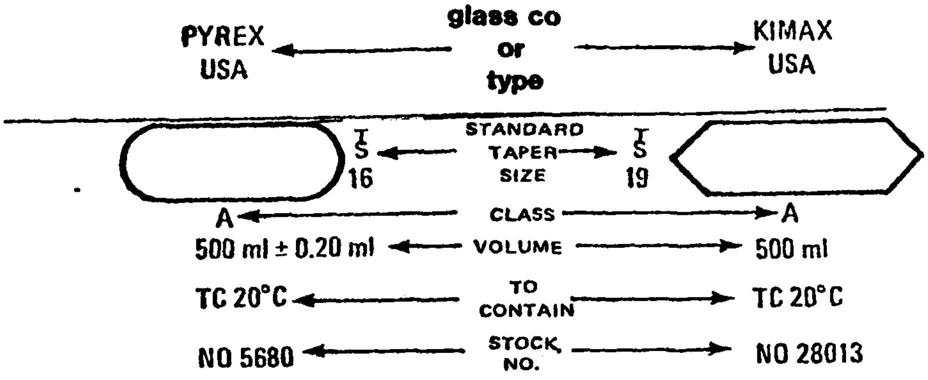

Figure 4. Volumetric Ware Markings

Disposable

When the risk of washing a pipet for reuse becomes too great, as in the case of use with toxic materials, or when the cost of washing glassware becomes prohibitive, disposable vessels may be the answer, provided they meet the necessary specification. Various types are available including bacteriological, serological, and microdilution pipets. Disposable glassware generally is made of soft glass although plastic vessels and pipets are also available.

Specialized

The use of vessels and glassware fitted with standard-taper, ground-glass, and ball-and-socket joints has increased because of certain advantages such as less leakage and fewer freezeups. Standard-taper, interchangeable ground joints save time and trouble in assembling apparatus. They are precision ground with tested abrasives to insure an accurate fit and freedom from leakage. Ball-and-socket joints increase flexibility of operation and eliminate the need for exact alinements of apparatus. Symbols and their meaning as applied to standard joints, stoppers and stopcocks are shown below.

Standard Taper

The symbol $\underset{S}{T}$ is used to designate interchangeable joints, stoppers, and stopcocks that comply with the requirements of reference 5. All mating parts are finished to a 1:10 taper.

The size of a particular piece appears after the appropriate symbol. Primarily because of greater variety of apparatus equipped with $\underset{S}{T}$ fittings, a number of different types of identifications are used.

a. For joints--a two-part number as $\underset{S}{T}$ 24/40, with 24 being the approximate diameter in millimeters at the large end of the taper and 40 the axial length of taper, also in millimeters.

b. For stop-cocks--a single number, as $\underset{S}{T}$ 2, with 2 mm being the approximate diameter of the hole or holes through the plug.

c. For bottles--a single number, as $\underset{S}{T}$ 19, with 19 mm being the approximate diameter at top of neck. However, there are differences between the bottle and flask stoppers.

d. For flasks and similar containers--a single number, as $\underset{S}{T}$ 19, with 19 mm being the approximate diameter of the opening at top of neck.

Spherical Joints

The designation $\underset{J}{S}$ is for spherical (semiball) joints complying with reference 5. The complete designation of a spherical joint also consists of a two-part number, as 12/2, with 12 being the approximate diameter of the ball and 2 the bore of the ball and the socket, also in millimeters.

Product Standard

The symbol $\underset{S}{P}$ is used for stopcocks with Teflon plugs, the mating surfaces being finished to a 1:5 taper. As with $\underset{S}{T}$ stopcocks, a single number is used. Thus, $\underset{S}{P}$ 2 means a Teflon stopcock with a hole of approximately 2-mm diameter in the plug.

Fritted Ware

For certain laboratory operations the use of *fritted ware for* filtration, gas dispersion, absorption, or extractions may be advantageous.

There are six different porosities of fritted ware available, depending on its intended use. Porosity is controlled in manufacture, and disks are individually tested and graded into these classifications. The extra-coarse and coarse porosities are held toward the maximum pore diameters as listed. The medium, fine, very fine, and ultrafine are held toward the minimum pore diameter as listed in Table 3.

Pore sizes are determined by the method specified in reference 6.

Recommended Procedures for Maximum Filter Life

a. New Filters. Wash new filters by suction with hot hydrochloric acid, followed by a water rinse.

b. Pressure Limits. The maximum, safe, differential pressure on a disk is 15 lb/in^2.

c. Thermal Shock. Fritted ware has less resistance to thermal shock than nonporous glassware. Hence, excessive, rapid temperature changes and direct exposure to a flame should be avoided. Heating in a furnace to 500°C may be done *safely, provided* the heating and cooling are gradual. Dry ware may be brought to constant weight by heating at 105°C to 110°C.

Never subject a damp filter of ultrafine porosity to a sudden temperature change. Steam produced in the interior may cause cracking.

Table 3

FRITTED-WARE POROSITY

Porosity Grade	Designation	Pore Size (μm)	Principle Uses
Extra Coarse	EC	170-220	Coarse filtration; gas dispersion, washing, and absorption
Coarse	C	40-60	Coarse filtration; gas dispersion, washing, and absorption
Medium	M	10-15	Filtration and extraction
Fine	F	4-5.5	Filtration and extraction
Very Fine	VF	2-2.5	General bacterial filtration
Ultrafine	UF	0.9-1.4	General bacterial filtration

Cleaning of Glass and Porcelain

The method of cleaning should be adapted to both the substances that are to be removed, and the determination to be performed. Water-soluble substances are simply washed with hot or cold water, and the vessel is finally rinsed with successive small amounts of distilled water. Other substances more difficult to remove may require the use of a detergent, organic solvent, dichromate cleaning solution, nitric acid, or aqua regia (25 percent by volume concentrated HNO_3 in concentrated HCl). In all cases it is good practice to rinse a vessel with tap water as soon as possible after use. Material allowed to dry on glassware is much more difficult to remove.

Volumetric glassware, especially burets, may be thoroughly cleaned by a mixture containing the following 30 g of sodium hydroxide, 4 g of sodium hexametaphosphate (trade name, Calgon), 8 g of trisodium phosphate, and 1 L of water. A gram or two of sodium lauryl sulfate or other surfactant will improve its action in some cases. This solution should be used with a buret brush.

Dichromate cleaning solution (chromic acid) is a powerful cleaning agent; however, because of its destructive nature upon clothing and upon laboratory furniture, extreme care must be taken when using this mixture. If any of the solution is spilled, it must be cleaned up immediately. Chromic acid solution may be prepared in the laboratory by adding 1 L of concentrated sulfuric acid slowly, with stirring to a 35-ml saturated sodium dichromate solution. This mixture must be allowed to stand for approximately 15 min in the vessel that is being cleaned and may then be returned to a storage bottle. Following the chromic acid wash, the vessels are rinsed thoroughly with tap water, then with small successive portions of distilled water. The analyst should be cautioned that when chromium is included in the scheme of analysis, it is imperative that the last traces of dichromate be removed from the apparatus. To this end, a substitute for dichromate cleaning solution may be used to advantage.

We will routinely use a substitute for the dichromate cleaning solution. Chromic acid cleaning solution will only be used for special occasions when a substitute fails to do the job.

The glassware used in the metals laboratory will be cleaned as follows:

1. Rinse thoroughly in tap water.
2. If a visible residue is still present, run the glassware through a dishwasher; otherwise, follow succeeding steps.
3. Rinse well with deionized water and immerse in nitric acid bath for at least 24 hours (1:5 nitric acid-water mixture, 2 bottles of concentrated HNO_3 and 10 bottles of deionized water).
4. Remove and rinse 10-12 times in deionized water.
5. Dry in an oven for 3 hours or longer at 350°F.

Cleaning of glassware used in the analyses for pesticides will be as follows:

1. Rinse thoroughly in tap water.
2. Soak in Contact 70* solution (chromic acid substitute), for 5 minutes.
3. Rinse in tap water.
4. Soak in a mixture of concentrated sulfuric acid and water 1:1.
5. Machine wash and rinse (without a cleaning agent).
6. Dry in an oven at approximately 350°F for a minimum of 3 hours.

Cleaning of glassware used in the trace organics work center will be as follows:

1. Rinse thoroughy in tap water.
2. Soak in Contact 70* for 5 minutes.
3. Rinse in tap water.
4. Soak in sulfuric acid/Chromerge^R mixture (chromic acid substitute).
5. Machine wash and rinse (without a cleaning agent).
6. Dry in an oven at approximately 350°F for a minimum of 3 hours.

Routine glassware cleaning will be as follows:

1. Rinse thoroughly in tap water.

2. Wash in the glassware washing machine with detergent followed by deionised water rinses.

3. Dry in an oven at approximately 350°F for 3 hours or longer.

Other Glassware Cleaning

Absorption cells, used in spectrophotometers, should be free of scratches, fingerprints, smudges, and evaporated film residues. The cells may be cleaned with detergent solutions for removal of organic residues, but should not be soaked for prolonged periods in caustic solutions because of the possibility of etching. Organic solvents may be used to rinse cells in which organic materials have been used. Nitric acid rinses are permissible, but dichromate solutions are not recommended because of the adsorptive properties of dichromate on glass. Rinsing and drying of cells with alcohol or acetone before storage is the preferred practice. Matched cells should be checked to see that they are equivalent by placing portions of the same solution in both cells and taking several readings of the transmittance (T, percent) or optical density (OD) values.

Glassware to be used for phosphate determinations should not be washed with detergents containing phosphates. This glassware must be thoroughly rinsed with tap water and distilled water. For ammonia and Kjeldahl nitrogen, the glassware must be rinsed with ammonia-free water.

Cleaning of Used Filters

In many cases, precipitates can be removed by rinsing with water, passed through from the underside, with the pressure not exceeding 15 lb/in^2. The suggestions that follow will be helpful in dealing with material that will not be removed by such a reverse water wash.

Material	Removal Agent
Albumen............................	Hot ammonia or hydrochloric acid
Aluminous and siliceous residues.....	Hydrofluoric acid (2 percent) followed by concentrated sulfuric acid; rinse immediately with water until no trace of acid can be detected.
Copper or iron oxides...............	Hot hydrochloric acid plus potassium chlorate
Fatty materials.....................	Carbon tetrachloride

Material	Removal Agent
Mercuric sulfide......................	Hot aqua regia
Organic matter........................	Hot, concentrated cleaning solution, or hot concentrated sulfuric acid with a few drops of sodium nitrite.
Silver chloride......................	Ammonium or sodium hyposulfite

REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Office of Research and Development, EMSL (1978).

2. Hughes, J.C., Testing of Glass Volumetric Apparatus, NBS Circular 602, National Bureau of Standards (1959).

3. Peffer, E.L., and Mulligan, G.C., Testing of Glass Volumetric Appartus, NBS Circular 434, National Bureau of Standards (1941).

4. Willard, H.H., and Furman, N.H., Elementary Quantitative Analysis-Theory and Practice, D. Van Nostrand Co., Inc., New York (1947).

5. Interchangeable Ground Glass Joints, Commercial Standard CS-21-30, National Bureau of Standrds (Aug 25, 1930).

6. Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use, E-128, American Society for Testing and Materials, Philadelphia (1968).

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