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Standard Guide for Accountability and Quality Control in the Chemical Analysis Laboratory¹

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1. Scope

1.1 This guide covers the essential aspects of an accountability and quality control program for a chemical analysis laboratory. The reasons for establishing and operating such a program are discussed.

2. Referenced Documents

2.1 *ASTM Standards:*

MNL 7 *Manual on Presentation of Data and Control Chart Analysis*²

2.2 *ANSI Document:*

ANSI/ASQC A1 *Definitions, Symbols, Formulas, and Tables for Control Charts*³

3. Significance and Use

3.1 An accountability and quality control system is established by laboratory management to improve the quality of its results. It provides documented records which serve to assure users of the laboratory's services that a specified level of precision is achieved in the routine performance of its measurements and that the data reported were obtained from the samples submitted. The system also provides for: early warning to analysts when methods or equipment begin to develop a bias or show deterioration of precision; the protection and retrievability of data (results); traceability and control of samples as they are processed through the laboratory; good communication of sample information between submitters, analysts, and supervision; and information on sample processing history. This guide describes such a system. Other accountability and quality control programs can be developed. Such

programs can be equivalent to the program in this guide if they provide all of the benefits mentioned above.

4. Accountability

4.1 Accountability means assurance that the results reported refer directly to the samples submitted.

4.2 Prior to submitting samples to the laboratory, the prospective user should consult with laboratory personnel concerning his needs and the capability of the laboratory to satisfy them. It is the responsibility of the originator of the samples to select and identify proper samples for submission to the laboratory, to decide what information is required (especially, to define the use to be made of the information), and, after consulting with laboratory personnel, to submit the samples in suitable containers, properly labeled, and accompanied by written instructions identifying the samples, their nature, and the information sought through chemical analysis. This should be done formally, using a well-defined document for information transfer to initiate work in the laboratory.

4.3 Laboratory management establishes a written accountability system to be used throughout the laboratory at all times. This implies traceability and documentation of all reported results through the laboratory back to the submitted sample. This system should have the following general characteristics:

4.3.1 Each nonroutine job submitted by a user of the laboratory's services is assigned an internal laboratory identification number (ID), which is used to correlate all samples, work, time and cost accounting, consultation, and reports and other paperwork associated with that job. The final report that is returned to the originator will always bear the number (ID) for future reference. Moreover, it is convenient for laboratory data to be filed according to sequential ID numbers. For example, "86/0428" might identify the associated work as the 428th request submitted in the year 1986. The *Data Record* should provide all data generated during the analyses, names of persons performing the analyses, dates the analyses were performed, and any unusual occurrences that happened during the analyses. Accountability for production control samples is normally maintained separately from the nonroutine records because results from production control samples are usually

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² ASTM Manual Series, ASTM, 6th Edition, 1990.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

reported on routine report forms, the samples being identified with the day, shift, run, or lot from which they were taken.

4.3.2 Each sample, specimen, sample site, or other unique piece of material or container identified as a separate sample by the originator should be assigned a sequential item number (NN) for internal laboratory use. As soon as the samples are accepted by the laboratory, laboratory personnel will mark each sample or sample container with its own laboratory sample number (ID-NN) in such manner that the label is not likely to become separated from its sample or rendered unreadable during its residence in the laboratory. For example, the fifth sample on the above-mentioned request might be identified as “86/0428-05.”

4.3.3 All laboratory work records, intermediate sample containers, data, and reports for a specific sample will be identified by the same laboratory identification and item number to avoid any opportunity for samples or data to be lost or intermixed within or between jobs.

4.3.4 The first and last steps in the accountability procedure are functions of technical supervision. Before any work is performed, the compatibility of the work requested with the physical condition of the samples and the capabilities of the laboratory must be verified. When the analysts have completed their work, the results must be reviewed to be certain that all information requested has been determined and that the work has been performed with the required care and precision. In this latter regard, quality control procedures prove invaluable both to the analysts performing the work and the reviewing supervisor. The supervisor also verifies that the results are calculated in units that are most meaningful to the submitter and that the units and basis on which the results are calculated are clearly stated.

4.3.5 Except for the most routine work, the original analyst's data book, a serial listing of laboratory identification numbers and descriptions, and a copy of each job report sheet are retained in the laboratory's records for the periods of time established by laboratory policy. Intermediate calculations and samples are normally discarded after the submitter has had a reasonable opportunity to submit questions concerning the results and request return of his samples. In some cases, customer specifications may dictate the records that must be retained and the retention times for both analytical records and laboratory samples.

5. Quality Control

5.1 Quality control of analytical methods provides the information needed to ensure that procedures, equipment, and personnel are performing at the levels of precision and accuracy required by the intended use of the data.

5.2 *General Characteristics*—The following factors have been found helpful in maximizing the effectiveness and minimizing the cost of quality control procedures:

5.2.1 Involve the operators or analysts who actually perform the work to the greatest possible extent.

5.2.2 Use the simplest, most direct statistical procedures that will provide the necessary degree of control. This means that graphical or simplified arithmetic procedures are preferred.

5.2.3 Perform the quality control measurements as early in the measurement process as possible. This prevents waste of

analytical effort if the method is not initially in control. However, when a prolonged series of measurements is made, it is also necessary to verify that the method remains in control throughout the run.

5.2.4 Provide specific action limits and describe exactly what must be done when these limits are exceeded.

5.2.5 For each method (for each sample type), choose a control material that is known to be stable and homogeneous and has measured values within the range of interest. Any inhomogeneity in the control sample will add to the variance of the results. Any increase in variability that is not related to the measurement process will reduce the sensitivity of the quality control procedure to detect changes in the measurement process. Where possible, the control material should be similar to the samples to be analyzed. Obtain as large an amount of control material as can be prepared in a homogeneous state because considerable effort is required to prepare a new control. Always prepare a new control material well in advance of exhausting the old one so that the new chart is ready when needed. In situations where satisfactory control material cannot be obtained, alternative techniques (such as, retest by a senior analyst) may be substituted for the control chart approach.

5.2.6 Give analysts specific instructions concerning their response to an out-of-control condition. Supervision may decide that, if the analyst can correct the problem so that the control sample results again plot within limits, the process may continue without immediate contact with the supervisor. In other situations, the supervisor may need to become involved with each out-of-control incident. In either case, adjustments to the process should be recorded to explain each shift in the control measurements.

5.2.7 Provide for a periodic in-depth review by supervision and management of the overall effectiveness of the laboratory quality control system. Operating experience may indicate that methods should be added to, or dropped from the program, that the frequency of specific control samples should be increased or decreased, or that a different strategy might be more appropriate for control of a specific method. The interval for such reviews should be determined by the uniformity of the processes that generate the samples. Any anticipated or observed change in the character of the samples being analyzed should initiate at least a cursory review of the control procedures for the methods that apply to those samples.

5.3 *Laboratory Quality Control Strategies*—Control chart methods are suitable for laboratory quality control programs. The choice of which control strategy to use depends on circumstances: the type of instrument or laboratory procedure, the number of samples and frequency of the analyses, and the closeness of control required. The following are appropriate:

5.3.1 The \bar{X} - and R -chart method is most frequently used. The control sample is run two or more times during the run, batch, or shift. The average is plotted on the \bar{X} -chart and the absolute value of the difference between the high and low values, the range, is plotted on the R -chart. If the average falls between the upper and lower control limits and the range falls below the upper control limit, the process is considered to be in control. Fig. 1 shows the essential features of charts for averages and ranges.

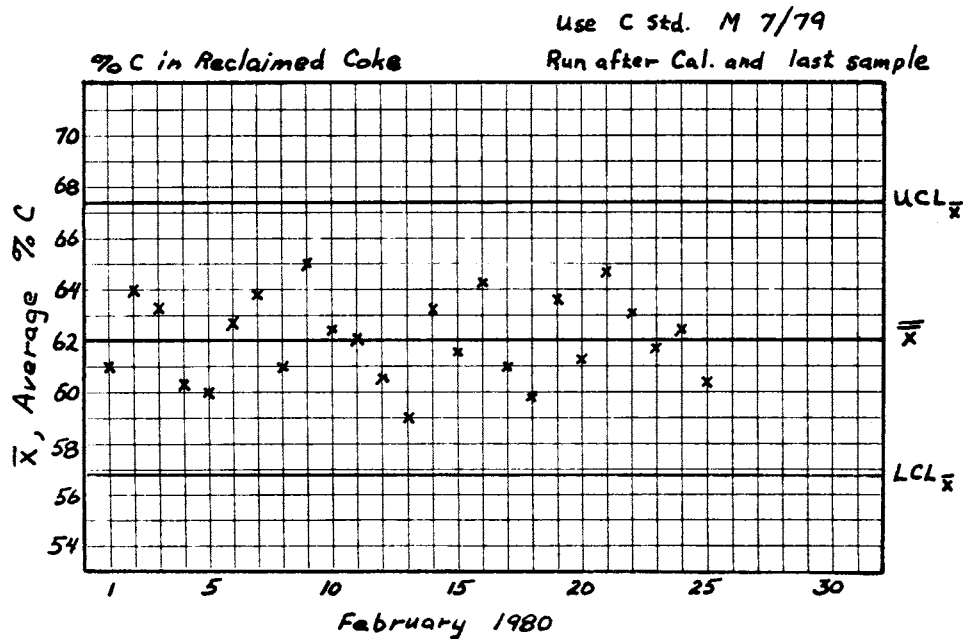


FIG. 1 Control Chart for Averages

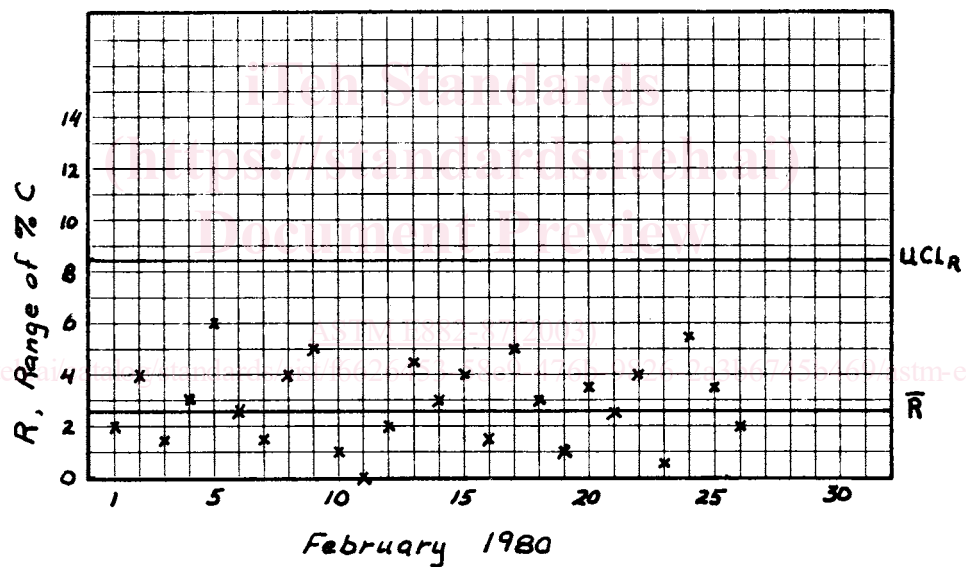


FIG. 2 Control Chart for Ranges

5.3.2 The X-chart method (often called the control chart for individuals) is useful for measurements that are made on a frequent or continual basis. It is appropriate for methods or instruments for which the usual mode of failure produces relatively large shifts in results and the cost of a determination precludes performing replicate analyses of control samples. Its main characteristic is that it responds rapidly to sudden relatively large changes in the analytical process, but it is not as sensitive to small changes as the \bar{X} - and R -chart method. Each time the control material is analyzed, its value is plotted on the X-chart. If the point plots between the upper and lower control limits, the analytical process is considered to be in control. Fig. 3 shows the essential features of charts for individuals.

5.3.3 A combination of the above two methods constitutes a useful strategy. A fixed number of control sample runs are made during a period that samples are being analyzed (such period could, for example, be a shift or a day in a continuous analysis process). Each individual value is plotted on the X-chart as the measurement is completed. Their average value and range are plotted on the \bar{X} - and R -charts. The additional effort to prepare and maintain both types of control charts may be justified in situations where erroneous assays would cause large economic losses. Other control chart techniques that may be appropriate for special circumstances may be found in the ANSI/ASQC A1 document.

5.3.4 Comparison with standard reference materials (SRMs) is frequently the only strategy that can be employed for