Designation: E882 – 10 (Reapproved 2016)

Standard Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[1]

This standard is issued under the fixed designation E882; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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:*[2]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis

MNL 7A Manual on Presentation of Data and Control Chart Analysis[3]

2.2 *ASQC Document:*[4]

ASQC Standard A1 Definitions, Symbols, Formulas, and Tables for Control Charts

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology E135.

4. Significance and Use

4.1 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.

5. Accountability

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

5.2 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, 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.

5.3 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:

5.3.1 Each testing request 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 request. 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

[1] This guide is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.22 on Laboratory Quality.

Current edition approved Dec. 1, 2016. Published December 2016. Originally approved in 1982. Last previous edition approved in 2010 as E882 – 10. DOI: 10.1520/E0882-10R16.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] ASTM Manual Series, ASTM, 7th Edition, 2002.

[4] Available from American Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI 53203, http://www.asq.org.

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 other testing records because results from production control samples are usually reported on routine report forms, the samples being identified with the day, shift, run, or lot from which they were taken.

5.3.2 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."

5.3.3 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 requests.

5.3.4 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.

5.3.5 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 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.

6. Quality Control

6.1 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.

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

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

6.2.2 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.

6.2.3 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.

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

6.2.5 For each method (for each sample type), choose a control material that is known to be stable, 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 supply 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 material approach.

6.2.6 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 are again 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.

6.2.7 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.

6.3 *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:

6.3.1 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.

6.3.2 The $\bar{X}$-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 -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.

6.3.3 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 $\bar{X}$-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 ASQC Standard A1 document.

6.3.4 Comparison with certified reference materials (CRMs) is frequently the only strategy that can be employed for infrequently used analytical methods or for non-routine sample types. If a CRM (from the National Institute of Standards and Technology or other CRM producer) similar to the samples in composition is tested with the samples, comparison of the measured value to the assigned value of the CRM provides a measure of confidence in the sample assays. Lacking a CRM, any previously analyzed material may be used. In all cases, it is important to retain as large a portion of such a material as possible and to tabulate the results, the method used, the date, and the analyst. Materials and data thus obtained may have important future statistical or control chart use.

6.4 *Definitions:*

6.4.1 *mean:*

$$\bar{X} = (X_1 + X_2 + \ldots . X_n)/n \quad (1)$$

where:

n = the number of analytical values.

6.4.2 *grand mean:*

$$\bar{\bar{X}} = \left(\bar{X}_1 + \bar{X}_2 + \ldots \bar{X}_k\right)/k \quad (2)$$

where:

k = the number of individual means.

6.4.3 *range:*

$$R = X_h - X_l \quad (3)$$

where:

X_h = highest observed value, and
X_l = lowest observed value in the data.

6.4.4 *average range:*

$$\bar{R} = (R_1 + R_2 + \ldots R_k)/k \quad (4)$$

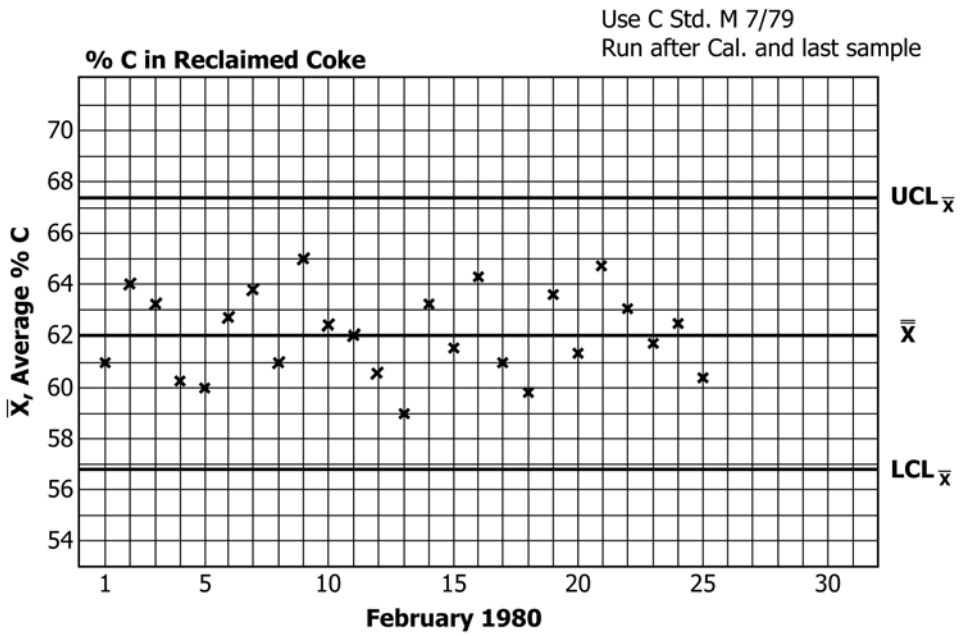

FIG. 1 Control Chart for Averages

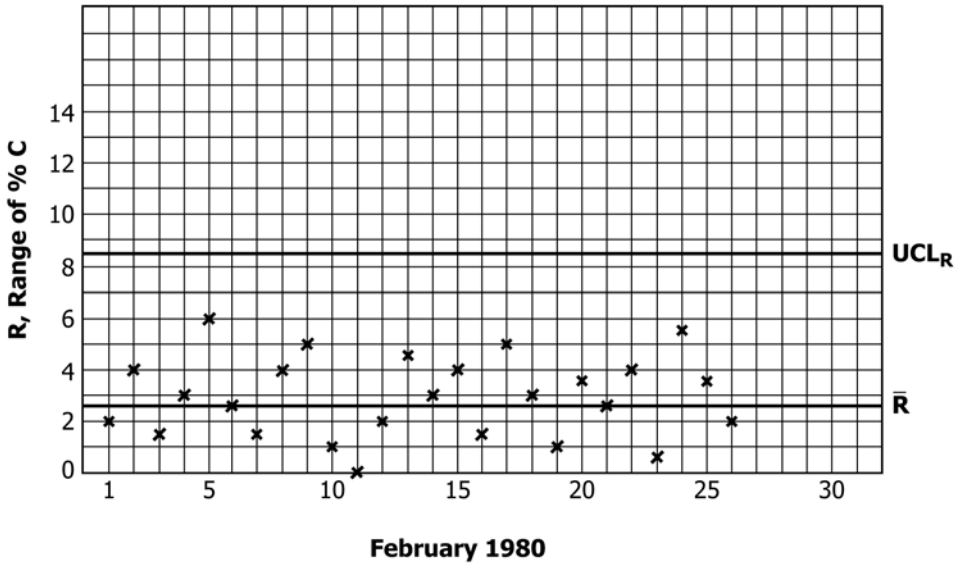

FIG. 2 Control Chart for Ranges

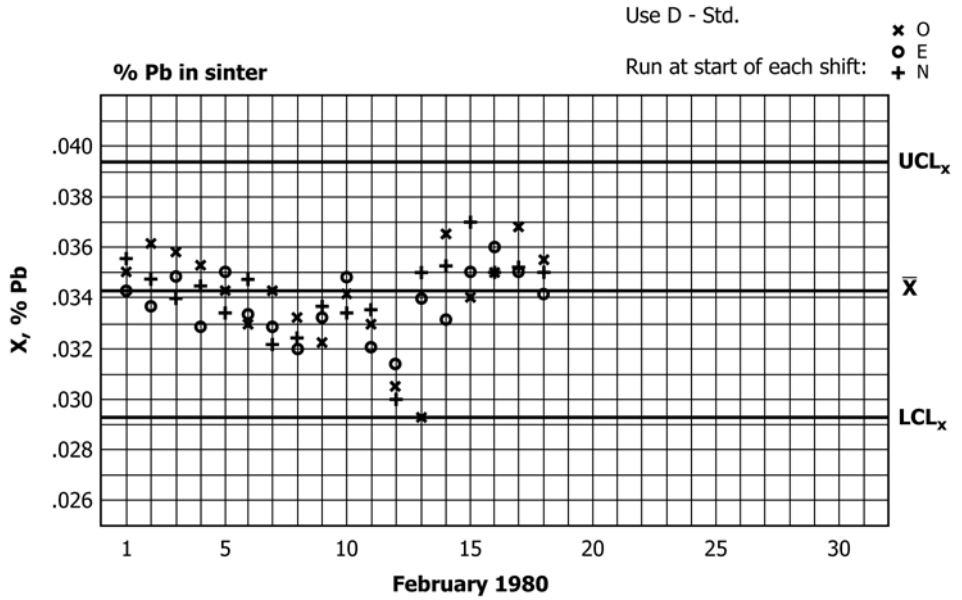

FIG. 3 Control Chart for Individuals

6.4.5 *estimated standard deviation:*

$$s = \sqrt{\frac{\sum (X_i - \bar{X})^2}{n - 1}} \quad (5)$$

where:

x_i = the individual values of successive observations,
$\bar{X}$ = the mean of the values, and
n = the number of values.

6.5 *Control Chart Construction*—Calculate the central value and control limits. Prepare the control chart with the vertical scale labeled so that the central value is approximately midway on the graph. Select a scale factor to permit the results to be plotted accurately and easily. The horizontal axis is labeled by shift, day, run number, or run date, as appropriate. Draw and label the central line and the lines representing the upper and lower control limits. The central line may be the grand average of the values obtained during the base period described below, or it may be a specifed value based upon experience. Title the chart with the method, the control sample identification, and any special instructions. Show all of the original data points that were used to calculate the control limits.

6.5.1 *$\bar{X}$- and R-Chart*—At least two independent measurements must be made on the control material during each run. The average of the n values obtained on each run is plotted on the $\bar{X}$-chart and the range is plotted on the R-chart. To calculate the positions of the lines on the charts, obtain at least 20 groups of n values (that is, 20 runs) during a period when the method is believed to be performing normally.

Formulas for $\bar{X}$ and R Control Chart Lines

Number of Measurements per Run[A]	Chart for Averages, $\bar{X}$	
	Central Line	Control Limits
2	$\bar{X}$	$\bar{X} \pm 1.880\ \bar{R}$
3	$\bar{X}$	$\bar{X} \pm 1.023\ \bar{R}$
4	$\bar{X}$	$\bar{X} \pm 0.729\ \bar{R}$
	Chart for Ranges, R	
	Central Line	Upper Control Limit
2	$\bar{R}$	$\bar{R} + 3.267\ \bar{R}$
3	$\bar{R}$	$\bar{R} + 2.574\ \bar{R}$
4	$\bar{R}$	$\bar{R} + 2.282\ \bar{R}$

[A] See MNL 7A if more than four measurements are made per run.

6.5.2 *X-Chart*—Each individual result is plotted on the *X*-chart. To calculate the positions of the lines on the chart, obtain at least 20 values for the control material when the method is believed to be performing normally. These measurements should be taken at the same intervals to be used for control purposes.

Formula for *X* Control Chart Lines

Central Line	Control Limits
$\bar{X}$	$\bar{X} \pm 2.66\ \bar{R}$

where $\bar{R}$(the moving range) is calculated as the average of the differences of each measurement from the preceding measurement, ignoring the sign. (Note that there will be one less such difference than the number of individual measurements.)

6.5.3 *Individuals Used as Grouped Data*—If the individual values used to produce the $\bar{X}$-chart can be grouped in some logical way such that, *n* results are obtained each day, each shift, and so forth, then $\bar{X}$- and *R*-charts may be constructed by treating these grouped data by the formulas of 6.5.1. Each result is then plotted as it is produced on the $\bar{X}$-chart for an immediate control decision. The average and range of the grouped data are plotted on the $\bar{X}$- and *R*-charts at the end of each period for detection of less abrupt drifts in the analytical procedure.

6.5.4 A complete discussion of control chart theory and practice (including examples of each procedure) is included in . See Practice E1329 for discussion of control charting applied to spectrochemical test methods.

7. Limitations of Control Chart Methods

7.1 In common with all statistical procedures, control chart methods depend upon certain basic assumptions. For the purposes of this guide, the most important of these assumptions is that the analytical method is capable of yielding consistent results in the hands of those who use it. If the performance of the method itself is erratic, or if the personnel who apply it do not exercise sufficient care, then no period of time will exist when the data collected can predict future behavior.

7.2 The limitation discussed above is not serious in the practical laboratory environment. The reason is obvious: If the method or the way it is employed is actually out of control (another way of saying erratic), that fact will become apparent because the calculated initial control limits will be unacceptably wide.

7.3 A second limitation of the control chart method is that it only indicates whether or not the analytical method is performing as expected. When it is not, the control chart gives no direct indication of the reasons for the poor performance. An analytical chemist must exercise his chemical skills and ingenuity to discover and correct the causes of the problem. The control chart will, however, provide analysts with quantitative indication of the effectiveness of their efforts.

8. Procedures for Improving Precisions or Accuracy of Analytical Results

8.1 Although complete coverage of the topic of improving the precision of analytical methods is beyond the scope of this guide, several aspects of statistical or supervisory control have the effect of improving precision or accuracy of analytical results without changes in the analytical methods.

8.1.1 *Replication*—It is a simple statistical fact that averaging a larger number of independent measurements improves the precision of the average reported values. In general, for readings randomly obtained from a normally distributed population of readings, the expected standard deviation of the average of *n* readings is $\frac{1}{\sqrt{n}}$ times the standard deviation of a single reading. A second important fact is that the first few replications improve the precision much more than would the same number of additional readings beyond, say 30. The improvement from averaging four observations is to reduce the standard deviation of the reported result by one half; nine replicates reduces it by two thirds, and so forth. The nature of chemical analyses is such that we must be careful when we seek to improve results in this manner. First, it is generally not difficult to justify making two or even four replicate observations for an important measurement. It is rarely justified to perform the same assay more than nine times. The second consideration is even more important. Truly independent replications are obtained only by replication of the *entire* analytical process. Replication of less than the entire analytical process (which includes sampling and sample preparation) usually produces much less reduction in the error of the final reported value than would be expected from the square root law. By way of illustration, the average of two separate samples taken from a rail car containing 100 tons of ore will probably be closer to the true content of that car than the average of two successive readings of an atomic absorption spectrometer while it is aspirating the solution obtained from one of those samples. Yet, because sampling variability is not included, the observed precision of the latter seems to be the better of the two.

8.1.2 *Improvements in Instruments and Personnel*—Proper maintenance of equipment with periodic checks on its performance against reference materials is necessary to minimize the contribution of instrumental errors. Proper training of personnel in good techniques and in understanding the significance of each step performed is necessary if a laboratory is to approach the accuracy that methods and equipment are capable of

delivering. The attitude of pride in good work and confidence in one's ability to perform well can be increased by involving all laboratory personnel in the planning and implementation of a quality control program. After all, the analyst will be the first one to see how his own performance looks on the control chart.

8.1.3 *Reference Materials and Calibration*—This last factor is included because it is crucial to accurate and meaningful laboratory results. A method or an instrument can be no more accurate than the reference materials with which it is calibrated. Not only must the reference materials be correct for the method and characterized with the accuracy demanded by the ultimate analysis to be performed, but the standardization or drift correction process must be performed in such a manner that this accuracy is transferred to the instrument or solution being calibrated. More care needs to be taken in the calibration process than for any other single measurement. In general, each calibration observation is subject to the same measurement error as each sample observation. Thus, it makes no sense to draw a calibration curve from single observations on reference materials and then to measure each sample four times. The random error for the calibration measurement would appear as a perceptible bias in all sample measurements. No general rule can be given for the proper emphasis to be placed on calibration. The guide to proper effort in this area is to combine common sense with an awareness that each calibration measurement is usually no more precise than any other measurement made with the same equipment.

9. Keywords

9.1 accountability; chemical analysis; control charts; quality control