7. MISCELLANEOUS SUBJECTS

7.1 NBS Support for the National Measurement System

Measurements are essential for almost every aspect of human existence. They are the keystone of our modern civilization. Measurements are always estimates of the true value of some property and have some degree of uncertainty which ordinarily needs to be known. Moreover, measurements made by various individuals and/or at various times need to be relatable and compatible. This requires that measurements must be made systematically in what may be called a national measurement system. The basic operational aspects of the national measurement system are depicted in Figure 7.1. The upper part of the figure represents physical measurements while the lower part relates to chemical measurements. Essentially one can visualize samples of materials that are measured to obtain data on some physical or chemical property that is needed for an end use. The measurement is made with respect to some physical or chemical standard. The basis for all measurements are the seven basic units shown in the table below. These are internationally accepted and NBS maintains national primary standards compatible with the international standards for fixing the magnitude of the units.

Table -- Units of Measurements

Basic

Length -	meter
Mass -	kilogram
Time -	second
Electric Current	ampere
Temperature -	kelvin
Amount of Substance	mole
Luminous Intensity	candela

Supplementary

Plane	Angle	-	radian
Solid	Angle	-	steradian

A hierarchy of standards can be envisioned, each relatable and hence traceable to the ones above it as shown in Figure 7.2. It is possible to use working standards for applied measurements with calibrations traceable to the national standards in virtually all areas of physical measurements. The traceability may be because of NBS calibrations or by measurements by others (a State weights and measures laboratory, for example) using higher hierarchy standards with certified values traceable to NBS. Obviously, the intercomparison measurements that are required need to be made with a high degree of reliability since the uncertainty of measurement must be added to the uncertainty of the calibrated value of the standards used.

The propagation of error in a measurement chain is illustrated in Figure 7.2. After several iterations, a lower hierarchy standard (LHS) may have an intolerable uncertainty for some uses due to the uncertainties associated with intervening calibration steps so that a user may need to have a particular working standard calibrated closer to the primary standard (see chain C in Figure 7.2). In such a case, a calibration laboratory higher in the hierarchy must provide the service (or use of a standard higher in the hierarchy is another possibility). Of course, NBS provides collibration services in some cases.

In consideration of the above, it should be noted that physical standards may need to meet rigid specifications as to their form, material, workmanship, and stability to qualify for use as upper hierarchy standards.

Physical measurements may be considered in two general classes. The first consists of those made to fix the values of measurement standards and can be called calibration and tolerance testing. The second is applied measurement and includes all but those in the first. Indeed, one might consider that the first is really a special class of the second kind. In both cases, measurement essentially is a comparison of an unknown with a standard and the uncertainty of the result includes that of the standard used together with the uncertainty of the intercomparison. Whether for calibration or for application, it behooves every metrologist and every measurement laboratory to minimize the uncertainty of its measurements. Improvements can be sought in two directions. Higher quality standards will reduce the systematic error (heavy lines of Figure 7.2) while improvements in precision (dotted lines of 7.2) can result from better quality control and/or increasing the number of replications. While replication can make random error uncertainties small, there is a practical upper limit to which this can be done. In high quality measurement, the two kinds of uncertainties may equal each other but the systematic should not exceed the random component. Reduction of systematic uncertainty to one-third of the random uncertainty is often practical in which case it does not contribute appreciable error to a measurement process.

NBS supports the measurement process as shown by Figure 7.3 by maintaining the basic primary standards and by calibrating or otherwise providing routes to them for traceability of working standards. NBS conducts fundamental research to increase understanding of measurement in its broadest interpretation. Along with others, NBS investigates and develops new and or improved methods of measurement and provides reference materials to evaluate the measurement process.

The responsibility for the reliability of any specific measurement is that of the metrologist/laboratory that reports it. A measured value without limits of uncertainty (error bars) is virtually useless since such limits are always needed in any application and are not implicit in the measurement process.

The measurement process should follow the procedure outlined in figure 7.3. The measurement laboratory uses existing methodology, appropriate calibrations and quality control techniques to attain statistical control of the measurement process. When acceptable precision is attained, the laboratory can evaluate its bias and set limits of uncertainty for the data. If either bias or precision are unacceptable, assignable causes should be sought and appropriate corrective actions should be taken.

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Any laboratory can and should evaluate its own precision of measurement and its ability to do so is a measure of its competence. Evaluation of bias can be extremely difficult and facilitated by externally provided calibrations, reference materials, definitive measurements, and other approaches. Obviously, biases smaller than the precision of measurement will be difficult to identify.

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A laboratory must be capable of evaluating its own precision and maintaining its measurement system in a state of statistical control. Otherwise, it must be considered as incapable of providing the services it offers. Furthermore, a laboratory should assign limits of uncertainty and maintain documented evidence for the basis of such assignment. In on-going measurement processes, control charts provide the basis for such assignment. In other cases, the redundant process of repetition is the only means to make such an assignment.

In addition to the above, NBS provides a limited amount of support for the measurement system in the form of education and training. Basic metrological information is contained in a number of papers. Statistical treatment of measurement data is discussed in a group of papers contained in NBS Special Publication 300 (17) and in NBS Handbook 91 (19). NBS presents a number of seminars in several areas of metrology. Information about them and the current schedule can be obtained from the NBS Office of Measurement Services (16). Specialized training for metrologists of State Weights and Measures laboratories is possible from the NBS Office of Weights and Measures.

TABLE 7.1

OPERATION OF A MEASUREMENT PROCESS WITH NBS SUPPORT

- 1. NBS provides
 - 1.1 Fundamental research to extend frontiers of methodology
 - 1.2 Basic research to develop new methodology
 - 1.3 Applied research to improve existing methodology
 - 1.4 Reference materials
 - 1.5 Calibration services
- 1.6 Other approaches for evaluating bias, e.g., definitive methods
- 2. Measurement laboratory
 - 2.1 Utilizes existing methodology
 - 2.2 Applies appropriate quality control techniques
 - 2.3 Demonstrates statistical control of the measurement process
 - 2.4 Evaluates precision
 - 2.4.1 Precision acceptable
 - 2.4.1.1 Evaluates bias using reference materials, calibration services, other approaches
 - 2.4.1.1.1 Limits of bias acceptable
 - 2.4.1.1.1.1 Reports measured value with assigned limits of accuracy
 - 2.4.1.1.2 Limits of bias unacceptable
 - 2.4.1.1.2.1 Assignable cause identified to improve methodology or quality control. Material remeasured
 - 2.4.1.1.2.2 Assignable cause not identified
 - 2.4.1.1.2.2.1 Seek other methods of measurement, or measurement not possible and major research required
 - 2.4.1.1.2.2.2 Report biased measured value with precision, only, reported
 - 2.4.2 Precision unacceptable
 - 2.4.2.1 Assignable cause(s) identified to improve methodology and/or quality control; remeasure
 - 2.4.2.2 Assignable cause(s) not identified; measurement not possible; seek other methodology or major research required

Notes:

1. Measurement laboratories have sole responsibility for evaluating their precision and can do so with little or no NBS support

NBS can provide education/training/workshops to assist laboratories to achieve quality control/statistical control

NBS can develop GLP's, GMP's, SOP's in critical areas of national concern (including industrial productivity)

- 2. Measurement laboratories can utilize 1.4, 1.5, and 1.6, with little or no NBS assistance, to evaluate bias
- 3. NBS has responsibility to establish and maintain the National Measurement System which involves 1.1, 1.2, 1.4, 1.5, and 1.6
- 4. NBS engages in state-of-the-art R & D in 1.2 and 1.3 to develop 1.4, 1.5, and 1.6. Much of this is transferable to measurement laboratories.
- 5. NBS cannot attest for the precision or accuracy of measurement values reported by others than itself.
- 6. NBS can make judgments whether a measurement process is or is not potentially capable of reliable measurements, but the demonstration of such capability is the responsibility of the measurement laboratory and must be supported by an adequate quality assurance program.
- 7. The development and maintenance of an adequate quality assurance program is the sole responsibility of the measurement laboratory. This is a prerequisite for offering and providing measurement services. NBS can assist by conducting research in quality assurance techniques, development of reliable methodology and conducting seminars or workshops, all intended to make measurement assurance self-sustaining.
- 8. If a measurement assurance program requires extensive NBS direct involvement, it needs to be redesigned to eliminate such involvement and or to remove the reason why such involvement is necessary.



FIGURE 7.1 NATIONAL MEASUREMENT SYSTEM

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B. Propagation of Uncertainty C. Alternate Calibration of Chain Uncertainty

FIGURE 7.2 PROPAGATION OF CALIBRATION UNCERTAINTY



FIGURE 7.3 NBS SUPPORT FOR THE MEASUREMENT PROCESS

7.2 Tolerance

Tolerances for weighing and measuring devices are established on the following basis: tolerance values are so fixed that the permissible errors are sufficiently small that there is no serious injury to either the buyer or seller of commodities, yet not so small as to make manufacturing or maintenance cost of equipment disproportionately high. With respect to the standards used to test weighing and measuring devices, the error in the standard should be less than 25 percent of the smallest tolerance to be applied to the device when the standard is used, otherwise, a correction factor must be applied to the standard so used.

Whenever a customer's standard is tested in the laboratory and returned to the user, the standard should be expected to remain within tolerance until the time it is scheduled to be recertified. Whenever a standard is out of tolerance or near the tolerance limit such that it is likely to go out of tolerance before it is expected to be retested, the standard should be adjusted as closely as practical to zero error. The rate with which the standard changes its value depends upon the material of which it is made, the frequency of use, the care it receives, and the environment to which it is subjected. Any standard that is damaged or is subject to an incident that may have changed its value significantly, resulting in a question of its validity, should be removed from use until it can be tested in the laboratory.

The tolerance applicable to the device for which the standard is to be used is not the only consideration for establishing the tolerance for the standard. An additional factor is that the tolerance on the standard should not be an excessively large fraction of the value of the standard. The standard should be a reasonably accurate representation of its nominal value. A determination of what is "reasonably accurate" depends upon the type of standard, its nominal value, its use, and the accuracy required in the measurement process in which the measuring device (under test) will be used.

Tolerances for Commercial Equipment

Acceptance and Maintenance Tolerances - The official tolerances prescribed by a weights and measures jurisdiction for commercial equipment are the limits of inaccuracy officially permissible within that jurisdiction. It is recognized that an errorless value or performance of mechanical equipment is unattainable. Tolerances are established, therefore, to fix the range of inaccuracy within which equipment will be officially approved for commercial use. In the case of classes of equipment on which the magnitude of the errors of value or performance may be expected to change as a result of use, two sets of tolerances are acceptance tolerances and maintenance tolerances. Acceptance established: tolerances are applied to new or newly reconditioned or adjusted equipment, and are smaller than (usually one-half of) the maintenance tolerances. Maintenance tolerances thus provide an additional range of inaccuracy within which equipment will be approved on subsequent tests, permitting a limited amount of deterioration before the equipment will be officially rejected for inaccuracy and before reconditioning or adjustment will be required. In effect, there is assured a reasonable period of use for equipment after it is placed in service before reconditioning will be officially required. The foregoing comments do not apply, of course, when only a single set of tolerance values is established, as is the case with equipment such as glass milk bottles and

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graduates, which maintain their original accuracy regardless of use, and single service measure-containers, which are used only once.

<u>Tolerances and Adjustments</u> - Tolerances are primarily accuracy criteria for use by the regulatory official. However, when equipment is being adjusted for accuracy, either initially or following repair or official rejection, the effect should be to adjust as closely as practicable to zero error. Equipment owners should not take advantage of tolerances by deliberately adjusting their equipment to have a value or to give performance at or close to the tolerance limit. Nor should the repairman or serviceman bring equipment merely within tolerance range when it is possible to adjust closer to zero error.

<u>Tolerances for Standards</u> - A general principle that has long been recognized by the National Bureau of Standards is that the error in a standard used by a weights and measures official should be known and corrected for when the standard is used; or if the standard is to be used without correction, its error should not be greater than 1/3 of the smallest tolerance to be applied when the standard is used. The reason for this is to keep at a minimum the proportion of the tolerance on the item tested that will be used up by the error of the standard. Expressed differently, the reason is to give the item being tested as nearly as practicable the full benefit of its own tolerance.

Field testing operations are complicated to some degree when corrections to standards are applied. Except for work of relatively high precision, it is recommended that the accuracy of standards used in testing commercial weighing and measuring equipment be so established and maintained that the use of corrections is not necessary. Also, whenever it can readily be done, it will be desirable to reduce the error on a standard below the 1/3 of the smaller tolerance previously mentioned.

The numerical values of the tolerances recommended by the National Bureau of Standards for the standards of length, mass, and capacity used by weights and measures officials may be obtained upon request from the Office of Weights and Measures of the National Bureau of Standards.

<u>When Corrections Should Be Made</u> - When testing a measuring device the weights and measures official has expressly only one official duty, and that is merely to determine whether equipment is or is not suitable for commercial use. If a device conforms to all of the official requirements, the official seals it to indicate approval. If it does not conform to all official requirements, he is required only to reject it and prohibit its use until the device is brought into proper conformance.

Some officials contend that it is justifiable for the official to make minor corrections and adjustments in order to correct faulty equipment if there is no service agency nearby or if the owner or operator depends on this single device and would be "out of business" during the repair of the device.

Adjustments should be made, with the permission of the owner or his representative, only when the official is thoroughly competent to make such an adjustment and when he is certain that the real cause of the inaccuracy will be corrected thereby and is not due to faulty installation or a defective part. He should never undertake major repairs, or even minor corrections if the services of commercial agencies are readily available. <u>Gauging</u> - In the majority of cases, when the weights and measures official tests commercial equipment, he is verifying the accuracy of a value or the accuracy of the performance as previously established either by himself or by someone else. There are times, however, when the test of the official is the initial test on the basis of which the calibration of the device is first determined or its performance first established. The most common example of such gauging is in connection with vehicle tanks, the compartments of which are used as measures. Frequently the official makes the first determination on the capacities of the compartments of a vehicle tank, and his test results are used to determine the proper settings of the compartment indicators for the exact compartment capacities desired. Adjustments of the position of an indicator under these circumstances are clearly not the kind of adjustments discussed in the preceding paragraph.

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Inspection versus Testing - A distinction may be made between the inspection and the testing of commercial equipment that should be useful in differentiating between the two principal groups of official requirements -- specifications and performance requirements. Although frequently the term inspection is loosely used to include everything that the official has to do in connection with commercial equipment, it is useful to limit the scope of that term primarily to examinations made to determine compliance with design, maintenance, and use requirements. The term testing may then be limited to those operations carried out to determine the accuracy of value or performance of the equipment under examination by comparison with the actual physical standards of the official.

Accuracy of Standards - The accuracy of testing apparatus should invariably be verified prior to the official use of the apparatus. Standards should be reverified as often as circumstances require. By their nature, metal volumetric standards are more susceptible to damage in handling than are standards of some other types. Whenever damage to a standard is known or suspected to have occurred, and whenever repairs that might affect the accuracy of a standard have been made, the standard should be recalibrated. Routine recalibration of standards, particularly volumetric standards, even when a change of value is not anticipated, should be made with sufficient frequency to affirm their continued accuracy, so that the official may never be in an indefensible position with respect to the accuracy of his testing apparatus. If use is made of secondary standards, such as special fabric testing tapes, these should be verified much more frequently than such basic standards as steel tapes or volumetric provers to demonstrate their constancy of value or performance.

Accurate and dependable results cannot be obtained with faulty or inadequate standards. If either serviceman or official is poorly equipped, it cannot be expected that their results will check consistently. Disagreements between servicemen and officials can be avoided, and the servicing of commercial equipment can be indefensible improved if servicemen and officials will give equal attention to the adequacy and maintenance of their testing apparatus.

7.3 Distinction Between Mass and Apparent Mass

The mass of an object can be simply defined as the quantity of matter that comprises the object. The mass of an object remains constant regardless of its location. Thus, the mass does not vary as the object is moved from one part of the country to another although the forces acting on the mass may change. In mass measurement, the term "true mass" is frequently used to mean the mass of an object. The adjective "true" is redundant and will not be used in the remainder of this paper.

The purpose of mass measurement is to determine the mass of an unknown object; i.e., the unknown object is calibrated by comparing its mass to that of a known mass standard. A mass calibration is performed in air as are virtually all mass measurements. Thus, when two objects are compared, each object is being subjected to a lifting force equal to the mass of air displaced by the object times the force of gravity in addition to the downward force on each object resulting from the earth's gravity. The mass of air displaced by an object depends on the density of the air and the volume of the object. Since all mass measurements are made in air and mass calibrations are performed by comparing an unknown standard to a known standard, the mass of a standard is frequently reported as the apparent mass of the standard. The apparent mass of an object is the mass of a (hypothetical) reference standard of a specified density that will produce a balance reading equal to that produced by the object if the measurements are made at 20 °C in air with a density of 1.2 mg/cm³. Whenever the term "apparent mass" is used, it is necessary to specify the density of the (normally hypothetical) reference standard against which the unknown standard is being compared. This statement of the density of the reference standard, called the reference density, is necessary because the apparent mass value depends in part upon the volume of the hypothetical reference standard. The reference density of 8.0 g/cm³ is normally used to report the apparent mass of a standard or object. This is called the apparent mass versus 8.0 g/cm³. In the past, the apparent mass was reported against the density of brass at 20 °C. This density is 8.3909 g/cm³. This apparent mass value is referred to as the apparent mass versus brass.

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The definition of apparent mass versus a reference density specifies the conditions under which the apparent mass of a standard or object is to be determined. To compute apparent mass, it is necessary to know:

- 1. the density of the hypothetical reference standard;
- 2. the mass of the hypothetical reference standard which is the mass of a reference standard that will give the same balance reading as the unknown standard under specified conditions;
- 3. the temperature (20 °C) at which the "comparison" of the masses is made;
- 4. the density of the air (1.2 mg/cm^3) in which the "comparison" is made; and
- 5. the density of the standard being calibrated.

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The density of the object being calibrated must be known since its volume is involved in the apparent mass determination. It is the difference in the volumes between the object being calibrated and the hypothetical reference standard that determines the apparent mass of an object versus a specified reference density. The apparent mass versus 8.0 g/cm³ of an object can be defined as the mass the object would appear to have if it was compared against a standard which has density of 8.0 g/cm³ and a mass giving the same balance reading as the object when the comparison is made at 20 °C in air having a density of 1.2 mg/cm³.

The definition for apparent mass versus brass is exactly the same except that the density of brass at 20 °C (8.3909 g/cm³) is substituted for 8.0 g/cm³. An example will be given to illustrate the apparent mass concept.

Suppose that we have three weights made of aluminum (A1), brass, and stainless steel (SS). Assume that the densities of these weights are 2.7 g/cm³, 8.3909 g/cm³, and 8.0 g/cm³; respectively, and that all three weights have a mass of exactly 1 kg. The weights then have volumes of 370.37 cm^3 , 119.18 cm³ and 125.00 cm³, respectively. When the weights are in air, there will be a lifting force due to the effect of the displaced air and a downward force due to the mass (m) of the weight.



It can be seen that the larger the volume, the greater the upward force. Since gravity appears in all terms when the weights are intercompared, the gravity factor (g) cancels.

Suppose now that a perfect equal-arm balance exists and is used to compare the weights in both a vacuum and in air. Suppose that the aluminum weight is placed on one pan of the balance and the stainless steel weight is placed on the other pan and the balance and weights are placed in a vacuum. Because there is no air present, there is no lifting force so the weights will appear to have equal masses since only the downward force is acting on the weights. If the aluminum weight was compared in a vacuum against the brass weight, the two weights would also appear to have equal masses. The same would be true if the brass weight was compared to the stainless steel weight. Thus, the mass of an object can be visualized as the mass an object would appear to have when it is compared in a vacuum against a known standard.

Now suppose that the aluminum and stainless steel weights are compared on the balance in air that has a density of 1.2 mg/cm^3 . The air will exert a lifting force on each weight as described earlier. This can be illustrated in mass units (since gravity cancels out) as



The aluminum weight experiences a much larger lifting effect than the stainless steel weight, namely, 444.4 mg versus 150 mg. Thus, the aluminum weight appears to be 294.4 mg lighter than the stainless steel weight. This is simply the air density times the difference in weight volumes. Hence, the apparent mass of the aluminum weight versus 8.0 g/cm³ is approximately

1 kg - 294.4 mg - 999.7056 g

If the aluminum weight was compared in air against the brass weight, the situation is illustrated as



This means the aluminum weight appears to be 301.4 mg lighter than the brass weight. Hence the apparent mass of the aluminum weight versus brass is approximately

$$1 \text{ kg} - 301.4 \text{ mg} = 999.6986 \text{ g}$$

To carry the example further, suppose the brass weight and the stainless steel weight were compared in air with a density of 1.2 mg/cm^3 . The effect can be illustrated as



Using the stainless steel weight as the reference standard, the brass weight appears to be 7 mg heavier than the stainless steel weight. This is due to the difference in the lifing effect of air on the weights. The lifting effect is less on the brass weight because its volume is less than that of the stainless steel weight. This condition can also be reported using the brass weight as the reference standard. In this case, the stainless steel weight appears to be 7 mg lighter than the brass weight. Hence, we can say that the apparent mass of the brass weight versus 8.0 g/cm³ is 1 kg + 7 mg or 1000.007 Similarly, the apparent mass of the stainless steel weight versus brass is g٠ 1 kg - 7 mg or 999.993 g. Thus, the apparent mass of a weight depends upon the density chosen for the reference density. This is particularly clear for the results for the aluminum weight. Thus, the apparent mass versus a reference density is the mass an object would appear to have if it was compared against a standard of a specified density and having a mass that would give the same reading as the object when the comparison is made at 20 °C in air with a density of 1.2 mg/cm^3 .

The equations recommended for use to compute the apparent mass of an object are given in SOP No. 2.

7.4 Control Charts

Control charts provide a graphical means to decide whether a measurement system has attained and is maintaining a state of statistical comprol. They can indicate drifts warn of the initiation of potential problems and reveal the need for corrective actions.

Control charts were first proposed in 1934 by Walter Shewhart for use in statistical process control. Since that time, various modifications of his original format have been proposed and used. Cameron and co-workers at NBS [7] were among the first to use control charts for monitoring measurement processes, using essentially the Shewhart format. A general discussion of control charts and extensive tables useful for calculating control limits will be found in ASTM Manual for Presentation of Data and control Chart Analysis [3]. Croarkin discusses control charts as they are used in measurement assurance programs [11]. The following discussion reviews the kinds of control charts considered to be most useful for laboratory use.

Several kinds of control charts will be found to be useful. The simplest is based on the repetitive measurement of a stable test object and either the results of single measurements (X chart) or the means of several (\overline{X} chart) are plotted with respect to sequence or time of measurement. The results should be randomly distributed about the mean (\overline{X}) in the case of an X chart and about the mean of means (\overline{X}) in the case of an \overline{X} chart when the measurement system is in a state of statistical control. Furthermore, the results should lie within defined limits, based on statistical considerations.

There is nothing really wrong in maintaining a simple X chart. However, an \overline{X} chart is preferable to an X chart because average values will indicate a change in performance more conclusively than individual values. This advantage must be evaluated against the increased effort required to maintain the former. An \overline{X} chart based on the average of two measurements is a good compromise when possible.

In addition to the above, there are precision control charts in which either the standard deviation, estimated at various times, or the range, R, of a set of measurements is plotted and interpreted similarly. Because of the economy of effort, an R chart is preferable to an s chart. When a propertyvalue control chart (X chart or the chart) and a precision chart are maintained in parallel, diagnosis of out-of control situations as due to imprecision or bias and the identification of assignable causes for such are facilitated.

X Control Chart

Single measurements are made of a stable test object, at least once on each test day or at least monthly (if a measurement system is to be maintained in statistical control over a period of time). The results are plotted sequentially and the process is considered to be in control when they are randomly distributed within limits as defined below.

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Central Line, \overline{X}

Measure the test object on at least 12 occasions (recommended) but no more frequently than daily, i.e. never twice on the same day. The initial central line is the mean of the n measurements, X_i for i=1,...,n.

Central line,
$$\overline{X} = \frac{\Sigma X_i}{n}$$

Calculate s_x the estimate of the standard deviation of X in the usual manner. Note that this is an estimate of the long-term standard deviation. Calculate the upper and lower control and warning limits as:

 $UCL = \overline{X} + 3s_{X}$ $UWL = \overline{X} + 2s_{X}$ $LWL = \overline{X} - 2s_{X}$ $LCL = \overline{X} - 3s_{X}$

When so set, approximately 95% of the plotted points should fall between the warning limits (LWL and UWL) and rarely should any fall outside of the control limits (LCL and UCL) if the system is in a state of statistical control. The control limits are conservative.

X Control Chart

A stable test object is measured in replicate, periodically. It is recommended that these should be duplicate measurements made at least once on each test-day or at least monthly, whichever is the more frequent. The means of the measurements, \overline{X} , are plotted sequentially. Statistical control is judged when the plotted points are randomly distributed within the control limits, determined as outlined below.

Initial Control Limits, \overline{X} Chart

Central Line X

Measure the test object, in duplicate on at least 12 occasions (recommended) and no more frequently than daily, i.e. never twice on the same day. The initial central line is the mean of the means of n duplicate measurements, \overline{X}_i for i=1,...,n.

central line,
$$\bar{X} = \frac{\Sigma X_i}{n}$$

Calculate $s_{\overline{x}}$, the estimate of the standard deviation of \overline{X} in the usual manner.

Note that this is a long-term standard deviation of the mean of n measurements and will ordinarily be larger than the short-term (within day) standard deviation which may be calculated from the value of \overline{R} (see later).

However, if the long-term exceeds the short-term standard deviation by more than a factor of 2, the quality control should be improved to decrease the former to more acceptable values.

Control Limits

 $UCL = \overline{X} + 3s_{\overline{X}}$ $UWL = \overline{X} + 2s_{\overline{X}}$ $LWL = \overline{X} - 2s_{\overline{X}}$ $UCL = \overline{X} - 3s_{\overline{X}}$

The limits are set so that approximately 95% of the points should fall within the warning limits (LWL and UWL) and rarely should any fall outside of the control limits (LCL and UCL) if the system is in a state of statistical control. The limits are conservative.

R Control Chart

The absolute differences (R) of duplicate measurements of the test object, and also of similar test specimens may be plotted sequentially to evaluate the precision of the measurement process. This constitutes an R (range) control chart. Note that the range is related to the short-term standard deviation, i.e. the repeatability of measurements over a relatively short period of time.

Initial Control Limits, R Chart

Central Line \overline{R}

The observed ranges R_i (absolute values) for k sets 'at least 12 is recommended) of duplicate measurements are averaged to obtain a value for \overline{R} .

$$\overline{R} = \frac{\Sigma R_1}{k}$$

The control limits for duplicate measurements are as follows:

The control limits have the same significance as in the case of an X or \overline{X} chart.

For triplicate measurements, the control limits are:

Updating Control Charts

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After additional control data are accumulated (at least as much as originally used) the control limits may be updated. At test is made to see whether the second set of data for \overline{X} or \overline{X} is significantly different from the first set (see Chapter 8.11). If not, all data may be combined to obtain a new and more robust estimate of \overline{X} or \overline{X} . If the second set is significantly different from the first, only the latter should be used in revising the control chart.

The value for the standard deviation s for the second set of determinations should likewise be compared with the first estimate using the F test (see Chapter 8.9) to decide whether to pool it with the first (see Chapter 7.4) or to use it separately in setting new control limits. A smaller value for s, may result from improvement of the precision as the result of a learning experience, for example. A larger value for s could be due to an original poor estimate of the standard deviation of the measurement process, or to a decrease of precision resulting from an assignable cause(s). In either case, the reason should be ascertained.

If the values of R show no systematic trends, and if \overline{R} has not changed significantly, all of the values of R may be combined to obtain an updated estimate of \overline{R} , from which updated control limits can be computed. Judgment of the significance of apparent changes in \overline{R} can be made by computing the corresponding values of s (see Table 9.1) and conducting an F test (see Chapter 8.9).

Interpretation of Control Chart Data

Plotted points should be randomly distributed within the warning limits when the system is in a state of statistical control. If a plotted point lies outside of the warning limits, a second set of measurements should be made. If this point lies outside of the warning limits, corrective action is required and demonstrated attainment of control is necessary before measurements may be reported with confidence. Barring blunders, one point outside of the control limits is reason for corrective action. The nature of the corrective action to be taken, in either case, will depend on the kind of measurement made. If the X or \overline{X} point is outside the limits but the R point is not, a source of bias should be sought and eliminated. If the R point is outside of limits, X or \overline{X} will probably but not necessarily be outside, as well (note that compensating fluctuations could cancel one another). Sources of extraordinary random error should be sought and eliminated, before any possible bias can be detected.

Control charts may be used to evaluate the uncertainty of measurement in some cases. When an appropriate control chart is maintained, an X or \overline{X} chart may be used to evaluate bias and to document the standard deviation of the measurement process. Then the values for s on which the control limits are based may be used in calculations of confidence limits for measurement values (see Chapter 8.6).

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