

GUIDELINES FOR LABORATORY QUALITY CONTROLS AND QUALITY ASSESSMENT OF SOIL ANALYTICAL DATA

ERWDA Soils Bulletin No. 1

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Guidelines for Laboratory Quality Controls and Quality Assessment of Soil Analytical Data

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FOREWORD

Laboratory soil analysis is the first step for evaluating soil conditions and to judge whether the soil has potential for a specified use or can be improved and in what manner; in this way we can keep the land productive and sustainable.

Soil analysis data is always required to supplement field description of soil survey from regional, national to farm level. Soil survey is the basic information required by natural resource planners, users, managers and serves an educational tool as well. Soil survey is not merely drawing lines on a map, it is the soil resource inventory and database, and is the basis for all other decisions that will be made. If this database is of poor quality, everything else that follows will be unreliable and agricultural and environmental research and development will suffer.

It is necessary to run laboratory quality control while analyzing the soil samples, and also run quality assurance tests on analytical soil data for accuracy and reliability. This publication presents the guidelines for quality assessment of soil data obtained through laboratory analyses of soil samples, leading to academic degree programs and other research activities. The publication demonstrates an easy format and language that is designed to be user-friendly. It is hoped that soil scientists, agronomists and soil survey staff as well as laboratory personnel will use the useful information. This publication is a product of the commitment manifested in the vision of future soils research in the Emirate of Abu Dhabi and as an educational tool, and should be used as guidelines only.

Environmental Research and Wildlife Development Agency
Abu Dhabi Emirates

PREFACE

The quality of soil analytical data produced during any soil project is the keystone to its success. This is an area difficult to establish, but certainly if the analyses are made with high degree of integrity i.e., testing the soils utilizing the recommended methods, and if it incorporates quality assurance program, quality can be achieved. To achieve this important goal it is important to use the techniques suited to the type of soils being analyzed, or use recommendations that are suited the conditions of the country. This publication presents guidelines for laboratory controls, and the application of standard rules of thumb, which allow to assess the quality of a large number of soil samples for saturated paste pH, electrical conductivity of the saturation extract, anions and cations in the saturation extract, gypsum, CaCO_3 , exchangeable sodium percentage, sodium adsorption ratio, cation exchange capacity, water retention at 15 bar, available water, saturation percentage, clay content and Atterberg limits etc. It is visualized that quality assurance should commence with planning of the sampling and will continue through sampling, preparation, sub-sampling, analysis, and interpretation of the data and report writing. Some simple statistical approaches to check the quality of the data are also presented.

ABOUT THE AUTHORS

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INTRODUCTION

Soil is a structured natural environment that provides the source and nurturing ground for all terrestrial life. It has therefore it has a fundamental role in the terrestrial ecosystem, as a three dimensional body performing a wide range of ecological functions. Soil and environmental related projects are always supported by quantitative analyses of specific soil qualities. Lack of soil related information usually leads to failure many projects particularly the irrigation, agricultural and environmental management, because the soils were not studied systematically prior to implementing the projects, and the responses of soil to human influences were merely ignored.

It is essential to understand the *raison d'être* for soil analysis. Once the reasons for analyses are established and the data are produced, the next logical step is to run quality assurance tests for validity and accuracy, for interpretation purposes.

Soil data are required for soil survey and classification (Kimble, 1995; USDA-NRCS, 1995a,b,c; USDA-NRCS, 1996a,b; USDA-NRCS, 1998; Shahid and Omar, 1999), fertilizer recommendations (Hardter, 1996), soil conservation (Sajjapongse, 1996), environmental monitoring (Kimmo, 1996) and assessment of sustainability (Syers, 1996). The type of analysis and the frequency of sampling (USDA-SCS, 1988) differ with the intended use of the data; for example for soil characterization, sampling and analysis is required only once, whereas for assessment of sustainability, repeated sampling and analysis are often needed.

Sustained environmental management and agricultural production is now a major target of many agronomic or soils management research development projects. Such sustainability is possible only if the soils of the selected sites are well characterized. A reliable method to analyze plant, soil and water for agricultural purposes is now considered necessary for sustainability and increasing agricultural production, as is the case with soil survey and environmental management projects. The surveys are generally conducted on different scales to classify and describe the soils for a number of uses. To increase agricultural production, and rational basis for broad land use planning are the important soil survey objectives in a number of arid and semi-arid regions of the world.

The purpose of a soil survey is to determine types of soils in an area, how variable they are and what are their physical and chemical characteristics. Soil surveys are always completed with a field description of soils, supplemented by quantitative and qualitative analyses of specific

soil characteristics. Once the soil information is generated, quality assessment is important for proper utilization of the outcome of the projects. Quality assurance scientists review the results for consistency and technical accuracy.

The quality of the results is, again, the keystone to success. This is an area that is very difficult to establish, but if the analyses are made with a high degree of integrity, i.e., testing the soils utilizing the recommended methods, and they adhere to quality assurance program, the required quality can be achieved. To achieve this important goal, it is important to use the techniques that are suited to the types of soils being analyzed, or use recommendations that suit the conditions of the country or area; otherwise, this will affect badly the quality of the results and cause the eventual weakening of the project output. Realizing the importance of the quality assessment of soil data, the present publication is produced to help scientists to have confidence in the results for their satisfactory interpretation.

Standard Terms Used in a Quality Assessment Program

The quality assessment of soil data is a process of providing directions, inspection, and coordination of activities to ensure that the final document meets the defined standards for content, accuracy and precision. To ensure quality, the soil data are always tested against available standards and their differences are reported. It is always relevant to introduce some of the terms that are related to quality assurance, these are as follows:

Accuracy - The degree of agreement of an experimental or measured value with the true or expected value of the quantity of concern. It is expressed in terms of ratio or percent. Accuracy is often expressed in terms of relative percent error i.e.

$$\text{Relative \% error} = (\text{Experimental value} - \text{True value}) \times 100$$

Analyte - Specific component measured in a chemical analysis.

Error - Discrepancies between result of measurement and the true value.

Absolute Error - The difference between the experimental or measured value of a quantity X_o and its actual (absolute) value X , given by $\Delta X = X_o - X$.

Random Error - An error which varies in an unpredictable manner, in magnitude and sign, when a number of measurements were conducted under effectively identical conditions.

Systematic Error - An error which (in the measurements of a given quantity) remains constant or varies according to a definite law when conditions are changed.

Bias - It is a measure of systemic error. It has two components, one due to method and the other to a laboratory use of the method. It can be best measured by laboratory inter comparison.

Precision - Extent to which reading/result repeat. The two most important aspects of precision are reliability and validity. Reliability refers to the reproducibility of a measurement. One can quantify reliability simply by taking several measurements on the same subjects. Poor reliability degrades the precision of a single measurement and reduces the ability to track changes in measurements in experimental studies. Validity refers to the agreement between the value of a measurement and its true value. To quantify validity compare the measurements with true values (Certified values). Poor validity also degrades the precision of a single measurement and it reduces the ability to characterize relationships between variables in descriptive studies.

Quality - An estimate of acceptability or suitability, for a given purpose, of an object, item, or tangible entity. It may also be defined as the degree of excellence relative to the actual or the degree to which a measurement made meets a prescribed specification.

Quality Assurance - A system of activities whose purpose is to provide the producer and /or the user of a product or service the assurance that defined standards of quality have been met. Quality assurance consists of quality control and quality assessment, i.e., two separate but related activities.

Quality Assessment - An overall system of activities whose purpose is to provide assurance that the quality control activities are carried out effectively. Quality assessment involves a continuing performance evaluation of the production system and the product quality.

Quality Control - An overall system of activities whose purpose is to control the quality of a product or service so that it meets the user's needs, i.e., to provide quality that is satisfactory, adequate, dependable and economic.

Range - With a finite number of values, the simplest measure of variability is the range, which is the difference between the largest and smallest values.

Repeatability - Values below which absolute difference between two results obtained with the same test method on identical test material under the same conditions may be expected to lie with a certain specified probability (95% in absence of other indications).

Reproducibility - Values below which absolute difference between two results obtained with the same test method on identical test material under different (Operator, apparatus location conditions, time) conditions may be expected to lie with a specified probability; in the absence of other indications, the probability is 95%.

Significant Figure - Numerical values in standard methods (standard operating procedures) and reports.

Standard Method (standard operating procedures) - A procedure that has been developed by standard-writing organizations, based on consensus, opinion or other criteria and often evaluated for reliability by a collaborative testing procedure.

Traceability - Property of the result of a measurement whereby it can be related to appropriate measurement standards, generally international or national standards, through an unbroken chain of comparisons.

Uncertainty - Range within which true value probably lies. The uncertainty measurement is a parameter associated with the results of measurements. It characterizes the dispersion of the values that could reasonably attribute to the measured values.

Quality of Analytical Procedures within a Laboratory

The utilization of standard analytical procedures is a keystone for the success of the soil data. The procedure must also be satisfactory in the following aspects: sensitivity of the method (measurable standard deviation), precision (reproducibility and repeatability), accuracy, safety (to the analyst, and residues discarded) and cost (in terms of money for chemicals, manpower and equipment). The precision and accuracy are two important criteria. The accuracy of the analytical method in a laboratory is determined by the use of an internal reference sample (Standard or certified reference materials). This reference sample should be included with each batch of analyses (about 20 to 25 samples) or a minimum of one analysis of the reference should be conducted daily. A quality control chart may also be maintained in the laboratory. This can be prepared by plotting the value of the internal reference: y-axis is the variable value and the x-axis

the successive analysis of the reference sample. If the values of the reference sample during any analysis exceed the predetermined value by more than ± 3 standard deviation (SD) limits or if in two successive analyses the values of the reference sample exceed ± 2 SD limits, the analytical procedures need to be examined and the analysis repeated.

At least three to four replicates of each sample should be analyzed to obtain a measure of the coefficient of variation ($CV = SD/\text{mean}$).

Evaluation and treatment of analytical data

All measurements in quantitative analyses are subject to error. To minimize errors; they must first be identified then corrective action applied. Factors like time, expense and technique applied are responsible for the deviation of measured values from true value. As these limitations are minimized/removed, the measured values become closer and closer to the true value; however, the true values are never obtained with a high degree of confidence. For this reason, a measurement is normally made in replicate or triplicate, and the results treated statistically to provide an estimate of the error of the method. During evaluation of analytical data, the analyst must first determine the extent and direction of errors in the individual measurements before the data set can be treated statistically.

The *error* of a measurement (as defined above) is the difference between the measured value and the true value. When the error is within certain limits, the measurement is termed as accurate. In assessing the quality of any analysis, the two important criteria are precision (or reproducibility and repeatability) and accuracy (bias between the estimated and true value).

The *precision* refers to how closely individual measurements within the group of measurements agree. High precision does not necessarily mean high accuracy (Figure 1). In fact, a group of measurements may be very precise, but inaccurate because of large systematic error. Precision is influenced by the conditions (operator, apparatus and laboratory) that may differ (reproducibility) or remain the same (repeatability) for the same analytical method performed on identical test materials. The standard deviation and variance are measures of precision.

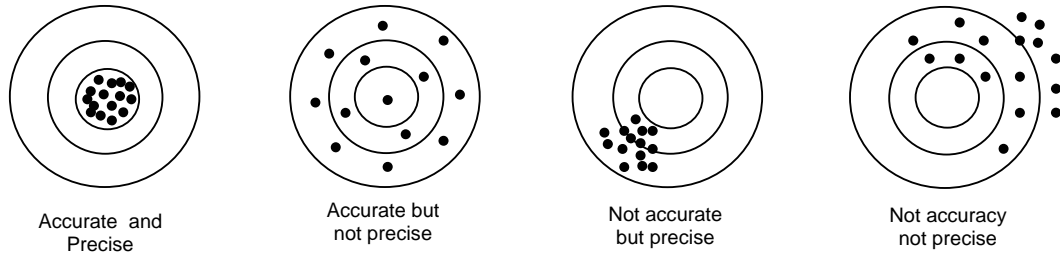


Figure 1. Accuracy and Precision

Most errors are a combination of indeterminate and determinate error. An *indeterminate error*, sometimes referred to as random error, is one that varies in a non-reproducible way above and below the true value. Most indeterminate errors are small and can be treated statistically because they are evenly distributed around the true value. *Determinate error* (systematic error) is reproducible from one analysis to the next and generally biases a result in one direction. When the indeterminate error is large, or the precision low, the results are not trustworthy unless the central value of a large number of values is taken. This central value is referred to as the average or means (m), which is defined as:

$$\text{Average } (m) = \frac{\sum_{i=1}^{i=N} X_i}{N}$$

Where X_i represents the individual measurements and N is the total number of measurements. When determinant errors are absent, the average gives a good estimate of the true value.

In some cases, the *median* or middle value of a set of measurements may be used. By arranging the measurements in numerical order, the median is the middle value in the set if the number of measurements is odd and the average of the central pair of measurements if the number of measurements is even.

The standard deviation gives a measure of the scatter in a set of measurements and is statistically useful, particularly for random errors:

$$\text{Standard deviations (SD)} = \left[\frac{\sum_{i=1}^{i=N} d_i^2}{(N-1)} \right]^{1/2}$$

where i denotes observation number and N is total number of observations e.g.,

$i = 1$ then $d_1 = x_1 - m$, $d_2 = x_2 - m$ (m is the mean, and x_1, x_2, \dots are the observations). The square of the standard deviation, called the variance, is also widely used in the treatment of data.

Consider an operation in which a large almost infinite number of measurements are made and the frequency of the various values is plotted against the values. In this operation, a symmetrical curve is obtained if the measurement involves only random errors. This curve, known as a standard error, normal-error, normal distribution or Gaussian-distribution curve, shows the distribution of measurements. The broader and flatter the curve, the less precise the measurements. The mean and the standard deviation define the curve. The mean locates the curve along the horizontal axis. Broader curves, therefore, have larger standard deviations.

Reliability of an average

It is not always practicable to take a large number of measurements, and often a small set of values must be used to estimate the range within which the true value will probably fall. The limits of the confidence interval around the average can be determined using a statistically derived correction factor and taking into consideration the number of measurements in the set. Generally, the true value is estimated as follows:

$$\text{True value} = m \pm t \sigma_m$$

$$\text{Where } \sigma_m = \sigma/\sqrt{N}$$

where t is the tabulated correction factor for a given number of measurements N at a particular probability level, and σ is standard deviation. The 95% level is often used in estimates of validity of analytical measurements. Calculation of confidence limits by this procedure is only valid when determinate errors are absent. The value of t for N equal to 10 or 20 is already close to limiting value of t where N equals infinity (Table 1).

Table 1: t Values for calculating confidence limits of averages

N	t for 50% Probability	t for 95% Probability	t for 99% Probability
2	1.00	12.71	63.66
3	0.82	4.30	9.92
4	0.76	3.18	5.84
5	0.74	2.78	4.60
10	0.70	2.26	3.25
20	0.69	2.09	2.86
∞	0.67	1.96	2.58

The Q Test : Criteria for Rejection

Often when performing analytical determinations, the analyst is faced with one or more results that seem to be out of line with the others. When the number of values is large and the mean to be calculated, the suspect value may be rejected if (not clear) it lies outside the confidence limits. However, when the number of values is small, the confidence limits may be spread so widely that no values can be rejected.

The Q test is an excellent method for handling data rejection when a small number of observations are involved (Winkleman et al, 1990).

$$Q = [(suspect\ value - nearest\ value) / (largest\ value - smallest\ value)]$$

The suspect value should not be used in calculating means and standard deviations if the calculated Q value exceeds that found on a Q table. If the result is to be discarded, there is 90% confidence that the result was subject to error that did not occur to other values. For example, the following were obtained for normality of a standard acid solution: 0.1001, 0.1013, 0.1011, 0.1014 and 0.1016. The analyst suspects the 0.1001 result:

$$Q = (0.1001 - 0.1011) / (0.1016 - 0.1001) = 0.67$$

Thus the Q value found is 0.67. On comparing this value from the Q table (Table 2), $Q_{90}\%$ for $N = 5$ is 0.64, which is less than found value and therefore, the value 0.1001 can be rejected with 90% confidence.

Table 2: Value of Rejection Quotient- Q at different confidence levels (Winkleman et al, 1990)

N	Q90	Q95	Q99
3	0.941	0.970	0.994
4	0.765	0.829	0.926
5	0.642	0.710	0.821
6	0.560	0.625	0.740
7	0.507	0.568	0.680
8	0.468	0.526	0.634
9	0.437	0.493	0.598
10	0.412	0.466	0.568

The square of the standard deviation is called the variance, which is also widely used in the treatment of data.

Significant figure

All the reports contain certain numerical values, these numerical values or reported digits in any result are expected to be known definitely, but the last digit is always doubtful. If in a report result is expressed as 6.3 mg/L for an analyte, it explains that '6' is certain but may be uncertain for '.3', which could be either .2 or .4. The precision in such a case would be 6.2 to 6.4. On the other hand if the method was so good that the result 6.31 mg/L could be reported, then the result will be certain up to 6.3 and uncertain value will be .01. Therefore, report only such figures those are justified by the accuracy of work (Table 3).

Table 3. Reporting number and implied precision

Reporting number	Implied precision
6	5 to 6
6.3	6.2 to 6.4
6.31	6.30 to 6.32
6.310	6.309 to 6.311

A good example explaining the importance of significant figure is the density of water at various temperatures (Table 4).

Table 4. Water density (g cm^{-3}) at different temperature and significant figure

Temperature	Actual density	Reporting way with Significant Figure		
		One	Two	Three
10	0.99970	1.0	1.00	1.000
20	0.99820	1.0	1.00	0.998
30	0.99565	1.0	1.00	0.996
36	0.99369	1.0	0.99	0.994

Significant figure is also important while documenting a method e.g. in a method sheet if 5 ml is seen when referring a sample aliquot, the immediate interpretation is dispensing of 4 to 6 ml by technician and implied initial error in the analysis. On the other hand 5.00 ml (dispensing 4.99 to 5.01ml) will significantly decrease the error (20% to 0.2%)

Control Chart

Control charts were originally designed to monitor manufacturing processes with set tolerance limits. In analysis these charts are used to monitor accuracy and precision of the method. These are one of the essential tools for quality control of the analysis. It can be maintained for any individual quality control check by daily plotting of new points on the control chart. These charts can be used for laboratory control standards (LCS) or calibration check standard (CCS) or for replicate analysis of calibration check standard. These are set by calculating the mean values and standard deviation and/or by using percentage. Most important among these charts are the mean and accuracy charts. These charts are constructed from the average and SD of a specified number of measurements of analyte of interest. It includes upper warning level (UWL) and lower warning level (LWL) and upper control level (UCL) and lower control level (LCL). Normally $X \pm 2SD$ and $X \pm 3SD$ are used as limits for warning and control levels respectively, where X is the mean and SD is the standard deviation. Actual QC data produced in the Environmental Laboratory of ERWDA is represented in Figures 2 & 3.

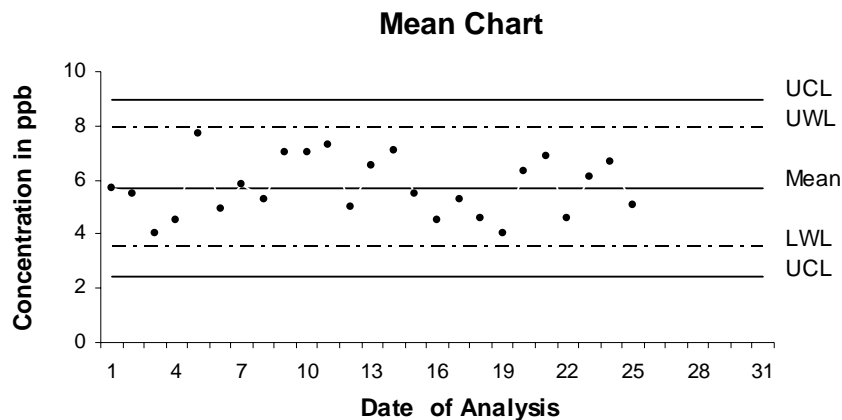


Figure 2. Mean Chart

Figure 2 represents a mean chart where the concentration of analyte is presented against the date of analysis.

Figure 3 represents accuracy chart where recovery of analyte is presented against the date of analysis.

Accuracy Chart

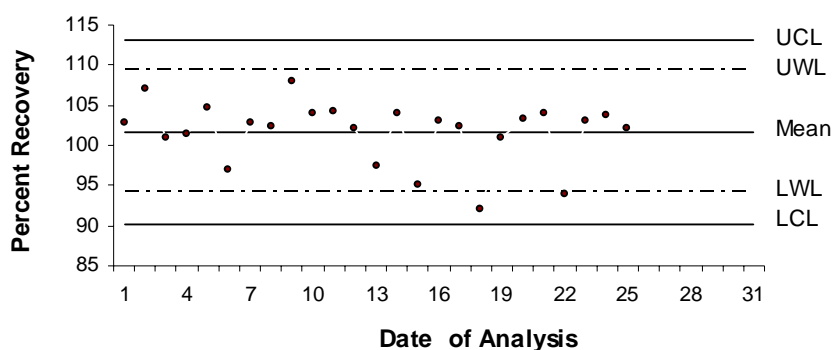


Figure 3. Accuracy Chart

A typical control chart is shown in Figure 4. Analytical results occurring outside the control limits indicate that the method should be checked as described in the following brief description about the use of control chart with limits e.g. control limit, warning limit etc.

Control limit (CL). If one result exceeds the CL (Upper or Lower) analysis should be repeated. If repeat result is within CL, analysis can be continued. If it again exceeds, investigate the problem and rectify it before further analysis.

Warning limit (WL). If two out of the three successive readings exceed a WL (Upper or Lower) analyze another sample, if the result is within the WL, analysis may continue. If the next result again exceeds the WL, investigate and rectify the problem before further analysis.

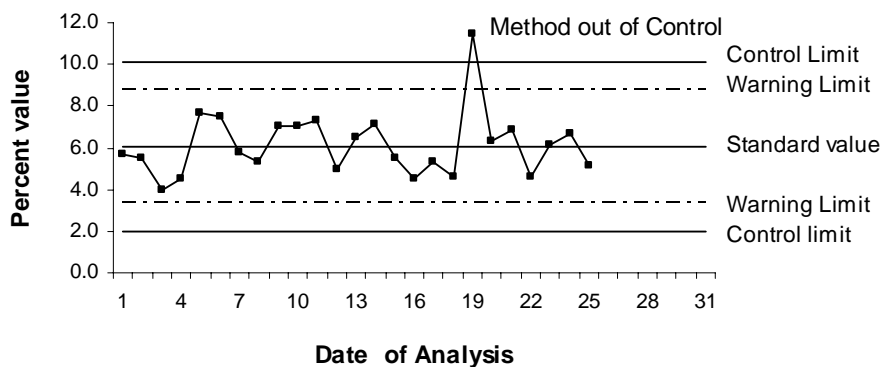


Figure 4. A typical quality control chart for an analyte

Standard deviation (SD) - If four out of five successive readings exceed ± 1 SD, or found in increasing or decreasing order, analyze a new sample. If the next result is < 1 SD or the order is changed, analysis may be continued. Otherwise, the problem should be investigated and rectified before further analysis.

Continuous Improvement Chart

Lack of knowledge and complexity of process/method is the normal cause of inconsistency and delay. A stable, simple and well documented process provides better productivity and reliable data/results. A systematic approach can improve the process in terms of minimizing faults and reducing the complexity of the process, which increases productivity. Steps shown in Figure 5 are useful to achieve an improved process.

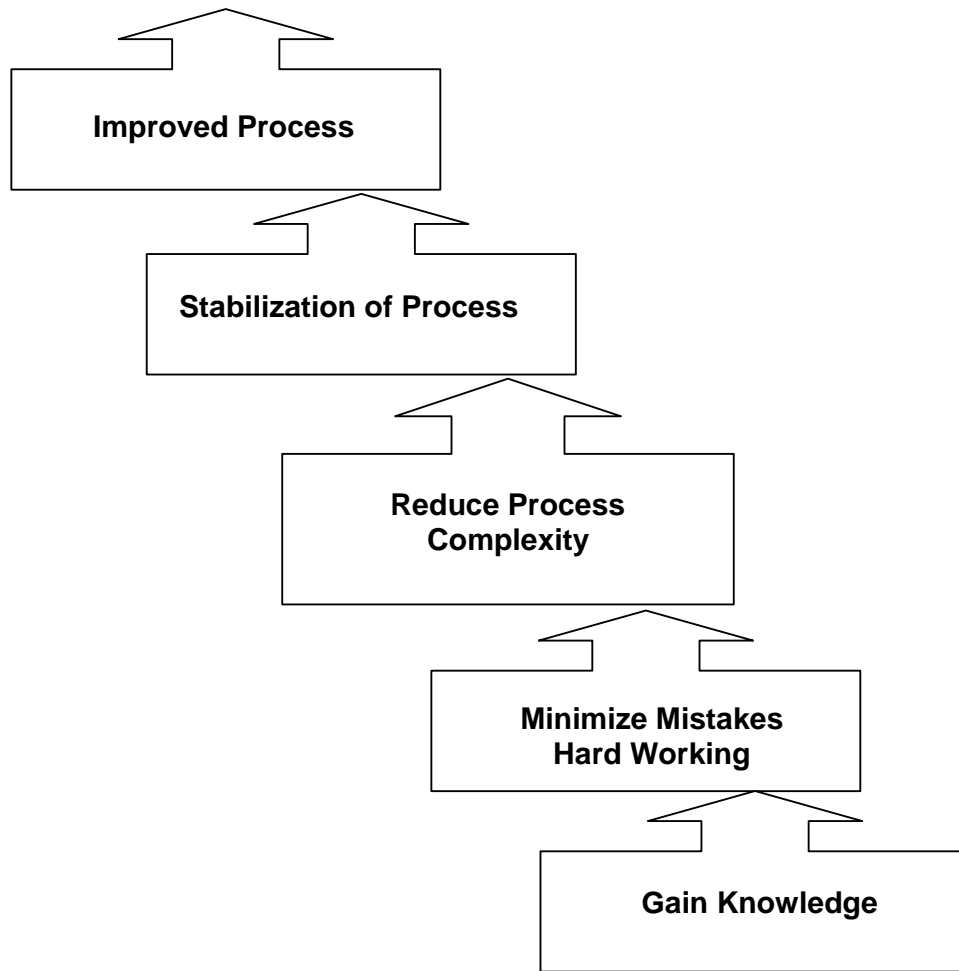


Figure 5. Steps for Improvement of Work Quality and Productivity
(Source: PRISM Quality Enhancement Corporation, 2000)

Important points involved in Laboratory Quality Control

- Quality System Documentation
 - Description of laboratory policies/ procedures/ operations
 - Aid to ensure laboratory continues to operate effectively in the absence of key individuals
 - Communication
 - Training aid
 - Evidence of systematic planning of Quality Management System
- Supervision of staff
- Checks on sample processing, equipment and consumables
- Adherence to specific test procedures
- Use of retained samples, standards, reference materials, replicate measurements
- Inter-laboratory comparisons, proficiency testing
- Controlled laboratory environment
- Control of testing environment
- Checking of data, checking of calculations, records, reports
- Preparation of Quality Control Chart
- Statistical calculations
- Planned and Systematic actions

Laboratory Management for Soil Data Collection

Management of various tasks of analytical work in the laboratory is very essential to achieve reliable results in acceptable time frames and at reasonable cost. It encompasses all stages of sample analysis from receipt of sample to the finalization of results after conducting quality assurance tests. To obtain reliable data from multi-analysis determinations, the laboratory poses serious challenges. Good laboratory practices in the soil analytical laboratory are essential for the generation of reliable analytical results. This is achieved by the standardization of analytical methods using procedures suitable for available equipment. To generate reliable results, a quality control program incorporating reference sample into each batch of samples must be undertaken as

a part of the routine analytical determinations carried out using standard procedures. This will lead to a good laboratory management that will guarantee the generation of reliable results.

A critical management is the means of verifications of the reliable analytical data generated by the laboratory. It is important, rather, mandatory to compare the laboratory results from an alternative laboratory or by the inclusion, within the sample batches, of soil reference samples of known analytical values.

The errors may arise from calculation by different procedures e.g., using scientific calculators or manually. This problem can be overcome in the initial data-processing stage and in subsequent evaluation process, it is facilitated by the use of the PC and suitable software packages such as the spreadsheet. A spreadsheet once set up for data processing will automatically recalculate results when new input data is entered. This eliminates the possible source of errors that may arise from different laboratory staff calculating results by different procedures. In addition, the spreadsheet may act as a simple database for both storage and presentation of data.

Quality Assurance Programs

As mentioned earlier, soil data are required for: soil survey and classification (viz. Soil or site characterization); fertilizer recommendations; soil conservation, environmental monitoring; and assessment of sustainability etc. Quality assurance commences with the selection of the indicators to be analyzed. The procedure adopted must be satisfactory in the following aspects:

- Sensitivity of the method (measurable standard deviation)
- Precision (reproducibility and repeatability)
- Safety (to the analyst and residues discarded)
- Cost (in terms of money for chemicals, manpower etc.).

The precision is influenced by the conditions (operator, apparatus and laboratory) which may differ (reproducibility) or remain the same (repeatability) for the same analytical method performed on identical test materials. The standard deviation and variance are measure of precision.

Quality assurance programs i.e. quality control can be implemented in almost any organized activity or work. In soil research, quality assurance programs need to be in place in the

field during site selection and sample collection, in the laboratory (for sample preparation, analysis), and in the office (for data evaluation, storage, retrieval, and interpretation). The quality of sampling will influence the value of the analytical data obtained. Field sampling is often the weakest link in obtaining the quality soil data, as the sampling work is normally assigned to unskilled or semi-skilled workers. For reliable sampling, it is mandatory that a soil scientist should be on the sampling site, and direct the workers for sampling or even participate in sampling mission. The team must be very clear about the objective of sampling, type of sample and intensity of sampling. A representative sample should always be collected from a sampling unit. To ensure that samples are not interchanged, a label should be placed in the bag and also tie a label to the outside of the bag. The samples should be handled to ensure that there is no contamination or deterioration. The samples, when placed in drying cabinets or tray for drying should be accompanied by the tag label to prevent confusion. Subsequent to drying, the soil samples should be ground to pass through a 2 mm sieve. Care should be exercised to ensure that the samples are not ground too finely. If coarser fractions e.g., gravels, CaCO_3 concretions are in significant quantities, they may be reported separately.

Sources of Errors in the Laboratory Determination of Soil Samples

There are a number of sources of errors in the laboratory determinations of soil samples. They should be carefully checked. The errors may be due to following reasons-improper calibration of the equipment, faulty equipment or lack of maintenance, use of expired chemicals, unstable electricity supply, heating up of equipment, improper dilution of chemical used, contaminants in chemicals used, standard solutions prepared from stock solution stored too long, operator not following directions, lack of understanding, improper mixing order of chemicals, error in the method, contamination in the glassware, error in the calculation procedure, error in copying the data, etc.

The analysis of the soil saturation extracts collected from the soil paste is a standard procedure for routine soil characterization. The extract, if left uncovered, usually reduces its volume and distorts the ion balance and E_{Ce} values. This could be caused when the analyst analyzes the extract for some parameters and stores the extract for a longer period for other analyses. For such determinations, batches of 10-15 soil samples are recommended for extract

analyses. All the determinations must be completed on each batch on the same day to avoid such errors.

Inter-laboratory cross-checks at times involve national, regional and international collaboration. It is also advisable that other collaborating laboratories in the country or abroad should analyze few selected samples. The results obtained from different laboratories should be assessed statistically to determine variability. If it is high, the laboratories with the anomalous results should re-examine the analyses to ascertain the sources of error(s) and reanalyze the samples and ensure that they can obtain a high standard of accuracy.

METHODS OF QUALITY ASSESSMENT FOR SOIL ANALYTICAL DATA

The mechanisms of arriving at and maintaining reliable analytical data fall into the category of a quality assurance and quality control system. The quality of the laboratory physical and chemical results determines the success of the project. To achieve the quality of the results, it is necessary to first review what methods are being utilized, are these standard methods and, are they suited to the local conditions? In this publication, the procedures described for data assessment are of international scope. Overall, the objectives are to enhance the quality of the results for better interpretation and management.

Cross-Checking Chemical Analyses for Consistency and Reliability

A means of locating gross errors in the chemical analyses of soils is provided by the considerable number of interrelations that exist among the values obtained for various determinations. An understanding of the principles involved in these interrelations aids in the interpretation of the analyses (Richards, 1954).

The saturated soil paste pH is the apparent pH of the soil : water mixture and is a key indicator in many soil interrelations. The pH is dependent upon the dissolved CO₂ concentration, moisture content of the mixture; exchangeable cation composition, soluble salt composition and concentration, and the presence and amount of gypsum and alkaline earth carbonate (USDA-NRCS, 1995c).

The saturated paste pH (pH_s) is popular in regions with soils containing soluble salts such as arid and semi-arid region soils. The water content of the saturated paste varies with soil water storage characteristics. The pH_s is more indicative of the saturated, irrigated pH than is the soil pH measurement at a constant soil : water ratio (Richards, 1954).

The pH_s is also a pH at which the saturation extract is removed for salt analyses, and, hence, is pH and the dilution at which the sodicity level, i.e. sodium adsorption ratio (SAR) is determined. SAR could not be determined at any other soil/water ratios (USDA-NRCS, 1995c).

Soil pH is one of the most common and important measurements in standard soil analysis. Many soil chemical and biological reactions are controlled by the pH of the soil solution in equilibrium with the soil particle surfaces.

Standard Rules of Thumb

Some standard rules of thumb (Richards, 1954; USDA-NRCS, 1995c; USDA-NRCS, 1996a,b) applied to the saturated paste pH and saturation extract are as follows. These rules are given and the methods of their utilization to assess the quality of the data produced are discussed.

ECe and Total Cations and Anions Concentrations

Rule (Total cations = Total anions, expressed on equivalent basis)

Soil salinity is a widespread limitation to agricultural production and a feature of land degradation in semi-arid and arid region soils throughout the world. The accumulation of soluble salts in the soil profile curtails crop growth by increasing the osmotic potential of the soil solution and inducing specific ion toxicity or nutrient imbalances. Soil structure is affected by salinity and sodicity. A number of approaches have been devised to characterize soil salinity. Most conventional methods employ aqueous or direct extraction of the soil solution and subsequent analysis of salt concentrations. The standard way of establishing soil salinity is through the measurement of electrical conductivity of the soil saturation extract (EC_e).

To establish the chemical characteristics of the soils, the representative soil samples are subjected to standard routine chemical determinations. To obtain these results the soil samples are used to prepare standard saturated soil pastes to measure soil pH, and the saturated soil

paste is used to collect saturation extract under vacuum. The saturation extract is analyzed for the measurement of electrical conductivity (ECe) for salinity establishment, and for anions (CO_3^{2-} , HCO_3^- , Cl^- , SO_4^{2-}) and cations (Na^+ , K^+ , Ca^{2+} , Mg^{2+}). The ionic composition in the saturation extract is expressed on equivalent weight basis rather than on their actual weight basis. Equivalent is a large value and is not expected in soil solutions and, therefore, a small value as milliequivalent (meq) is used either on per 100 g or per liter basis. Having measured the cations and anions, according to the above rule, theoretically, the anions must equal cations when expressed on a meq/l basis and, therefore, is one basis for quality assessment of the chemical soil data through ion balance assessment.

Ion Balance. Soil saturation extracts collected from saturated soil pastes are electrically neutral and, therefore, the total charges on the cations and anions reported in the analysis should be equal. The total positive and negative charges are obtained by summing the equivalents of cations and anions, respectively. The ion balance error (IBE) is normally expressed by the difference as a percentage of the sum. The test is based on the percentage difference defined as follows (the criterion of acceptance is given in Table 5).

$$\text{Ion balance error (\% difference)} = [(\sum \text{cations} - \sum \text{anions}) / (\sum \text{cations} + \sum \text{anions})] \times 100$$

Where cations and anions are in me/l.

Table 5. Criteria for acceptance of Ion Balance Error (Clesceri et al, 1998)

Anions sum (meq/l)	Acceptable % Difference in IBE
0-3.0	± 0.2 meq/l (at very low EC)
3.0-10.0	± 2%
10.0-800	5%
& more	

Method of Assessment. There are two methods of assessment for ionic balance in the soil solutions/extracts. They are as follows.

Method 1. The ECe is a measure of flow of current through the soil extract containing electrolytes. The flow of current is proportional to the concentration of ions or soluble salts in the solutions, therefore, the concentration of soluble salts can be obtained from the ECe values. One way of obtaining the values of soluble salts is by comparing the ECe values on the standard line constructed from ECe and total soluble salts (TSS) (Richards, 1954, p. 12). Having obtained this

value, this becomes a standard to check the difference of the measured anions and cations. Keeping the TSS obtained as 100%, the difference of the total measured anions and cations in the laboratory is then calculated and the frequency of soil samples distribution is presented in the ranges of % difference from the standard. An example of such an assessment is given here.

Example: It is assumed that the ECe of an unknown soil saturation extract is measured as 15dSm^{-1} , and total measured anions and cations in the same extract are 125 and 250 me/l respectively. To assess the balance of these anions and cations, their values are compared with that obtained from the straight line against 15dSm^{-1} . The TSS against 15dSm^{-1} is obtained as 190 me/l. Keeping this value as 100%, the percent overestimation (OE) or underestimation (UE) is then calculated:

TSS from the ECe 15 dSm^{-1}	= 190 meq/l
Total anions	= 125 meq/l
Total cations	= 250 meq/l

According to above rule the total anions and total cations separately should have been measured to match approximately 190 me/l or close to this value. This is not the case with the above example, which shows 34% underestimation (UE) of anions and 32% overestimation (OE) of cations. Similarly, the OE or UE can be calculated for all the samples, and frequency distribution may be presented in the form of a bar diagram between the % difference range (UE or OE) from the standard value and the number of samples. This way, an overall quality of a large number of samples on the ionic data can be assessed at a glance. The TSS obtained from the curve may vary slightly from the measured values on different soils, however, this may be considered only a guide.

Method 2. The second method of assessing the balance of ionic data is described here by giving suitable examples. The concentrations of ions when presented as ppm (mg/l or $\mu\text{g/ml}$), may be converted to me/l by dividing with equivalent weight of the respected atom.

Example I. Calculation of the ion balance error for the following analysis:

Cations in mg/l:	$\text{Ca}^{2+} = 200$; $\text{Mg}^{2+} = 60$; $\text{Na}^+ = 230$; and $\text{K}^+ = 39$
Cations in me/l:	$\text{Ca}^{2+} = 10$; $\text{Mg}^{2+} = 5$; $\text{Na}^+ = 10$ and $\text{K}^+ = 1$
Total cations in	= 26 me/l
Anions in mg/l:	$\text{CO}_3^{2-} = 60$; $\text{HCO}_3^- = 122$; $\text{Cl}^- = 355$; and $\text{SO}_4^{2-} = 480$
Anions in me/l:	$\text{CO}_3^{2-} = 2$; $\text{HCO}_3^- = 2$; $\text{Cl}^- = 10$; and $\text{SO}_4^{2-} = 10$
Total anions	= 24 me/l

Therefore, IBE is:

$$\begin{aligned} & [(\sum \text{ cations} - \sum \text{ anions}) / (\sum \text{ cations} + \sum \text{ anions})] \times 100 \\ & = 4\% \end{aligned}$$

The above ion balance error (4%) is within the acceptable range and therefore the results are acceptable.

Example-II. Calculation of the ion balance error for the following analysis:

Cations in mg/l:	Ca ²⁺ = 400; Mg ²⁺ = 24; Na ⁺ = 460; K ⁺ = 78
Cations in me/l:	Ca ²⁺ = 20; Mg ²⁺ = 2; Na ⁺ = 20; K ⁺ = 2
<i>Total cations = 44 me/l</i>	
Anions in mg/l:	CO ₃ ²⁻ = 60; HCO ₃ ⁻ = 244; Cl ⁻ = 177.5; and SO ₄ ²⁻ = 480
Anions in me/l:	CO ₃ ²⁻ = 2; HCO ₃ ⁻ = 4; Cl ⁻ = 5; and SO ₄ ²⁻ = 10
<i>Total anions = 21 me/l</i>	

Therefore, IBE is:

$$\begin{aligned} & [(\sum \text{ cations} - \sum \text{ anions}) / (\sum \text{ cations} + \sum \text{ anions})] \times 100 \\ & = 35.4\% \end{aligned}$$

The ion balance error 35.4 % is not within the acceptable range and, therefore, the results are technically incorrect. A frequency diagram can be constructed to look at the imbalance of the ionic data at a glance. This assessment is important as a number of other relationships are based on these ions in the saturation extract.

In case of IBE higher than the acceptable range, which does not meet the criteria of acceptability, the sum is suspect and a reanalysis of the samples is recommended.

pHs, Ca and Mg Concentrations

Rule of Thumb: Concentrations of Ca²⁺ and Mg²⁺ are seldom > 2 meq/l at pHs > 9

This rule of thumb can be tested on the analyses of soil saturation extracts for Ca²⁺, Mg²⁺ concentrations and by comparing the values with pHs of the respective samples. The soil samples not following this rule should be indicated out. Reasons for deviation could be found by checking the original laboratory data sheets and calculation procedures. In the absence of reasons of error, the samples should be reanalyzed for their correct values.

pHs, CO₃²⁻ and HCO₃⁻ Concentrations.

Rule of Thumb:

- i) Carbonate concentration (meq/l) is measurable only if pHs >9
- ii) Bicarbonate concentration is rarely > 10 meq/l in the absence of carbonates

iii) Bicarbonate concentration is seldom > 3 or 4 meq/l if pHs < 7

The rule of thumb can only be tested provided the sum of anions and cations follows the acceptable IBE, and has a strong correlation with the ECe values. The pHs, CO_3^{2-} and HCO_3^- have a strong relationship in the saturation extracts. The presence or absence of one or the other and correlation with the pHs value was given earlier. During the quality assurance task, the values of HCO_3^- , CO_3^{2-} and pHs should be compared and any deviation from the rules of thumb should be indicated and reanalysis of the samples required for their correct values.

The pHs and Gypsum

Rule of Thumb:

- i) Gypsum is rarely present if pHs > 8 .
- ii) Gypsum has variable solubility in saline solutions (20 to 50 meq/l)
- iii) Check for the presence of gypsum if Ca concentration > 20 meq/l and pHs < 8.2

Similarly, the rule of thumb can only be tested provided the sum of anions and cations follows the acceptable IBE, and has a strong correlation with ECe values. The pHs of the soil samples and the gypsum presence are correlated. Similarly, gypsum has a correlation with Ca concentration and pHs value. During the quality assurance task, the values of Ca, gypsum and pHs are compared and any deviation from the rules of thumb should be indicated, and samples should be reanalyzed for their correct values.

The pHs, Exchangeable Sodium Percentage (ESP) and Alkaline-Earth Carbonates

Rule of Thumb:

- i) Alkaline Earth Carbonate and ESP > 15 are indicated if pHs > 8
- ii) ESP < 15 may or may not be indicated if pHs < 8
- iii) No alkaline earth carbonates are indicated if pHs < 7.5
- iv) The pHs of calcareous soil is invariably in excess of 7.0 and generally in excess of 7.5; a non-calcareous soil may have a pH reading as high as 7.3 or 7.4.

These relations show correlation between pHs, ESP and alkaline earth carbonates. The ESP can be measured through a standard calculation procedure using the SAR data, or through using exchangeable sodium and cation exchange capacity (CEC) values.

i) ESP calculation from the SAR values

$$\text{ESP} = [100 (-0.0126 + 0.01475 \text{ SAR})] / [1 + (-0.0126 + 0.01475 \text{ SAR})]$$

ii) ESP calculation from exchangeable Na and CEC values

$$\text{ESP} = [\text{Exch. Na}/\text{CEC}] \times 100$$

where exchangeable Na and CEC are expressed as me/100g

The alkaline earth carbonates can be measured by a number of methods. The calcimeter method is considered as the standard method. Similar to the stated rules, the values of pHs, ESP and CaCO₃ equivalents are checked for each sample. Wherever there is an inconsistency in the rule, this should be recorded separately, reasons should be justified or reanalysis of samples is required for all or some of the parameters.

Exchangeable Sodium Percentage (ESP) and Sodium Adsorption Ratio (SAR)

Rule of Thumb: In general, ESP increases with SAR

The exchangeable sodium percentage (ESP) is an important soil parameter indicating the soil sodicity. The ESP is a measure of relative amounts of sodium on the exchange complex of the soil. The ESP correlates well with soil physical properties, e.g., shrink swell capacity, hydraulic conductivity, dispersion and swelling volume of clay. Having established the ESP accurately, soils are then classified into salt-affected soil categories, and to identify sodic or natric horizons for soil taxonomy purposes.

To test the rules of thumb, the ESP calculated from SAR and from the exchangeable sodium (ES) and CEC are usually compared. The ESP either calculated from the SAR or from ES and CEC should be approximately equal. The direct comparison of the ESP obtained from both the methods and their percent difference between each other shows the validity of these values. If both the values have difference, then the reasons for these differences should be located in the basic data used for such calculations. In the absence of any reason, the samples should be reanalyzed for their correct values. It is also very important to be sure that standard methods suitable for the soils under investigation have been employed.

Water Retention

Water retention at 1/3 bar ($W_{1/3}$) is important as it approximately represents the moisture equivalent at field capacity. The field capacity has no fixed relationship to soil water potential, therefore, it cannot be considered as a soil moisture constant (Kramer, 1969).

Water retention at 1/10 bar ($W_{1/10}$) may be used as the upper limit of plant available water for coarser material, such as most soils of the United Arab Emirates; the coarse material must meet the sandy or sandy-skeletal family particle size criteria and also be coarser than loamy fine sand with < 2 % organic carbon and < 5 % water at 1500 kpa suction and computed total porosity of < 2 mm fraction must be > 35 %.

Water retention at 15 bar (W_{15}) is the permanent wilting percentage in the lower limit of soil water storage for plant growth and has considerable practical significance, as plants remain wilted at permanent wilting percentage (PWP). The water retention at 15 bar (W_{15}) has become identified with PWP and is frequently used as an index of PWP.

Water Retention at 15 bar (W_{15}), Data Assessment

Rule of Thumb: $(W_{15})/\% \text{clay} \sim 0.4$ or $\% \text{clay} = W_{15} \times 2.5$

where

W_{15} = weight percentage of water retained at 15 bar suction on a < 2 mm basis;

Clay % (estimate) = % water retention at 15 bar suction X 2.5

This relationship for quality assessment of W_{15} and clay % of the soil samples has been suggested by USDA-NRCS (1996a,b). It is reported that, as retention at 15 bar has been equated with 0.0002 mm (0.2 μm) pore diameter, a high correlation exists between 15 bar water and clay content (i.e., % clay). As such, this ratio is a good tool for data assessment. In the presence of disagreement between the W_{15} and clay relationship of soil samples, the reliability of the available water content calculated from the difference of W_{15} and $W_{1/10}$ becomes questionable. Clay-sized carbonates tend to decrease the ratio in most of cases. The W_{15} for carbonate clay is $\sim 2/3$ the corresponding value for the non-carbonate clays (Nettleton et al., 1991). This point should be taken into consideration while assessing the quality of W_{15} and clay values. The USDA-NRCS (1995c) reported that W_{15}/clay may be lower than 0.4 reference point. Low activity clay, e.g.,

kaolinite, chlorite and mica, tend to lower the ratio to <0.35 and high activity clay, e.g., smectites and some vermiculites, tend to increase this ratio. The USDA-NRCS (1995c) presents the ratio for different soil groups dominated by different kinds of clay minerals as: 0.45 for smectites, 0.42 for micas, and 0.32 for clay rich horizons.

The quality assessment of W_{15} and clay content are important as W_{15} indicates the available water contents and help irrigation scheduling in irrigated areas. Clay, being an active component in the soil mineral matter, controls most of the physical, chemical and nutritional properties of the soils. Clay is also an important component to establish soil texture, which is a permanent soil property and controls the physical behavior of soils.

To assess the quality of W_{15} results and the clay content, the ratio of all the samples should be calculated. The samples showing higher values than 0.4 should be pointed out, particularly the soils containing appreciable amounts of calcium carbonates equivalents and gypsum, the ratio for these soils should be lower than 0.4. The unexpected ratios from the number of soil samples should be indicated out for their justification and/or reanalysis of soil samples.

Saturation percentage (SP) and 15 Bar Water Content (W_{15})

Rule of Thumb: $SP \approx 4 \times 15\text{-bar water content}$
 $SP \approx 2 \times \text{upper end field soil moisture content}$
 $AWC \approx SP/4$

If texture (coarse, medium and fine) is known, the SP relationship is as follows:

- i) $SP \approx 6.33 \times 15\text{-bar water content}$ (coarse texture) when W_{15} is 2.6 to 6.5
 - ii) $SP \approx 4 \times 15\text{-bar water content}$ (medium texture) when W_{15} is 6.6 to 15.0
 - iii) $SP \approx 3.25 \times 15\text{-bar water content}$ (fine texture) when W_{15} is > 15
 - iv) $SP \approx 3.67 \times 15\text{-bar water content}$ (organic) when W_{15} is > 15
- AWC (Available water content for a medium textured soil) = $SP/4$

The review of the particle size distribution data should be made before deciding to use (i), (ii) or (iii) above. The soils of the United Arab Emirates are dominantly coarse and very few are medium and fine textured.

After using the W_{15} and factor 6.33 (for coarse textured) and 4 (medium textured), the calculated SP values can then be compared with the measured SP values (from saturation paste). Keeping the calculated SP as 100%, the difference of the reported SP is calculated and is shown as % OE or UE and a frequency diagram is constructed against % difference ranges.

The values of water retention difference (WRD) are a calculated value from W_{15} and other soil characteristics. To obtain the correct values of WRD, the quality of W_{15} must be checked.

The values of SP are important in a number of relations, e.g. they are used;

- to calculate available water content in soils;
- to convert the soluble anions and cations of the saturation extract from solution (meq/l) to soil weight basis (meq/100g); and
- the above conversion is necessary to calculate the amount of exchangeable cations, and for further calculation of ESP, to classify soils under different categories of salt-affected soils according to USDA classification. The inaccurate SP values could result in the wrong assessment of sodicity levels and question's the reclamation of saline-sodic and sodic soil by gypsum application based on the wrong sodicity level.

Cation Exchange Capacity and Clay Assessment

Rule of Thumb: In general, CEC increases with clay content and nature of clay minerals

In addition to the calculation of ESP, the CEC values are very important from a soil quality point of view. The CEC is the quantity of cations attracted to the surfaces of colloids (clay) and organic matter. The quantity of these cations expressed on a me/100 g basis is termed as CEC. It is one of the important chemical properties of soils and is usually closely related to soil fertility. Soils that have a low CEC hold fewer cations and may require more frequent applications of fertilizers than soils that have high CEC; soils with high CEC have the potential to retain cations, which prevents the pollution of groundwater (Soil Survey Division Staff, 1993). A thorough understanding of CEC is, therefore, necessary to understand soil fertility and acidity (Tisdale and Nelson, 1975). The CEC is usually related to the amount of clay and the type of clay minerals. Generally, Kaolinite (1:1) mineral colloids have CEC values of 1 to 10 meq per 100g; 2:1 mineral colloids such as montmorillonite and vermiculite (80 to 150 meq per 100g), chlorite (2:1:1) and micas (2:1), 20 to 40 meq per 100g; and organic colloids, 100 to 300 meq per 100g. The USDA-NRCS (1995a) and Rhoades (1982) quoted the common CEC values for soil components (Table 6).

Table 6. Soil components and their respected CEC values

<u>Soil component</u>	<u>meq per 100 g</u>
Organic matter	200 - 400
"Amorphous" clay	160 (at pH 8.2)
Vermiculite	100 - 150
Montmorillonite	60 - 100
Illite	20 - 40

Chlorite	10 - 40
Kaolinite	2 - 16
Halloysite 4H ₂ O	40 - 50
Halloysite 2H ₂ O	5 - 10
Sequioxides	0

It is also possible to approximate the CEC of a soil in a given area if the type and contents of the dominant clay colloids is known. Brady (1990) recommended 0.5 meq/100 g CEC for each 1% clay and, for humus 2 meq/100g, in humid temperate region soils. He further reported that for soils dominated by kaolinite, comparable figure might be 0.1 meq/100g for each 1% of clay. Brady (1990) recommended approximating CEC of any area where the types and contents of dominant clays and humus are known. From the information on soil minerals and organic matter contents, it is possible that CEC values can be estimated and compared with those determined by laboratory methods. Such a comparison would help in assessing the quality of CEC and clay contents. For example, if a soil contains 24% clay and 1% organic matter, the expected approximated CEC values could be 14 me/100g (Brady, 1990).

If this rule does not correlate with the reported values, then it could be speculated initially that either the measurement of clay and organic matter is not accurate or the CEC is inaccurate. The error may occur in both if the standard methods of their determination are not followed. Care should be taken in using this assessment on monomineralic (soils with only one type of clay minerals) soils which may not meet these criteria; therefore, such an assessment may be taken only as a guide.

Weight % of < 2 mm Whole Soil Basis (Particle Size Distribution)

Rule of Thumb: Weight % of < 2 mm (passing 10 mesh sieve) = Weight % of < 2 mm

The single most important soil physical property is particle size, and precise meaning is given to the term soil texture through the concept of particle size distribution-PSD (Skopp, 1992). It is important as the behavior of soil physical and chemical properties is greatly influenced by particle size classes present and their relative abundance. The PSD is a measurement of the size distribution of individual particles in a soil sample. The classes are used in engineering and soil science (Gee and Bauder, 1986). In the USDA classification system (Soil Survey Division Staff,

1993), soil texture refers to the relative proportion of clay, silt and sand on a < 2 mm basis. The USDA classification scheme uses a textural triangle.

There is no standard rule of thumb to check the quality of the PSD data, however, a rule of thumb exists to assess the quality on clay content, the most reactive mineral content of soil. Having assessed the quality of clay content, the comments could then be given on the sand and silt values. The clay may or may not correspond to both the CEC and W_{15} values; if it does not correspond, this questions the quantity of sand and silt particles in these soils as well because sand + silt + clay all must sum up to 100%. The underestimation of clay is usually made due to poor dispersion of soil particles in the presence of gypsum and CaCO_3 and other flocculating agents, if they are not removed successfully; as a result, the values of sand and silt will also be changed, and this will further change the textural class of the soil.

The engineering data (percent passing through sieve # 4, 10, 40 and 200 mesh) if available could be useful in checking the quality of physical soil analysis. The > 2 mm weight % on whole soil basis is used to calculate the weight % of < 2 mm soil. In the engineering data sheet, the percent passing from a 10-mesh sieve (2 mm opening) can be used as weight % of < 2 mm soil as comparison. The values of < 2 mm soil obtained from the two sources are compared for quality assessment. If there is a difference between the two values measured at two different locations, this could question the quality of engineering soil classes (USCS and AASHTO) determined from the passing of 4, 10, 40 and 200 mesh sieves.

Engineering Properties

Atterberg Limits

These are the water content points at which wetted soil samples exhibit physical phase changes and, subsequently, very different behavior. It is a general term that encompasses liquid limit (LL), plastic limit (PL) and, in some cases, shrinkage limit.

Liquid Limit (LL) is the percent water content of soil at the arbitrary defined boundary between the liquid and plastic states. The LL is the water content at which the paste begins to flow as a liquid. The LL is reported as percent water on a < 0.4 mm bases (40 mesh). If it is not measured, it can be estimated by the following equation:

$$LL = 0.9 \times \% \text{ clay} + 10$$

$$LL = 2 \times W_{15} + 10$$

where LL = liquid limit; clay = % clay on a less than 2 mm basis; and W_{15} = water retention at 15-bar suction on a < 2 mm basis.

If the liquid limit is measured, the quality of the LL data assessment could be made comparing the measured value with those obtained from the above equations. Any deviation can be reported, and if the values are not consistent, samples may be reanalyzed or justification for inconsistency should be reported.

Plasticity Limit (PL) is the water content at which a soil paste can be deformed without breaking. Samples that do not deform without breaking at any water content are reported as non-plastic (NP).

Plasticity Index (PI) is the range of water content over which a soil behaves with plasticity. Numerically, it is the difference in the water content between the LL and the plastic limit (PL). The plasticity index (PI) is reported as percent water on a < 0.4 mm basis. If the PI is not measured, it can be estimated as follows:

i) When clay is < 15%

$$PI = \text{clay} \times 0.3$$

ii) When clay ranges between 15-35%

$$PI = \text{clay} \times 0.4$$

iii) When clay ranges between 35-55%

$$PI = \text{clay} - 21$$

iv) When clay is > 55%

$$PI = \text{clay} - 15$$

Based on the clay content of the samples, the PI can be estimated and compared with the measured values. If the differences are significant, and no justification is given, then the samples should be re-assessed for their accurate values.

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