2.3. Quality Control and Standardization Procedures

What follows in this section is a synthesis from the manual of Okalebo et al. (1993). **Quality control is an essential part of good laboratory practice**. During routine analyses, errors may gradually appear due to contamination, changes in reagent quality, environmental differences, operator error, and instrument calibration or failure. Maximum reproducibility and adequate accuracy of results are the important objectives.

Repeated measurement of an air-dried soil sample should provide consistent results when analyzed over time for most routine chemical procedures. The deviation of an observed value from its absolute "true" value results from either *systematic* or *random* errors. Once identified, systematic errors are more easily corrected than those which occur at random. Three precautions are essential for laboratory quality control and should be routinely included among the test samples. These precautions involve the use of **blanks**, **repeats**, and **internal references**, as elaborated below.

Whenever a new procedure is introduced to the laboratory, its **accuracy** should be evaluated and compared to the test already in use. Both methods should be compared for a homogeneous test sample using ten-fold replicates, with the standard deviation calculated for each set. This provides a measure of **precision**. Known amounts of reagent should be added to the homogeneous test sample, the procedures repeated, and the mean and standard then deviation calculated. The agreement between the increases in the values obtained to the known increase in test sample concentration provides a test of **accuracy**. For procedures in which the test material is known to interact with the added reagent, as with phosphorus-sorptive soils, this test can be conducted by reagent solutions.

Blanks

Blanks are reaction vessels that are subjected to identical procedures as the sample in a given batch which has no added test material. **Blanks allow correction for any background contamination introduced from reagents, filter papers or other systemic sources of error**. Provided the blank values are consistent, the mean value can be subtracted from the sample value. When blanks yield large values, this suggests excess extraneous contamination; in such cases, the entire batch analysis be repeated.

Repeats

At least 1 in 10 samples selected from the test materials and placed at random within the batch should be analyzed in duplicate. The choice of 1 in 10 is a suggested compromise between the ideal of analyzing all samples in duplicate, considering the time, effort and expense of doing so. Obviously, the analytical results for given pairs of duplicate repeats should closely resemble one another, in general, repeat values should fall within ±2.5 – 5.0 % of their mean, depending on the analysis in question; any greater discrepancy must be investigated. If repeat values are not consistent, the entire batch should be re-analyzed.

Internal References

Internal reference samples are necessary for each type of test material and analysis practiced within the laboratory. The internal sample should not be the same as the homogeneous material routinely used in the testing new methods and analytical technique. A sample obtained from a large, well-mixed and homogeneous composite bulk sample should be included in each batch analyzed. Variation from the mean as calculated over previous batches may be indicated as an error.

Analytical results for the internal reference may be plotted on a quality control chart to monitor the performance of the analyses over time. *Corrective action could be taken if a single value exceeds the ± 3 standard deviation limits or if two successive values exceed the ± 2 standard deviations*. Periodically, the critical limits could be re-assessed by re-calculation of the overall standard deviation of the internal reference sample as more data are accumulated.

Standardization of Methods

Results can only be validly compared to one another when they have been obtained using standardized methods. Collaboration between laboratories can be improved by exchanging reference materials and then comparing their results (Ryan and Garabet, 1994). Such materials are referred as "**External References**". An example of such standardization is the exchange network of ISRIC (International Soil Reference and Information Center) in Wageningen, The Netherlands, and operating international soil and plant analytical exchange programs.

Most external reference samples are costly, and their frequent use increases operating costs of the laboratory. Internal reference samples are usually much less expensive. Thus, if a relationship between external and internal reference samples can be firmly established, frequent use of internal reference sample, with occasional use of the external reference sample, can reduce costs, while still providing acceptable quality assurance.

Errors in Quantitative Analysis

In dealing with analysis, the concepts of accuracy and precision (See Figure.1) are important.

Accuracy: A measure of systematic error or the degree of agreement of an experimental value with the true or expected value of the quantity of concern. Accuracy of the value is important, but it is important to know when to use a given analytical method and to know its limitations.

$$\boldsymbol{Accuracy} = \frac{\boldsymbol{mean - true\ value}}{\boldsymbol{true\ value}}$$

$$\boldsymbol{Recovery}\ (\%) = \frac{\boldsymbol{measured\ value}}{\boldsymbol{Know\ value}} \times \mathbf{100}$$

Precision: A measure of reproducibility affected by random error. It is usually described by the standard deviation, standard error, or confidence interval.

$$\boldsymbol{RSD\ or\ CV}\ (\%) = \frac{\boldsymbol{S}}{\boldsymbol{mean}} \times \mathbf{100}$$

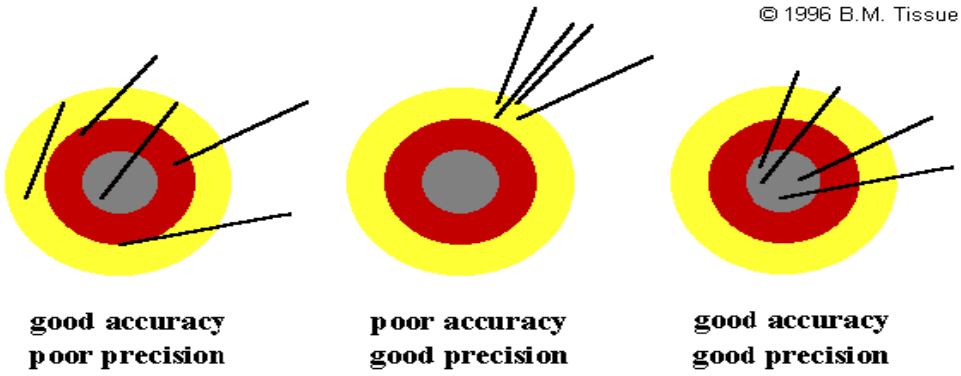

Figure. 1 Illustration of Accuracy and Precision