3. LABORATORY FACILITIES, QUALITY CONTROL, AND DATA HANDLING

3.1 Laboratory Organization

Soil and plant analyses are carried out by various institutions in the public or government sector as well as in the private domain. Laboratories are found in Ministries of Agriculture, National Research and Teaching Institutes, International Organizations, and in Commercial Companies. Analytical services should be closely linked to the Extension/Advisory services and should maintain a functional relationship with the universities, research stations, etc.

The kind of facility for such analyses depends on the type of institution it serves, the nature of the clientele, and the volume of samples to be analyzed. Nevertheless, all *laboratories,* regardless of the size, should be designed in a manner to facilitate *operational efficiency, minimize contamination,* and produce *reliable* and *repeatable* results.

Various publications deal with management considerations in the design and operation of soil testing (e.g., Walsh and Beaton, 1973). While the advantages of standardized laboratory designs are self-evident, many laboratories in the CWANA region have apparent deficiencies in this respect (Ryan, 2000; Ryan *et al*., 1999). All too often one sees soil samples stored or, worse still, ground in wet chemistry laboratories. Similarly, many laboratories are set up in a manner that inadvertently hinders efficient use of staff resources.

Soil, Plant, and Water analysis facilities should be located in the same building and be under one unified administration. The Soil-Plant Analysis Laboratory of **ICARDA's** Natural Resource Management Program was designed with these considerations in mind **(Fig. 3)**. The various components of the facility reflect a logical activity framework.

In *Soil Preparation Room* No. 1, large bulk samples, transported by truck, are received, dried, sieved, and stored in bulk bins. An inventory or catalogue of all soil samples is maintained. All samples are retained in this area for at least two years after analysis; bulk samples of special soil types are kept indefinitely. This facility is equipped with a large oven, freezer, soil grinder, exhaust hood, and a compressed air machine.

Small lots (~1 kg) of soil samples are taken to *Soil Preparation Room* No. 2 and weighed in appropriate containers for chemical analyses. The samples are then put in the *Soil Store*. Batches of weighed samples are wheeled in a trolley to the adjacent laboratory for analysis. A separate *Kjeldahl Room* exists for plant Kjeldahl analysis. Soil extracts, where necessary, are carried to a small *Instrument Room* (atomic absorption spectrophotometer, flow injection analyzer, computer, etc.) for analysis. Where large numbers of samples are involved, and where a delay would induce bacterial changes in soil material, samples are temporarily held in the *Freezer Room*.

While no two laboratories are ever the same or have the same complement of equipment, the details presented for **ICARDA's** laboratory **(Fig. 3)** will, hopefully, serve as a general guideline for laboratory arrangement and the type of equipment needed for routine service-oriented operations.

1. Oven
2,3. Freezers
4. Soil Grinder & Exhaust Hood
5. Compressed Air
6. Sub Sampling Bench
7. Soil Grinder
8. Plant Sample Drawers
9. Vacuum Filtering System
10. Nitrogen distillation &Titration unit
11. Conductivity Meter
12. Flame Photometer
13. pH Meter
14. Expandable Ion Analyzer
15. Spectrophotometer
16. Automatic Dispensor
17,18. Nitrogen distillation & Titration unit
19. Cooled Incubator
20. Refrigerator
21. P. C. (Computer)
22. Flow Injection Analyzer
23. Atomic Absorption Spectrophotometer.
24. Muffle Furnace
25. Kjeldahl unit
26,27,28. Dispensers
29. Titration Bench
30,31. Freezers
32,33. Refrigerators

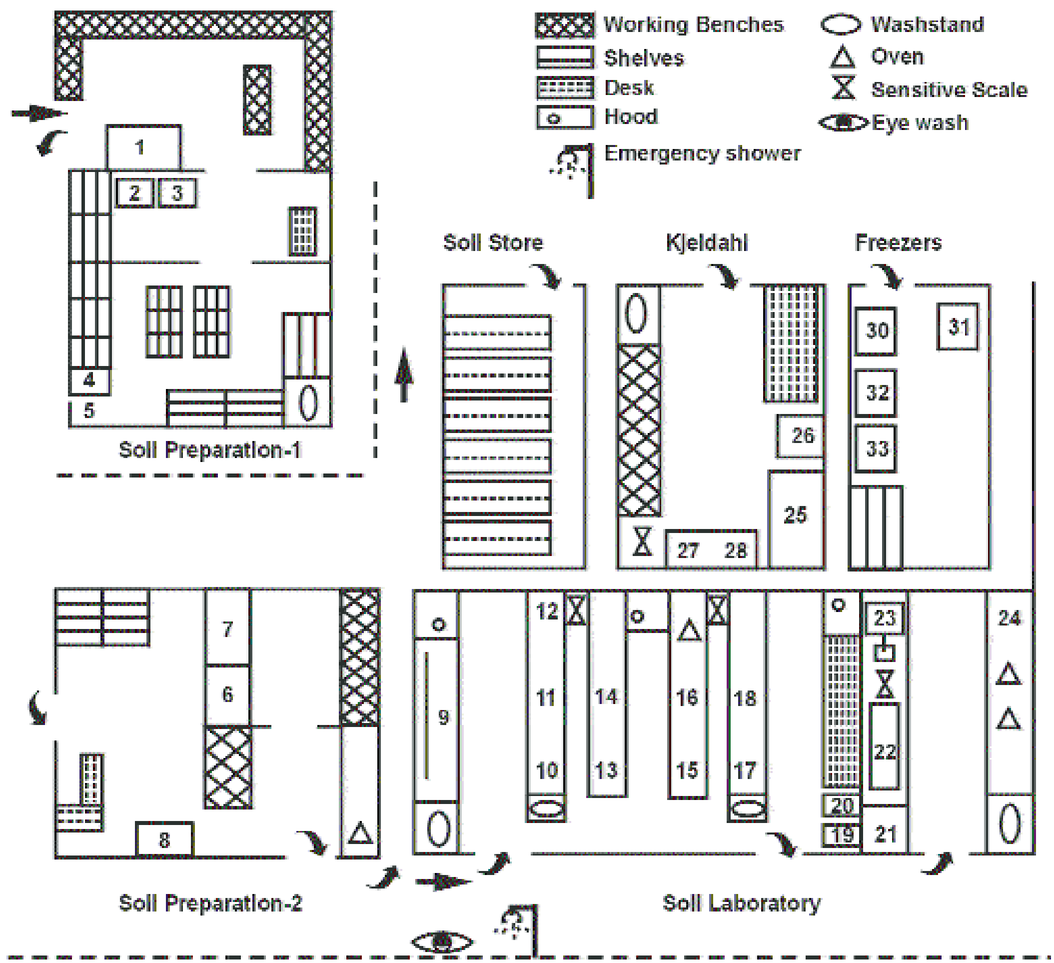

Fig. 3. Schematic layout of ICARDA's Soil-Plant Analysis Laboratory facilities.

3.2 Laboratory Safety

As with any place of work, safety is an important consideration in soil-plant analysis laboratories, and one that is frequently overlooked. Safety is in the interest of the employees who work there and the organizations that operate the laboratories.

All staff, irrespective of grade, technical skill or employment status should be briefed on all aspects of safety upon commencement of work. Periodic reminders of such regulations should be given to encourage familiarity with respect to regulations.

While rules pertaining to safety can be extensive, we have endeavored to concisely list the more important ones within different categories of concerns. These have been adapted from laboratory safety guides developed by Kalra and Maynard (1991) and Okalebo *et al*. (1993).

General Attitude

1. Develop a *positive* attitude towards laboratory safety.
2. Observe normal laboratory safety practices.
3. Maintain a safe and clean work environment.
4. Avoid working alone.

Instrument Operation

5. Follow the safety precautions provided by the manufacturer when operating instruments.
6. Monitor instruments while they are operating.
7. Atomic Absorption Spectrophotometer must be vented to the atmosphere. Ensure that the drain trap is filled with water prior to igniting the burner.
8. Never open a centrifuge cover until machine has completely stopped.

Accidents

9. Learn what to do in case of emergencies (e.g., fire, chemical spill, etc.). Fire fighting equipment must be readily accessible in the event of fire. Periodic maintenance inspections must be conducted.
10. Learn emergency **First Aid. First Aid** supplies are a necessity and laboratory staff should be well trained in their use. Replacement of expended supplies must take place in a timely fashion.

11. Seek medical attention immediately if affected by chemicals, and use **First Aid** until medical aid is available.
12. Access to eye-wash fountains and safety showers must not be locked. Fountains and showers should be checked periodically for proper operation.

Chemicals

13. Use fume hoods when handling concentrated acids, bases or other hazardous chemicals.
14. Do not pipette by mouth; *always* use a suction bulb.
15. When diluting, always add acid to water, *not* water to acid.
16. Many metals/salts are *extremely toxic* and may be *fatal* if swallowed. Wash hands thoroughly after handling such salts. Chemical spills should be cleaned promptly and all waste bins emptied regularly.
17. All reagent bottles should be clearly labeled and must include information on any particular hazard. This applies particularly to poisonous, corrosive, and inflammable substances.

Furnaces, Ovens, Hot Plates

18. Use forceps, tongs, or heat-resistant gloves to remove containers from hot plates, ovens or muffle furnaces.

Handling Gas

19. Cylinders of compressed gases should be secured at all times. A central gas facility is preferred.

Maintenance

20. All electrical, plumbing, and instrument maintenance work should be done by qualified personnel. Fume hoods should be checked routinely.
21. As most equipments operate on low wattage, an Uninterruptible Power Supply **(UPS)** provides stable power and allows the completion of any batch measurement in the event of power outage.

Eating and Drinking

22. Do *not* eat, drink, or smoke in the laboratory. This is essential both for reasons of health and to reduce contamination. Specific areas should be designated for staff breaks.
23. Do *not* use laboratory glassware for eating or drinking.
24. Do not store food in the laboratory.

Protective Equipment

25. Use personnel safety equipment as follows:
    - **Body Protection:**

      Use laboratory coat and chemical-resistant apron.
    - **Hand Protection**

      Use gloves, particularly when handling concentrated acids, bases, and other hazardous chemicals.
    - **Dust Mask**

      Essentially needed when grinding soil, plant samples, etc.
    - **Eye Protection**

      Use safety glasses with side shields. Persons wearing contact lenses should always wear safety glasses in the laboratory. Make sure that your colleagues know that you wear contact lenses. Contact lenses should never be worn around corrosives.
    - **Full-Face Shield**

      Wear face shields over safety glasses in experiments involving corrosive chemicals.
    - **Foot Protection**

      Proper footwear should be used; sandals should *not* be worn in the laboratory

Waste Disposal

26. Liquid wastes should be poured carefully down a sink with sufficient water to dilute and flush it away. Keep in mind that local ordinances often prohibit the disposal of specific substances through the public sewerage system.
27. Dispose off chipped or broken glassware in specially marked containers.

Continuing Education

- Display in a prominent place posters on **"Laboratory Safety"** which pictorially describe various phases of laboratory activities.
- Similarly, posters depicting **First Aid** after laboratories accidents should be prominently displayed. Such posters are *not* for ornamentation; they are for the *protection of laboratory personnel*, who should be thoroughly conversant with all procedures and eventualities.
- If the laboratory is a part of a large institution, the laboratory staff should know the **Safety Officer** or person responsible for safety. If it is a small operation, one laboratory staff member should be responsible for safety.

Contamination

The most insidious enemy in any laboratory is *contamination* and, therefore, its sources must be identified and eliminated. *Some common sources of contamination are:*

- External dusts blown from the surrounding environment;
- Internal dust resulting from cleaning operations;
- Cross-contamination derived from handling many samples at the same time (e.g., handling plant and soil samples together);
- Failure to store volatile reagents well away from the samples;
- Washing materials, particularly soap powder; and
- Smoking in the laboratory.

3.3 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*. Then a known amount of reagent should be added to the homogeneous test sample, the procedures repeated, and the *mean* and *standard deviation* calculated. The agreement between the increase 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-sorption soils, this test can be conducted by reagent solutions.

1. Blanks

Blanks are reaction vessels that are subjected to identical procedures as the sample in a given batch which have 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.

2. 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

3. 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.

4. Standardization of Methods

Results can only be validly compared to one another when these 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, operating an international soil and plant analytical exchange programs.

Most external reference samples are costly, and their frequent use would increase 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.

3.4 Data Processing

A considerable amount of information is generated in any soil-plant analysis laboratory. In order to economically justify the existence of a laboratory, it is necessary to have a record of the number of samples analyzed and the types of analyses performed. With the advent of the computer, such storage is easy and retrieval is greatly facilitated. Computer processing offers the advantage of:

1. Easier manipulation of large data sets;
2. Reduced errors in calculation of recommendations;
3. Preparation of reports;
4. Automated invoicing and addressing; and
5. Ready access to historical data for preparation of soil test summaries.

The degree to which laboratories should be computerized depends on sample volume, location, and user services offered. In general, laboratories with a large volume of samples, and which offer a range of analyses have more need for computer sophistication and automation than laboratories with a small sample turnover.

In order to facilitate data processing, standard information sheets are required. These vary from laboratory to laboratory, but usually include details of analyses required for the sample and information on the crop to be grown, the soil type and previous cropping history, particularly with respect to fertilization. Such information enables one to answer questions on the extent of nutrient deficiency in any area from which the samples were obtained, and how fertility levels change over the years.

Computer programs are increasingly used to interpret soil test data and making fertilizer recommendations. Several such programs do exist. Standardized report forms for making fertilizer recommendations combine inputs of soil test data together with other soil and crop information. In order to do this, the tests used (soil NO_3^--N, available P, etc.) must be calibrated with field crop response.

With a relatively large output of analyses associated with a large number of on-station and on-farm research trials, analytical results from **ICARDA's** laboratory are stored in a computer program to generate fertilizer recommendations based on soil test values. Where soil maps and rainfall data are available, the accumulated soil test values of known locations can help establish relationships with soil type, region, climatic zone, etc.