Technical Assistance
HYDROLOGY PROJECT

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Training module # WQ I-3

The need for good laboratory practice

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with
HALCROW, TAHAL, CES, ORG & JPS

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1 Module context

This module discusses the basic operations and practices that should be followed routinely in all chemical laboratories. No prior training in other modules is necessary to complete this module successfully.

While designing a training course, the relationship between this module and the others, would be maintained by keeping them close together in the syllabus and place them in a logical sequence. The actual selection of the topics and the depth of training would, of course, depend on the training needs of the participants, i.e. their knowledge level and skills performance upon the start of the course.

2 Module profile

Title	:	The need for good laboratory practice
Target group	:	As per training need
Duration	:	60 min
Objectives	:	After the training the participants will be able to*:* • Apply the adopted standard practices in laboratory operations
Key concepts	:	• Chemicals, distilled water, glassware • Maintenance of equipment • Laboratory safety • Data recording
Training methods	:	Lectures and discussions
Training tools required	:	Overhead projector, flipchart
Handouts	:	As provided in this module
Further reading and references	:	• Analytical Chemistry: An introduction, D. A. Skoog and D. M. West/1986. Saunders College Publishing • Standard Methods: for the Examination of Water and Wastewater, APHA, AWWA, WEF/1995. APHA Publication

3 Session plan

No	Activities	Time	Tools
1	***Preparations*** • Trainer may wish to actually demonstrate a few practices		
2	***Introduction***: • Ask participants to name some GLP. • Introduce the material to be covered in the module	3 min	flipchart OHS
3	***Chemicals & reagents*** • Discuss importance of purity of chemicals. • Describe various grades of chemicals available in the market and their uses. • Discuss the rules to be followed in handling reagents.	10 min	OHS
4	***Cleaning of glassware*** • Describe the recommended practice for cleaning of glassware and its importance.	5 min	OHS
5	***Distilled water*** • Name different types of water purifying systems and note the quality of their product. • Emphasise the necessity of proper maintenance.	5 min	OHS
6	***Weighing*** • Discuss the difference in mass and weight, accuracy of balances and need for proper maintenance. • Discuss the procedures to be followed in weighing.	10 min	OHS OHS
7	***Recording of data*** • Emphasise need for unambiguous records and procedure to be followed, text section 8.5	3 min	OHS
8	***Equipment maintenance*** • Discuss importance of preventive maintenance and type of entries to be entered in an instrument logbook.	3 min	OHS
9	***Sample collection*** • Describe the need for interaction between the laboratory chemist and field staff.	3 min	OHS
10	***Laboratory safety*** • Describe the sources of hazard and precautions to be followed.	10 min	OHS
13	***AQC*** • Discuss objectives of AQC programmes.	5 min	OHS
14	***Wrap up*** • Conclude by asking participants if something important has been missed.	3 min	OHS

4 Overhead/flipchart masters

OHS format guidelines

Type of text	Style	Setting
Headings:	OHS-Title	Arial 30-36, Bold with bottom border line (not: underline)
Text:	OHS-lev1 OHS-lev2	Arial 26, Arial 24, with indent maximum two levels only
Case:		Sentence case. Avoid full text in UPPERCASE.
Italics:		Use occasionally and in a consistent way
Listings:	OHS-lev1 OHS-lev1-Numbered	Big bullets. Numbers for definite series of steps. Avoid roman numbers and letters.
Colours:		None, as these get lost in photocopying and some colours do not reproduce at all.
Formulas/ Equations	OHS-Equation	Use of a table will ease alignment over more lines (rows and columns) Use equation editor for advanced formatting only

Good laboratory practice

Chemicals

Glassware cleaning

Distilled water

Weighing

Recording of data

Equipment maintenance

Sample collection

Laboratory safety

Analytical quality control

1. Chemicals: grades

- Technical reagent
- Laboratory or analytical reagent
- Primary standard reagent
- Special purpose reagent

1. Chemicals: handling reagents

- Use small packing
- Avoid contamination
- Storage requirements
- Shelf life

2. Glassware cleaning

- Cleaning agents
  - detergent solution
  - chromic acid
  - other acids
  - distilled water
- Break in water film indicates dirt or oil
- Volume calibration is affected if glassware is dirty.

3. Distilled water

- Types of distilled water

Type	ECμmho/cm	Source
I	< 0.1	ion exchange
II	< 1	double distilled, glass
III	<10	single distilled, steel

- Cleaning of stills
- Regeneration of exchange resin

4. Weighing

- Fundamental requirement for all analyses
- mass and weight are synonymous
- accuracy of balances
- care in operation
    - beam arresting mechanism
    - protection from corrosion
    - cleaning after every use
    - vibration free table
    - use tongs

4. Weighing

- Solids are dried in oven
- Constant weight
- Cooled in desiccator before weighing
- Desiccants
    - calcium chloride
    - calcium sulphate
    - other

5. Recording of data

- Use bound notebooks
- Number pages
- Date & label all entries
- Do not crowd entries
- Do not delete, over write or erase, instead cross out
- Do not remove pages from notebook

6. Equipment maintenance

- Logbook for each major equipment
- Maintenance schedule
- Breakdown maintenance
- Supply of spares & consumables
- Future planning

7. Sample collection

- Interact with field staff
- Advise on procedures
- Occasional field visits
- Training of field staff

8. Laboratory safety: sources of hazards

- Corrosive and poisonous chemicals
- Broken glass
- Explosion
- Fire
- Electric shock

8. Laboratory safety: precautions

- Locate eye fountain, emergency shower, fire extinguisher
- Wear eye protection
- Avoid skin contact with chemicals, wash immediately
- Do not store food, smoke, drink or eat in the work area
- Use suction bulbs
- Take care in working with glass
- Use fume hoods
- Dispose waste appropriately

9. Analytical quality control

- Internal mechanism for checking performance
- Gives warning of errors, random or systematic
- Practised by certified laboratories

Find out how to start AQC program in your laboratory

5 Evaluation

6 Handouts

Good laboratory practice

Chemicals
Glassware cleaning
Distilled water
Weighing
Recording of data
Equipment maintenance
Sample collection
Laboratory safety
Analytical quality control

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Find out how to start AQC program in your laboratory

Add copy of Main text in chapter 8, for all participants.

7 Additional handouts

These handouts are distributed during delivery and contain test questions, answers to questions, special worksheets, optional information, and other matters you would not like to be seen in the regular handouts.

It is a good practice to pre-punch these additional handouts, so the participants can easily insert them in the main handout folder.

8 Main text

The need for good laboratory practice

A number of laboratory operations and precautions related to analysis of water quality parameters must be routinely performed in a laboratory to obtain reliable information. These practices are termed good laboratory practices. Some analyses that may measure extremely small level of contaminants or use advanced level instrumentation would require special precautions. Practices that are to be followed routinely and are basic in nature to all determinations are described here.

1. Chemicals and reagents

Purity of reagents has an important bearing upon the accuracy that can be attained in an analysis. Commercially available chemicals are routinely classified as, technical grade, laboratory or analytical reagent grade, primary-standard grade and special purpose reagents.

Technical grade reagents in general are not used in a laboratory. Where bulk quantities may be required or when purity is not of major concern, such as preparation of chromic acid cleaning solution, technical grade reagents may be used.

Routine analyses in a water testing laboratory may be performed mostly using laboratory or analytical reagent grade chemicals. Where primary standards are to be made, primary standard grade reagent should be used.

Special purpose reagents are required for analyses in which micro-level contaminants are measured, such as atomic absorption spectroscopy and gas chromatography.

The following rules should always be followed while handling reagents and chemicals:

1. As far as possible, use the smallest packing of chemical that would supply the desired quantity.
2. Replace the cap or stopper of the bottle immediately after taking out your requirement.
3. Stoppers of reagent bottles should never be placed on the desktop.
4. Do not insert spatulas or pipettes in reagent bottles. Takes out a slightly excess amount in another container from where use the required quantity.
5. Never return any excess chemical or reagent to a bottle.
6. Some reagents require special storage conditions, such as dark colour bottles for light sensitive chemicals or low temperature for solvents and reagents subject to microbial degradation.
7. Do not use a reagent after the recommended shelf life.
8. Dry solid chemicals for making solutions, in a suitable container, as directed in the standard analytical procedure for the determination.

2. Cleaning of glassware

Volume calibrations are blazed on clean volumetric equipment. Cleanliness of volumetric glassware is therefore particularly important if callibration is to have any meaning. Only clean glass surfaces will support a uniform film of liquid; the presence of dirt or oil will tend to cause breaks in this film. The existence of breaks is a certain indication of an unclean surface. A brief soaking in warm detergent is usually sufficient to remove the grease and dirt responsible for causing the water breaks.

Where detergent is not effective, rinse glassware, except that used for chromium and manganese analysis, with a cleaning mixture made by adding 1L of conc. H_2SO_4 slowly with stirring, to 35 mL saturated sodium dichromate solution. Rinse with other concentrated acids to remove inorganic matter.

Use detergents or conc. HCl for cleaning hard rubber and plastic bottles.

After the glassware and bottles have been cleaned, rinse thoroughly with reagent water.

3. Distilled or reagent water

Laboratories are provided with stainless steel water distillation stills. Distilled water obtained from such stills is of adequate purity for making reagent solutions for routine analyses carried out in water testing laboratories and cleaning of glassware. However, in case of analyses for micro-level contaminants better quality distilled water may be needed.

Distilled water may be classified on the basis of its electrical conductivity (EC) as follows:

Type	EC, μmho/cm
I	< 0.1
II	< 1
III	< 10

Ion exchange columns and double distillation, all glass water stills are used to obtain type I and II distilled water, respectively.

In the operation of water stills care should be taken to run the condenser cooling water whenever the still is in use. All stills are prone to scaling and accumulation of precipitated salts, which if not attended to results in carry over of salts in distillate and will ultimately lead to overheating and damage the heating elements. The scales should be periodically removed by dissolving in dilute solution of HCl.

In the case of ion exchange columns a record should be kept of the volume of the product water and its EC value. The resin should be regenerated as per the supplier's instruction when the EC value exceeds the limit.

4. Measurement of mass or weighing

Accurately measuring mass of substances is a fundamental requirement for almost all types of analyses. In the laboratory the mass is determined by comparing the weight of an object with the weight of a set of standard masses using a balance. Because acceleration due to gravity affects both the known and unknown to the same extent, equality of weight indicates

equality of mass. The terminological distinction between mass and weight is, therefore, seldom made.
Analytical balances, with an accuracy of ±0.1 mg, are commonly used to determine the weight. For analyses for micro-level contaminants balances with a precision of ±0.01 may be used. It is of paramount importance that balances are used and maintained with great care. To avoid damage or minimize wear on the balance and obtain accurate weights adhere to the following rules:

1. Be certain that the arresting mechanism of the beam is engaged whenever the loading on the balance is being changed and when the balance is not in use.
2. Centre the load on the pan insofar as possible.
3. Protect the balance from corrosion. Only non-reactive glass, plastic or metal objects should be placed directly on the pan. In case of volatile or corrosive substances, take special precautions of sealing it in a weighed glass ampoule.
4. Do not attempt to adjust or repair the balance if you are not trained to do so.
5. Keep the balance and its case scrupulously clean. A camel's hair brush is useful for cleaning any spilled material or dust.
6. Do not weigh an object that has been heated until it has returned to the room temperature.
7. Do not touch a dried object with bare hands: use tongs to prevent the uptake of moisture.
8. Always place the balance on a vibration free platform.

The drying of a chemical or a container to constant weight is the process in which the object is first heated at an appropriate temperature, ordinarily for an hour or more, following which it is cooled and weighed.

Solids are conveniently dried and stored in weighing bottles of glass or plastic. Oven drying, usually at 105 to 110 ^{o}C is the commonest way of removing the absorbed moisture. The dried objects are stored in a desiccator while cooling. The base section of the desiccator contains a quantity of a chemical drying agent, such as anhydrous calcium chloride, calcium sulphate, or anhydrous magnesium perchlorate.

5. Recording of data

A laboratory notebook is needed to record measurements and observations concerning an analysis. The notebook itself should be permanently bound with a hard cover. The pages should be consecutively numbered. The first few pages should be reserved for a table of contents which should be kept up to date.

1. All data should be directly entered into the notebook.
2. Entries should be labeled. Entries should not be crowded.
3. Each notebook page should be dated as it is used.
4. An erroneous entry should never be erased, obliterated or written over. Instead it should be crossed out with a single horizontal line and the correct entry should be located adjacent to it.
5. Pages should never be removed from the notebook. It is sufficient to draw a single line diagonally across a page that is to be disregarded.

6. Maintenance of equipment

A chemical laboratory is provided with a variety of equipment ranging from simple heating devices to more complicated instrumentation. Their purpose, maintenance and care aspects are discussed in other modules. It is a good practice to keep a separate logbook for each major equipment where details of their use, maintenance schedule, breakdowns and repairs, accessories, supply of consumables, etc., are carefully entered. Such a record will help in properly maintaining the instrument and planning for future.

7. Sample collection and preservation

All laboratories interact with field staff who carries out sampling, conduct site analyses and transports samples. The staff of the chemical laboratory must advise the field staff regarding procedures for site analyses and method of sample collection. The laboratory staff should also specify preservation technique of samples for different analyses to be carried out in the laboratory and supply reagents required for preservation. The laboratory staff should make it a practice to periodically visit the sampling sites, observe the procedures being carried out and advise as necessary.

Sampling procedures, different types of samplers, preservation, etc., are described in the modules on surface and groundwater sampling procedures.

8. Laboratory safety

All laboratory employees must make every effort to adhere to certain basic safety rules to protect themselves and their fellow workers. The sources of hazard in a laboratory are corrosive and poisonous chemicals, broken glass, explosion, fire and electrical shock. Some common rules are given below. In case a health and safety programme has been developed for your laboratory, you should always follow it.

1. Learn the locations of eye fountain, emergency shower, fire blanket and fire extinguisher.
2. Eye protection must be worn at all times.
3. In handling all chemicals, avoid contact with skin. In the event of such contact, immediately wash the affected area with copious amounts of water.
4. Avoid working alone in a laboratory if the procedures to be conducted are hazardous.
5. Do not drink, eat or smoke in areas where laboratory chemicals are present. Do not drink from laboratory glassware.
6. Do not store food or beverages in storage areas and refigerators that are used for laboratory operations.
7. Always use a bulb to draw chemicals in a pipette. Never use the mouth to provide suction.
8. Be extremely tentative in touching the objects that have been heated.
9. Always fire polish the ends of freshly cut glass tubing. Never attempt to force glass tubing through a hole in the stopper. Instead, make sure that both the tubing and the hole are thoroughly wet with soapy water and protect hands with towel or heavy gloves.
10. Use fume hoods where toxic or noxious gases or fumes are likely to be evolved.
11. Use care in testing for odours; use the hand to waft vapours above containers towards nose.

12. In some locations it may not be permissible to flush heavy metals or poisonous substances down the drain. In case of such restrictions, alternative arrangements are required.

9. Analytical quality control

The subject of analytical quality control is discussed in other modules in detail. Briefly, it is an internal mechanism for checking your own performance. It indicates human errors in routine laboratory work and protects procedures from errors that may creep in due to various reasons. It is practiced by all responsible chemists and is a requirement for certification of laboratories. It is strongly recommended that laboratories conduct such a programme on routine basis.