



**NATIONAL OPEN UNIVERSITY OF NIGERIA**

**FACULTY OF HEALTH SCIENCES**

**DEPARTMENT OF ENVIRONMENTAL HEALTH SCIENCES**

**COURSE CODE: EHS 403**



**COURSE TITLE: ENVIRONMENTAL HEALTH LABORATORY**

**COURSE  
GUIDE**

**EHS 403: ENVIRONMENTAL HEALTH LABORATORY 3 CREDIT UNIT**

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National Open University of Nigeria 2019

First Printed 2019

ISBN:

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Printed by.....

For

National Open University of Nigeria

Headquarters  
91 Cadastral Zone  
University Village  
Nnamdi Azikiwe Express Way  
Jabi, Abuja

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**COURSE  
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## **Introduction**

This course *Environmental Health Laboratory* deals with the function of the laboratory in Environmental Science. Environmental Scientists monitor the effect of hazardous substances in the environment, they carry out analysis on samples taken from the environment in Environmental laboratories to enable them determine their effects on both plants and animals including humans. Environmental health laboratory enables the evaluation of the possible impact of chemical substances on human health and the environment using the latest scientific knowledge as well as state of the art technologies. Scientists use Environmental Health laboratory to rapidly assess public drinking water, swimming pools, ambient air etc. to determine levels of contamination and possible risk to the health. Environmental Health Laboratory provides analytical and technical information in support of state and national environmental health programs. Such programs include but are not limited to those associated with the surveillance of air, food, seafood, soil, water, and zoonotic diseases from domestic and wild animals.

## **What you will learn in this course**

In this course, you have the course units and a course guide. The course guide will tell you what the course is all about. It is general overview of the course materials you will be using and how to use those materials. It also helps you to allocate the appropriate time to each unit so that you can successfully complete the course within the stipulated time limit.

The course guide also helps you to know how to go about your Tutor-Marked Assignment which will form part of your overall assessment at the end of the course. Also, there will be regular tutorial classes that are related to this course, where you can interact with your facilitator and other students. Please, I encourage you to attend these tutorial classes.

## **Course Aim**

The aim of this course, Environmental Health Laboratory is to provide the required knowledge of the different ways of assessing pollution in the environment, thus providing information that is

useful for environmental management programs. In this course you will be introduced Environmental Health Laboratory and the different tools available in Environmental Science.

### **Course Objectives**

To achieve the aim set above, there are objectives. Each unit has a set of objectives presented at the beginning of the unit. These objectives will guide you on what to concentrate / focus on while studying the unit. Please read the objective before studying the unit and during your study to check your progress.

The Comprehensive Objectives of the Course are given below. By the end of the course/after going through this course, it is expected that at the end of this course, students should be able to:

- Understand the requirement for general laboratory construction
- Discuss the importance and functions of Environmental Health Laboratory
- Know how to prepare and store reagents
- Understand the principles and uses of some Environmental Health Laboratory instruments
- Explain the importance of quality assessment and quality control
- Explain the importance of laboratory log book and record keeping in the laboratory

### **Working through this course**

To successfully complete this course, you are required to read each study unit, read the textbooks materials provided by the National Open University.

Reading the referenced materials can also be of great assistance.

Each unit has self-assessment exercises which you are advised to do and at certain periods during the course you will be required to submit your assignment for the purpose of assessment.

There will be a final examination at the end of the course. The course should take you about 17 weeks to complete.

This course guide will provide you with all the components of the course how to go about studying and hour you should allocate your time to each unit so as to finish on time and successfully.

## **The Course Materials**

The main components of the course are:

- The Study Guide
- Study Units
- Reference / Further Readings
- Assignments
- Presentation Schedule

## **Study Unit**

The study units in this course are given below:

### **MODULE 1: INTRODUCTION TO LABORATORY SCIENCE**

Unit 1: Introduction to Laboratory Science

Unit 2: Establishment of Environmental Laboratories

Unit 3: Importance and Functions of Environmental Health Laboratory

Unit 4: Laboratory Record Keeping

### **MODULE 2: GENERAL LABORATORY CONSTRUCTION**

Unit 1: General Requirements for Laboratories Construction

Unit 2: Laboratory Ventilation Systems

Unit 3: Requirements for Environmental Health Laboratory

Unit 4: Safe Laboratory Practices

### **MODULE 3: PREPARATION AND STORAGE OF REAGENTS**

Unit 1: Preparation of Reagents

Unit 2: Reagent Preparation from Stock Standard Solution

Unit 3: Converting Between Concentration Units

Unit 4: Storage of Laboratory Reagents

## **MODULE 4: INSTRUMENTATION**

Unit 1: Instrumentation in Environmental Science

Unit 2: Noise, Sound and Radiation Measuring Instruments

Unit 3: Polymerase Chain Reaction (PCR)

Unit 4: Cryostat

## **MODULE 5: QUALITY ASSESSMENT AND QUALITY CONTROL PRACTICE**

Unit 1: Quality Assurance

Unit 2: Quality Control

Unit 3: Analytical Quality Control Procedures

Unit 4: Sample Collection and Handling

There are activities related to the lecture in each unit which will help your progress and comprehension of the unit. You are required to work on these exercises which together with the TMAs will enable you to achieve the objectives of each unit.

### **Assignment File**

There are two types of assessments in this course. First are the Tutor-Marked Assessments (TMAs); second is the written examination. In solving the questions in the assignments, you are expected to apply the information, knowledge and experience acquired during the course. The assignments must be submitted to your facilitator for formal assessment in accordance with prescribed deadlines stated in the assignment file.

The work you submit to your facilitator for assessment accounts for 30 percent of your total course mark. At the end of the course, you will be required to sit for a final examination of 3 hours duration at your study center. This final examination will account for 70 % of your total course mark.



## **Presentation Schedule**

There is a time-table prepared for the early and timely completion and submissions of your TMAs as well as attending the tutorial classes. You are required to submit all your assignments by the stipulated time and date. Avoid falling behind the schedule time.

## **Assessment**

There are three aspects to the assessment of this course.

The first one is the self-assessment exercises. The second is the tutor marked assignments and the third is the written examination or the examination to be taken at the end of the course.

Do the exercises or activities in the unit by applying the information and knowledge you acquired during the course. The tutor-marked assignments must be submitted to your facilitator for formal assessment in accordance with the deadlines stated in the presentation schedule and the assignment file.

The work submitted to your tutor for assessment will count for 30% of your total course work.

At the end of this course, you have to sit for a final or end of course examination of about a three hour duration which will count for 70% of your total course mark.

## **Tutor-Marked Assignment**

This is the continuous assessment component of this course and it accounts for 30% of the total score. You will be given four (4) TMAs by your facilitator to answer. Three of which must be answered before you are allowed to sit for the end of course examination.

These answered assignments are to be returned to your facilitator.

You're expected to complete the assignments by using the information and material in your readings references and study units.

Reading and researching into you references will give you a wider via point and give you a deeper understanding of the subject.

1. Make sure that each assignment reaches your facilitator on or before the deadline given in the presentation schedule and assignment file. If for any reason you are not able to complete your assignment, make sure you contact your facilitator before the assignment is due to discuss the possibility of an extension. Request for extension will not be granted after the due date unless there in exceptional circumstances.

2. Make sure you revise the whole course content before sitting or the examination. The self-assessment activities and TMAs will be useful for this purposes and if you have any comment please do before the examination. The end of course examination covers information from all parts of the course.

### Course Marking Scheme

Assignment	Marks
Assignments 1 – 4	Four assignments, best three marks of the four count at 10% each–30% of course marks.
End of course examination	70% of overall course marks
Total	100% of course materials.

**Table 2: Course Organization**

Unit	Title of Work	Weeks activity	Assessment (End of Unit)
1	Introduction to Laboratory Science	Week 1	Assignment 1
2	Establishment of Environmental Laboratories	Week 2	Assignment 2
3	Importance and Functions of Environmental Health Laboratory, Laboratory Record Keeping.	Week 3	Assignment 3
4	General Requirements for Laboratories Construction, Laboratory Ventilation Systems.	Week 4	Assignment 4
5	Requirements for Environmental Health Laboratory, Safe Laboratory Practices.	Week 5	Assignment 5
6	Preparation of Reagents from Stock Standard Solutions.	Week 6	Assignment 6
7	Converting Between Concentration Units, Storage of Laboratory Reagents.	Week 7	Assignment 7
8	Instrumentation in Environmental Science	Week 8	Assignment 8

9	Noise, Sound and Radiation Measuring Instruments	Week 9	Assignment 9
10	Polymerase Chain Reaction (PCR)	Week10	Assignment10
11	Cryostat	Week11	Assignment 11
12	Quality Assurance and Quality Control	Week12	Assignment 12
13	Analytical Quality Control Procedures	Week13	Assignment 13
14	Sample Collection and Handling	Week14	Assignment 14

### **How to get the most out of this Course**

In distance learning, the study units replace the university lecturer. This is one of the huge advantages of distance learning mode; you can read and work through specially designed study materials at your own pace and at a time and place that suit you best. Think of it as reading from the teacher, the study guide tells you what to read, when to read and the relevant texts to consult. You are provided exercises at appropriate points, just as a lecturer might give you an in-class exercise. Each of the study units follows a common format. The first item is an introduction to the subject matter of the unit and how a particular unit is integrated with the other units and the course as a whole. Next to this is a set of learning objectives. These learning objectives are meant to guide your studies. The moment a unit is finished, you must go back and check whether you have achieved the objectives. If this is made a habit, then you will significantly improve your chances of passing the course. The main body of the units also guides you through the required readings from other sources. This will usually be either from a set book or from other sources.

Self-assessment exercises are provided throughout the unit, to aid personal studies and answers are provided at the end of the unit. Working through these self-tests will help you to achieve the objectives of the unit and also prepare you for tutor marked assignments and examinations. You should attempt each self-test as you encounter them in the units.

### **The following are practical strategies for working through this course**

1. Read the Course Guide thoroughly.
2. Organize a study schedule. Refer to the course overview for more details. Note the time you are expected to spend on each unit and how the assignment relates to the units. Important details,

e.g. details of your tutorials and the date of the first day of the semester are available. You need to gather together all these information in one place such as a diary, a wall chart calendar or an organizer. Whatever method you choose, you should decide on and write in your own dates for working on each unit.

3. Once you have created your own study schedule, do everything you can to stick to it. The major reason that students fail is that they get behind with their course works. If you get into difficulties with your schedule, please let your tutor know before it is too late for help.

4. Turn to Unit 1 and read the introduction and the objectives for the unit.

5. Assemble the study materials. Information about what you need for a unit is given in the table of contents at the beginning of each unit. You will almost always need both the study unit you are working on and one of the materials recommended for further readings, on your desk at the same time.

6. Work through the unit, the content of the unit itself has been arranged to provide a sequence for you to follow. As you work through the unit, you will be encouraged to read from your set books.

7. Keep in mind that you will learn a lot by doing all your assignments carefully. They have been designed to help you meet the objectives of the course and will help you pass the examination.

8. Review the objectives of each study unit to confirm that you have achieved them. If you are not certain about any of the objectives, review the study material and consult your tutor.

9. When you are confident that you have achieved a unit's objectives, you can start on the next unit. Proceed unit by unit through the course and try to pace your study so that you can keep yourself on schedule.

10. When you have submitted an assignment to your tutor for marking, do not wait for its return before starting on the next unit. Keep to your schedule. When the assignment is returned, pay particular attention to your tutor's comments, both on the tutor-marked assignment form and also that written on the assignment. Consult your tutor as soon as possible if you have any questions or problems.

11. After completing the last unit, review the course and prepare yourself for the final examination. Check that you have achieved the unit objectives (listed at the beginning of each unit) and the course objectives (listed in this course guide).

### **Facilitators/Tutors and Tutorials**

Sixteen (16) hours are provided for tutorials for this course. You will be notified of the dates, times and location for these tutorial classes. As soon as you are allocated a tutorial group, the name and phone number of your facilitator will be given to you.

These are the duties of your facilitator: He or she will mark and comment on your assignment. He will monitor your progress and provide any necessary assistance you need. He or she will mark your TMAs and return to you as soon as possible. You are expected to mail your tutored assignment to your facilitator at least two days before the schedule date.

Do not delay to contact your facilitator by telephone or e-mail for necessary assistance if you do not understand any part of the study in the course material. You have difficulty with the self-assessment activities. You have a problem or question with an assignment or with the grading of the assignment.

It is important and necessary you attend the tutorial classes because this is the only chance to have face to face contact with your facilitator and to ask questions which will be answered instantly. It is also a period where you can say any problem encountered in the course of your study.

### **Final Examination and grading**

The final examination for EHS 403: Environmental Health Laboratory will be of 1½ hours duration. This accounts for 70 % of the total course grade. The examination will consist of questions which reflect the practice, exercises and the tutor-marked assignments you have already attempted in the past. Note that all areas of the course will be assessed. To revise the entire course, you must start from the first unit to the twelfth unit in order to get prepared for the examination. It may be useful to go over your TMAs and probably discuss with your course mates or group if need be. This will make you to be more prepared, since the examination covers information from all aspects of the course.

### **Summary**

This course, Environmental Health Laboratory contains relevant information on Environmental Health Laboratory management. The knowledge gained from the course is to enable you as an environmental scientist to take up any role involving Environmental Health Laboratory management.

I wish you success in this course.

**Course Code: EHS 403**

**Course Title: Environmental Health Laboratory**

**Course Developer/Writer: Dr. E.O. Balogun**

## **MODULE 1: INTRODUCTION TO LABORATORY SCIENCE**

Unit 1: Introduction to Laboratory Science

Unit 2: Establishment of Environmental Laboratories

Unit 3: Importance and Functions of Environmental Health Laboratory

Unit 4: Laboratory Record Keeping

### **UNIT 1: Introduction to Laboratory Science**

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1.0 Introduction

2.0 Objectives

3.0 Main content

3.1: Equipment and supplies

4.0 Conclusion

5.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

#### **1.0 Introduction**

For successful implementation of environmental protection programs, it is essential to identify and quantify the pollution sources, pollutants, conduct baseline survey, lay down standards and build-up monitoring systems. To meet these requirements, a competent laboratory is required with all necessary instruments, equipment, expertise, capabilities etc. This is the focus of Environmental Science Laboratory.

Scientific laboratories can be found as research room and learning spaces in schools and universities, industry, government, or military facilities, and even aboard ships and spacecraft. Early instances of "laboratories" recorded in English involved alchemy and the preparation of medicines (Oxford English Dictionary, 3<sup>rd</sup> Edition). The title “laboratory” is also used for certain facilities where the processes or equipment used are similar to those in scientific laboratories. These notably include: Medical laboratory (involves handling of chemical compounds), Public health laboratory, Industrial laboratory etc. The emergence of Big Science during World War II increased the size of laboratories and scientific equipment, introducing particle accelerators and similar devices.

Laboratory techniques are the set of procedures used on natural sciences such as chemistry, biology, physics to conduct an experiment, all of them follow the scientific method; while some of them involve the use of complex laboratory equipment from laboratory glassware to electrical devices, and others require more specific or expensive supplies.

## **2.0 Objectives**

At the end of this unit, students should be able to have a general overview of Laboratory Science.

## **3.0 Main content**

### **3.1 Equipment and supplies**

Laboratory equipment refers to the various tools and equipment used by scientists working in a laboratory: The classical equipment includes tools such as Bunsen burners and microscopes as well as specialty equipment such as operant conditioning chambers, spectrophotometers and calorimeters.

Laboratory equipment is generally used to either perform an experiment or to take measurements and gather data. Larger or more sophisticated equipment is generally called a scientific instrument.



Environmental Health Laboratory is a branch of Public Health and is dependent on a functional Environmental Laboratory which makes use of scientific methods in the assessment and management of environmental influences on human health. Environmental pollution alters the physical, chemical or biological characteristics of our air, water or land, these may harmfully affect human life, flora, fauna and materials.

#### **4.0 Conclusion**

Science laboratories make use of scientific methods to carry out experiments, the results of these experiments inform decision and policy formulation, especially in the field of Environmental Science which may impact on the Public Health

#### **5.0 Summary**

In this unit, we discussed the need for Science Laboratories.

#### **6.0 Tutor-Marked Assignment**

1. What is Laboratory Science?
2. What are laboratory techniques?
3. What is laboratory equipment?

#### **7.0 References/Further Reading**

Carlson, Adam (September 5, 2013). "Top 8 Tools for Building a Personal Prototyping Laboratory". EE Times.

ISO 13407:(1999), titled Human-centred design processes for interactive systems, is an ISO Standard providing Guidance on human-centred design activities throughout the life cycle of interactive computer-based systems.

Fritzsche, A (2017). "Corporate Foresight in Open Laboratories - A Translational Approach". Technology Analysis & Strategic Management. doi:10.1080/09537325.2017.1380180 .

Oxford English Dictionary (3rd ed.). Oxford University Press. September 2005. (Subscription or UK public library membership required.): "Originally: a room or building for the practice of alchemy and the preparation of medicines. Later: one equipped for carrying out scientific experiments or procedures, esp. for the purposes of research, teaching, or analysis; (also) one in which chemicals or drugs are manufactured."

World's Oldest Laboratory. *Analytical Chemistry*. 62 (13): 701A–701A. 30 May 2012. doi:10.1021/ac00212a716 .

## **UNIT 2: ESTABLISHMENT OF ENVIRONMENTAL LABORATORIES**

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1.0 Introduction

2.0 Objectives

3.0 Main content

3.1: Legal Provisions of Recognition of Environmental Laboratories

3.2: The Water (Prevention and Control of Pollution), Act, 1974.

3.3: The Air (Prevention and Control of Pollution) Act, 1981

3.4: The Environment (Protection) Act, 1986

4.0 Conclusion

5.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

### **1.0 Introduction**

Environmental health is the branch of public health concerned with all aspects of the natural and built environment affecting human health. Environmental Health (EH) seeks to assess and manage environmental influences (e.g. chemical, physical, biological, social and psychosocial factors) on human health. This entails the study of food safety and hygiene (including production, distribution and fitness for human consumption), occupational health and safety (including investigation and control of work-related ill health), community health (communicable and non-communicable disease control and prevention, disaster management, health promotion and education), the built environment (including homes, workplaces and public

spaces) and pollution control (including the control of the air, land and water). EH is about taking a preventative approach to tackling disease and ill-health rather than a curative approach. These is achieved though functional environmental laboratories. Most approaches in Environmental health include pollution control strategies and programmes.

The environmental laboratory plays a very important role in assessing the status of environment comprising both abiotic (soil, water and air) and biotic (flora, fauna and human being) components. An environmental laboratory processes samples taken from the environmental media (air, water, soil, and biota) both from the environment as well as from sources disposing into the environment (industries, domestic and agriculture sources, automobiles etc.).

The laboratories are the essential corner stones of any effective pollution control programme. The analytical laboratories provide qualitative as well as quantitative data for good decision making purpose. For generating this valuable data with a desired accuracy and to quantify concentration of the constituents present in the samples, the laboratory should have the desired facilities and capabilities to achieve the above goal. The mission of the Environmental Health Lab (EH) is to provide public health leadership in development of laboratory methods, information, and guidance for the assessment and prevention of indoor and outdoor air pollution, and human exposures to toxic chemicals.

## **2.0 Objectives**

At the end of this unit, the student will be able to understand the need for the establishment of Environmental Health Laboratory.

### **3.0 Main content**

#### **3.1: Legal Provisions of Recognition of Environmental Laboratories**

In the implementation of the various pollution control acts laid down for the protection of the environment, there is need for laboratories. There are several acts which strongly support the establishment of environmental laboratories. Some of these include:

- The Water (Prevention and Control of Pollution), Act, 1974
- The Air (Prevention and Control of Pollution) Act, 1981
- The Environment (Protection) Act, 1986

#### **3.2 The Water (Prevention and Control of Pollution), Act, 1974.**

Under Section 17(2) of this Act, the Board (State) may establish or recognize a laboratory or laboratories to enable the Board to perform its functions under this Section efficiently, including the analysis of samples of water from any stream or well or of samples of sewage or trade effluents. Under Section 25 & 26, any person desirous of discharging any effluent (domestic or industrial) into a stream or well has to obtain the consent of the pollution control board before discharging the same. The consent application is supported by an analysis report obtained from a recognized laboratory of the Board. Under Section 51 & 52, Central/State Government has to establish a Central/State Water Laboratory and under section 53 subsection (i) and (ii), a government analyst (Central/State) is appointed to analyse the samples. Under Section 53 Subsection (iii), the Central/State Board is required to appoint a Board Analyst(s) to any laboratory established or recognized under section 16 or 17 of the Water Act, 1974. The analysis report signed by a Government/Board Analyst is used as an evidence for the legal matters.

#### **3.3 The Air (Prevention and Control of Pollution) Act, 1981**

Under Section 17(2) of The Air (Prevention & Control of Pollution) Act, 1981, a State Board may establish or recognize a laboratory or laboratories to enable the Board to perform its

functions under this Section efficiently. Under Section 21, no person can operate any industrial plant in an air pollution control area without the previous consent of the Board. The analysis report obtained by the recognized laboratory in respect of the quantity of emissions is to be enclosed with the consent application. Under Section 28 & 29 Subsection (i), State Government should establish or specify one or more State Air Laboratories for the analysis of the samples of air. Also under Section 29 subsection (1), the State Govt. shall appoint the Government Analyst for the purpose of analyzing the samples received by the Government Laboratory under Section 28. Under Section 29 Sub-section (ii), The State Government is required to appoint the Board Analyst(s) for analysis of the air samples under section 17.

### **3.4 The Environment (Protection) Act, 1986**

Under Section 12, the Central Govt. shall establish or recognize one or more environmental laboratories to carry out the functions entrusted to an environmental laboratory under the said Act. Under Section 13, Central Govt. shall appoint the Government Analyst(s) for carrying-out the analysis of samples of air, water, soil or these substances sent for analysis to the environmental laboratory established under section 12 of the Act.

### **4.0 Conclusion**

Pollution control programs are dependent on the information from environmental laboratories. The establishment of Environmental Health Laboratories is supported by legal provisions, and these laboratories play central roles in environmental monitoring programmes and policy formulation by the government.

### **5.0 Summary**

In this unit, we discussed the different legal provisions for the establishment of Environmental Laboratories.

## **6.0 Tutor-Marked Assignment**

1. What is an Environmental Laboratory?
2. What is the role of Environmental Laboratory in pollution control programs?
3. What are the legal provisions for the establishment of Environmental Laboratories?

## **7.0 References/further reading**

Environmental Health Laboratory (2018). About the Environmental Health Laboratory.

<https://www.cdph.ca.gov/Programs/CCDPHP/DEODC/EHLB/Pages/About-the-Environmental-Health-Laboratory-.aspx/>

## **UNIT 3: IMPORTANCE AND FUNCTIONS OF ENVIRONMENTAL HEALTH LABORATORY**

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3.1: Functions of Environmental Laboratories

4.0 Conclusion

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7.0 References/Further Reading

### **1.0 Introduction**

The purpose of the Environmental Laboratory is to provide the highest value and quality of environmental testing, monitoring, assessment and information services to benefit both the human community and the natural environment. Among the varieties of samples that the Environmental Laboratory collects, tests and assess are: water, air, soil, hazardous materials and biological materials.

The Environmental Laboratory supports the environmental protection functions of federal and state governments by performing chemical, bacteriological and radiological analyses of environmental samples including drinking water, surface water, wastewater, sediment, air, fish, soil and hazardous waste. Water quality and biological monitoring of surface water is an



important section function. Quality environmental testing services are provided to a variety of customers in a cost-effective manner. The Environmental Health Laboratory maintains the capability to respond to pollution and radiation emergencies. Environmental laboratories develop new analytical methods and provide technical training and consultation at the request of clients. The Environmental Laboratory must ensure that testing capacity is available to support the public health and environmental protection objectives of the government.

The Environmental Laboratory includes some to these technical areas: General Chemistry, Metals Chemistry, Organic Chemistry, Radiation Chemistry, Water Microbiology, Air Quality, etc.

## **2.0 Objectives**

At the end of this unit, the students should be able to explain the importance and functions of Environmental Health Laboratory

## **3.0 Main content**

### **3.1: Functions of Environmental Laboratories**

The following are the functions of Environmental Laboratories:

1. Development of standardized methods for sampling and analysis if various types of environmental pollutants;
2. Analyses of samples sent by the government, agencies or others.
3. Carry out investigation as directed by the government to lay down standards for the quality of environment and discharge of environmental pollutants, to monitor and to enforce the standards laid down;
4. Prepare and make available periodical reports regarding its activities to the government
5. Carry out other functions as may be entrusted to it by the government from time to time

#### **4.0 Conclusion**

Public Health is largely dependent a reliable assessment of environmental pollution and its impact on the quality of human life. This is made possible by establishing functional Environmental Health Laboratories.

#### **5.0 Summary**

In this unit, we discussed the importance and functions of Environmental Health Laboratory.

#### **6.0 Tutor-Marked Assignment**

1. Why is Environmental Laboratory important in Public Health?
2. Mention five functions of Environmental Laboratories

#### **7.0 References/Further Reading**

The Environment (Protection) Rules (1986). Functions of Environmental Laboratories.

[www.medinda.net](http://www.medinda.net)

Environmental Laboratory Lee County Southwest Florida.

[www.leegov.com/naturalresources/EnvLab](http://www.leegov.com/naturalresources/EnvLab).

Environmental Laboratory (2018). Minnesota Department of Health.

[www.health.state.mn.us/divs/phl/environmental/index.html](http://www.health.state.mn.us/divs/phl/environmental/index.html)

State of Delaware: Environmental Laboratory Section. Division of Water.

[dnrec.alpha.delaware.gov/water/environmental-lab/](http://dnrec.alpha.delaware.gov/water/environmental-lab/)

## **Unit 4: LABORATORY RECORD KEEPING**

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### **1.0 Introduction**

Laboratory notebooks are used to keep records of daily activities in a laboratory. Lab notebooks contain: a chronological record and the primary document of a person's work in a research laboratory; a complete record of procedures, reagents, data, and scientific interpretations and hypotheses to pass on to other researchers; factual details of experiments, including thought experiments, ideas, inventions, etc.

### **2.0 Objectives**

At the end of this unit, students will be able to understand the use of Laboratory records.

### **3.0 Main content**

#### **3.1: Laboratory Notebooks**

Laboratory notebooks are used to keep records of daily activities in a laboratory. Lab notebooks contain: a chronological record and the primary document of a person's work in a research laboratory; a complete record of procedures, reagents, data, and scientific interpretations and hypotheses to pass on to other researchers; factual details of experiments, including thought experiments, ideas, inventions, etc.

A Lab Notebook is different from other documents e.g. logbook, diary etc. used in the Laboratory. Lab Notebook records factual details of experiments, which also include ideas, inventions, etc.

**Log book:** It is a list of measurements made on the autoclave, balance, centrifuge, etc.

**Diary:** a personal record, feeling, opinions (less factual than the notebook).

It is okay to place interpretations and opinions related to the experiments in a notebook, however, care should be taken to delineate fact from opinion.

#### **3.2 Laboratory Record Keeping Procedures**

Laboratory notebooks are the means for keeping a permanent record of the details of an individual's day-to-day research and development work in the laboratory or office. They provide a basic reference which the individual and others can refer to a later date; legal evidence with respect to the materials recorded, such as conception of an invention and the date thereof or date of reduction to practice and test results; in some instances are required for compliance with the provisions in many grant and contractual arrangements.

When considering the type of notebook to be used for record keeping, it is important to select a notebook with bound and numbered pages. This is extremely important. Loose-leaf notebooks and tablets with tear out pages are not to be used. Each researcher (scientist) should have a separate notebook. Notebooks should not be shared.

### **3.3 What a lab book should include**

A lab book does not have to be carefully structured and presented to the same degree as a formal lab report. However there are a number of key things that need to be included. A complete record of an experiment or project will probably contain the following:

<b>Heading</b>	A suitably descriptive title for the work.
<b>Date and time of start</b>	(Additional dates for each day you work on it).
<b>Aims</b>	What is it that you want to do?
<b>Background</b>	References to lab scripts, text books, manuals etc.
<b>Description of set up</b>	Best accomplished with the aid of simple figures. What apparatus did you use, how was it put together and what was it supposed to do?
<b>Initial set up and preliminary data</b>	It is very common to set out to do a quick sloppy job on an experiment first time round, with the expectation that this will show you what you need to go back and do well to get your real data.
<b>Review</b>	Almost nothing worthwhile works properly the first time. This is one of the key lessons that lab work will teach you. What went wrong first time round? How are you going to fix it?
<b>Definitive data</b>	Once you understand the experiment you can home in on the key results you need and do these with particular care. Usually a clear, well labeled table with units, errors on individual readings etc.
<b>Data analysis including error</b>	Any result from an experiment is meaningless without

<b>calculations</b>	consideration of the errors involved.
<b>Comparisons and conclusions</b>	Did the experiment agree with theory work as expected, need doing again etc.
<b>Summary</b>	What was the final result, what worked well and what did not, what could be improved in the future? This part is particularly critical for the “short” lab experiments
<b>Closure</b>	Be clear where one thing finishes and another starts.

### **3.4 Instructions for Recording Data in the Laboratory Notebook**

1. All entries should be made in a legible and orderly manner using permanent ink, preferably black. Make entries clear and complete so that someone else could repeat the experiment if necessary.
2. Avoid erasures. If an error is made, cross it out and make the correction immediately thereafter. Cancellations or insertions should be initialed, dated and explained (in the margin, if possible), by an appropriate notation.
3. Make sure the control page information is filled out prior to usage.
4. State the object and results of each experiment clearly and concisely. Give a complete, factual and self-explanatory account of the progress of the work and the procedure followed (reference to earlier work done by yourself or another maybe accomplished by noting a previous page of the same notebook or the page and number of an earlier notebook). All operating details and conditions should be reentered, indicating yields, conversion, etc., and identifying products. Describe and give quantities of all materials used. Explain all code numbers and abbreviations.
5. An entry dealing with a conception (invention, idea) should describe the thing conceived (Example: whether it is a chemical compound, a combination of compounds, a

combination of a compound and a solvent, etc.) as well as the utility for the thing conceived, how it is to be used and the method(s) by which it is to be prepared, including equivalent materials which could be used. Statements with regard to utility should be stated positively.

6. Work toward practical implementation of an invention should start as soon as possible after disclosure in a notebook.
7. Negative or disparaging entries should be avoided. (Example: If a reaction was expected to produce a 2% yield but instead produced 1%, do not state, "Process does not work"). Phraseology which expresses gratuitous comments or an opinion rather than a positive statement and should be avoided. This is not an instruction to omit the conclusion in an experiment, which had less than the expected results. Be factual.
8. Each day's work should, whenever possible, be started on a separate page with lines drawn diagonally across the unused portion of the previous page. (This gives legal evidence that additions were not made at a later date.) It is permissible, as stated above, to make reference to an earlier page by reciting "Continued from page..."
9. It is extremely important that each page show the date of entry.
10. Each page must be signed and dated by the individual who makes the entry and does the work. In addition, each page should be witnessed (signed and dated), using the notation "Read and Understood", preferably on the same day, but at least within one week. The witness should not be connected with the conception, should not have taken part in the experimental work performed by another, but should understand the technical field of the entry. Record the project number and the record book number on each page.

11. Where two or more individuals make a conception, it need only be entered in the notebook of one, but must be signed and dated by all the conceivers. Ideally, all the individuals should sign on the same day.
12. For copying purposes, graphs, charts, analytical data, etc. should be attached to the notebook pages with a permanent adhesive and should, when unfolded, be kept within the confines of the opened notebook. No entries should be made on the page beneath attached sheets and nothing must be obscured. Leave the heading at the top and the space provided for the witnessing signature at the bottom of each page exposed. Inserts should be signed and dated by the person making the entry and witnessed by another to provide the best legal evidence. If materials such as spectra, graphs, etc. are not kept in the notebook, they must be signed, dated and identified in such a manner as to provide a reference back to the pertinent page(s) of the Laboratory Notebook itself.
13. Research notebook should be keep in the laboratory or office in a file cabinet and protected from damage, they are legal evidence. Report the loss of theft of a research notebook.
14. All data and records should be archived and be available upon request.

#### **4.0 Conclusion**

Record keeping is very important in the laboratory. Keeping good record of all activities in the laboratory is good ethic. Record keeping allows researchers/scientists to work together effectively.

#### **5.0 Summary**



In this unit, we discussed the different procedures in laboratory record keeping. We highlighted the basic structure of a laboratory notebook and also distinguished between different documents used in record keeping in the laboratory.

### **6.0 Tutor Marked Assignment**

- i. Distinguish between notebook, logbook and diary.
- ii. Highlight the basic structure of a laboratory notebook.
- iii. Mention five practices in keeping a good laboratory record.

### **7.0 References / Further Reading**

Francis, L. M. (2014). *Scientific Integrity: Text and Cases in Responsible Conduct of Research*.

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Washington, D.C. ISBN: 0841209332 <http://www.amazon.com/Laboratory-Notebook-American-ChemicalPublication/dp/0841209332>

Kathy, B. (1998). *At the Bench: A laboratory Navigator*. Cold Spring Harbor Laboratory press.

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## **MODULE 2: GENERAL LABORATORY CONSTRUCTION**

Unit 1: General requirements for laboratories

Unit 2: Laboratory Ventilation System

Unit 3: Environmental Laboratory

Unit 4: Safety Precaution in the Laboratory

### **Unit 1: GENERAL REQUIREMENTS FOR LABORATORIES**

#### CONTENTS

1.0 Introduction

2.0 Objectives

3.0 Main content

3.1: General goals of laboratories

3.2: General Requirements for Laboratories

4.0 Conclusion

5.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

#### **1.0 Introduction**

The construction of laboratory facilities requires oversight. Regulatory requirements must be addressed and good practice must be considered. Laboratory facilities have architectural, space planning, HVAC (Heating Ventilation and Air Conditioning), environmental control, and fire/life safety requirements not generally found in most types of construction. The use of chemicals and other potentially hazardous compounds separates laboratories from other types of building spaces. Protecting the health and safety of laboratory and building occupants must be the primary concern. Comfort and energy-efficiency are also of considerable importance. The space temperature must remain comfortable for occupants while maintaining an appropriate temperature for chemical processes. At the same time, facilities are under pressure to minimize operating costs.

Even including the general criterion of safety, not all laboratories are alike. Different laboratories contain different hazard levels and uses. As an extreme example, it would be inappropriate to design a high-containment biological laboratory as if it were a general chemistry laboratory due to the high consequences should a biological laboratory's containment be breached. A host of criteria, including safety, comfort and energy efficiency, must be considered when a laboratory is planned or renovated in order to determine the optimal design. This module presents the guidelines construction of a laboratory.

## **2.0 Objectives**

At the end of this unit, the students should be able to explain the general requirements for laboratories

## **Main content**

### **3.1: General goals of laboratories**

The general goals in construction of a laboratory include safety, comfort and energy efficiency. These criteria should guide the construction or renovation of a laboratory.

### **Safety**

Laboratories are designed to maintain the health and well-being of occupants. Potentially hazardous substances used in different laboratories include chemicals, radioactive materials and infectious biological agents. These materials can be manipulated daily as part of experiments, research or production. Safety must remain the primary goal of a laboratory.

### **Comfort**

Laboratory safety has to be balanced with worker comfort. Comfort primarily is concerned with maintaining appropriate temperatures and air velocities. Worker productivity will suffer if the space is too warm or too cool. Similarly, spaces with high air currents are perceived as drafty and cool. Air currents also impact safety by limiting containment in fume hoods and other protective equipment.

Ease of use of the laboratory equipment is also a factor in worker comfort. Laboratories employing highly specialized equipment, like glove boxes, may be safest. However, this equipment carries an ease of use penalty inappropriate for the hazards encountered in most chemical laboratories. Laboratory equipment and layout must allow staff to perform necessary tasks with minimal additional effort.

### **Energy Efficiency**

Laboratories are normally designed as once-through systems, without recirculation. Conditioning, supplying and exhausting the large volumes of air used in laboratories consumes sizeable quantities of energy. Reducing these energy costs has a direct impact on cost of running

laboratories. Laboratories must be designed so that energy efficiency gains do not reduce safety and comfort.

### **3.2 General Requirements for Laboratories**

#### **i Scope**

The primary objective in laboratory design should be to provide a safe, accessible environment for laboratory personnel to conduct their work. A secondary objective is to allow for maximum flexibility for safe research and teaching use. Therefore, health and safety hazards should be anticipated and carefully evaluated so that protective measures can be incorporated into the design wherever possible.

#### **ii Building Design Issues**

Because the handling and storage of hazardous materials inherently carries a high risk of exposure and injury, laboratory should be segregated into compartments (laboratory and non-laboratory activities) to the extent possible, e.g. offices should be separated from laboratories.

#### **iii Laboratory Design Considerations**

1. The laboratory should be bound by walls and a roof or ceiling.
2. Design for adjacent spaces for storage and consumption of food and drink as needed.
3. Design laboratory workstations to accommodate the range of body dimensions that may be using the workstations. For example, computer and microscopes work stations may require height-adjustable work surfaces and chairs.
4. Each laboratory using hazardous materials, whether chemical, biological, or radioactive, should contain a sink for hand washing.
5. All work surfaces (e.g., bench tops, counters, etc.) should be impervious to the chemicals and materials used in the laboratory.

6. The laboratory should be designed so that it can be easily cleaned. Bench tops should be of a seamless one-piece design to prevent contamination. Penetrations for electrical, plumbing, and other considerations should be completely and permanently sealed. If the bench top abuts a wall, it should be covered or have a backsplash against the wall.
7. The walls should be non-porous and painted with a durable, impervious finish to facilitate decontamination and cleaning. High gloss paint is recommended.
8. Provide shelf lips for seismic restraint. Lips should be  $\frac{3}{4}$  inch above the shelf surface for bookshelves and 1  $\frac{1}{2}$ " inches above the shelf surface for shelves used to store breakable containers, chemicals, or other hazardous materials.
9. Design must put in consideration to provide spaces for sources containing Class 3B or 4 Lasers and spaces containing other sources of non-ionizing radiation (radio-frequency, microwave, ultraviolet, etc.) and large magnetic fields.

#### **iv Building Requirements**

1. Building Occupancy Classification and Control Areas - Occupancy classification and control areas should be based upon an assessment of the projected chemical inventory of the building.
2. Facilities using radioactive materials may need to be approved by relevant authorities and a Notice of Construction (NOC) may need to be filed with Ministries and agencies.

#### **v Hazardous Materials Design Issues**

- Facilities should be designed so that use of a respirator is not required for normal operations.

- There must be adequate in-laboratory storage cabinets to store reagents and chemicals and to provide segregation of incompatible materials. Storage design should be based on projected quantities and waste management practices.
- The laboratory should have a means of securing specifically regulated materials such as controlled substances regulated by the National Food Drug Administration and Control or National Drug Law Enforcement Agency etc. and radioactive materials, select agents, etc. (i.e., lockable doors, lockable cabinets etc.), where applicable.

**vi Entries, Exits, and Aisle Width**

1. Self-closing laboratory doors should be operable with a minimum of effort to allow access and egress for physically challenged individuals. A minimum of a 36-inch-wide door should be provided to facilitate equipment movement.
2. Laboratory benches, laboratory equipment and other furniture or obstacles should not be placed so that there is less than five feet of clear egress within the laboratory.
3. Laboratory doors that separate laboratory areas from non-laboratory areas are to be automatically self-closing.
4. Corridors should not be less than 6 feet wide to allow for movement of large equipment and allow for circulation of materials on carts, etc.
5. Common corridors should not be programmed for laboratory operations.
6. Equipment corridors should be provided with a system designed to allow for securing equipment to prevent movement during an earthquake.

**vii Electrical and Utility Issues**

1. Electrical receptacles above counter tops within six feet of sinks, safety showers, or other sources of water, should have GFCI (Ground Fault Circuit Interrupter) circuit protection.

2. Laboratories should be provided with light fixture on emergency power at the entrance/exit door. Hallway and corridor emergency light shall be provided based on the local code requirements.

3. Flexible connections should be used for connecting gas and other plumbed utilities to any freestanding device where rupture of the supply, return, exhaust or vent line could pose a hazard.

#### **4.0 Conclusion**

Laboratories have different construction requirements which are based on the type of research intended to be carried out. In planning a laboratory, health and safety of the occupants is of great importance, also types of chemicals and equipment to be housed in the laboratory must be put into consideration.

#### **5.0 Summary**

In this unit, we discussed the general requirements for laboratory construction.

#### **6.0 Tutor-Marked Assignment**

1. What are the general goals of laboratories?
2. State the primary and secondary objectives in laboratory design.
3. What are the building requirements for constructing a laboratory?

#### **7.0 References / Further Reading**

Biosafety Code of Practice, Safety Resources.

Laboratory Safety Design Guide (2014). Environmental Health and Safety, University of Washington. 206.543.7262, Page 7 of 74 [www.ehs.washington.edu](http://www.ehs.washington.edu)



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## **Unit 2: LABORATORY VENTILATION SYSTEMS**

### CONTENTS

- 1.0 Introduction
- 2.0 Objectives
- 3.0 Main content
  - 3.1: Air Supply to the Laboratory
  - 3.2: Determining Supply Air Needed
  - 3.3: Ventilation Control Types
- 4.0 Conclusion
- 5.0 Summary
- 6.0 Tutor-Marked Assignment
- 7.0 References/Further Reading

### **1.0 Introduction**

The key concept of laboratory ventilation is that air entering the laboratory must exit the laboratory. The inflowing air volume, normally composed of supply air and infiltration, will

exactly equal the outgoing air volume, or air exhausted through room exhaust, fume hoods, canopy hoods, biological safety cabinets and exfiltration. All airflows must be accounted for when designing a building with laboratories.

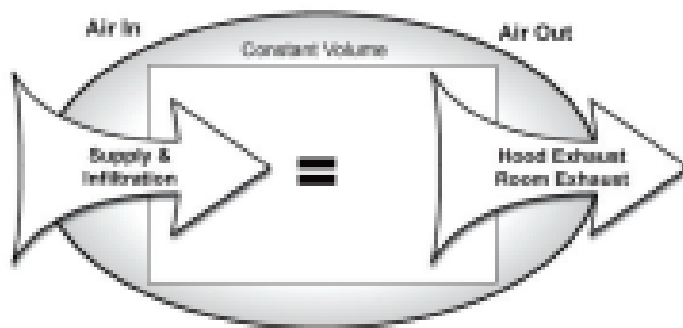
## 2.0 Objectives

At the end of this unit, the student should be able to understand laboratory ventilation systems.

## 3.0 Main content

### 3.1 Air Supply to the Laboratory

In practice, the volume of air supplied into a laboratory is less than the amount of air exhausted, creating negative pressure. The additional air exhausted creates a low-pressure vacuum, pulling air in from adjacent spaces through cracks in the wall, the door undercut, unsealed duct and piping openings or other wall penetrations.



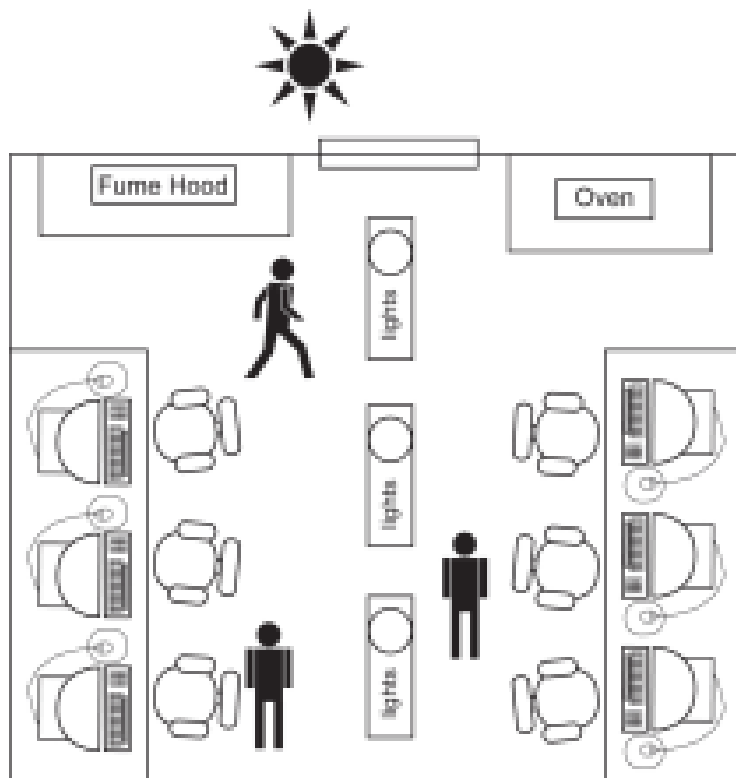
**Figure 1: All air exhausted from a lab must enter through either the building HVAC system or infiltration.**

### 3.2 Determining Supply Air Needed

Three drivers determine the required volume of supply air in a laboratory: temperature, exhaust, and ventilation.

Temperature-driven laboratories hold a lot of equipment to perform chemical analysis or ovens elements to speed up chemical processes. Without an adequate supply of cool air, the laboratory

housing this equipment will become uncomfortably warm. Lights, laboratory personnel, and even heat transmitted through the building also contribute to the cooling load of a laboratory. Determining the necessary supply air volume for cooling involves summing up all of these loads (see Figure 2). However, loads other than the building envelope should be determined according to expected usage. For example, engineers will revise supply air volume downward if all of a laboratory's equipment will not be used simultaneously.

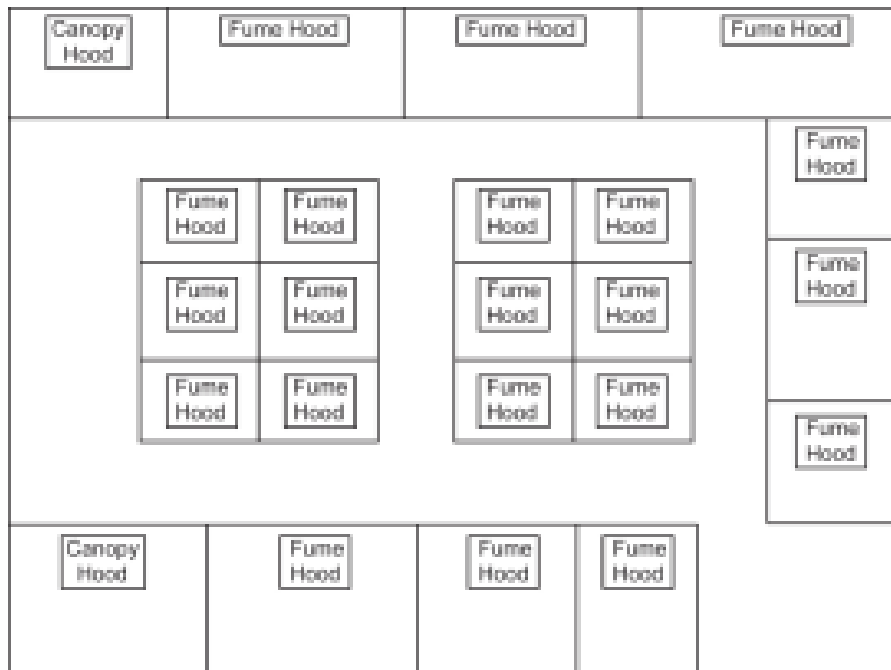


**Figure 2: Supply air volume in laboratories with high heat loads, such as computers, ovens and other electrical equipment, is driven by cooling requirements.**

Exhaust-driven laboratories, such as teaching laboratories, are virtually filled with fume hoods and canopy hoods (see Figure 3). Fume hoods exhaust large quantities of air in order to contain gasses, vapors, particles and other contaminants. All air exhausted from the laboratory, including

fume hood exhaust, must be replaced to prevent excessively negative room pressures within the laboratory and possibly the building.

Finally, some laboratories have low cooling loads and few, if any, fume hoods and other exhaust equipment. These ventilation-driven laboratories still require high supply air volumes to dilute contaminants. The ventilation rate is normally expressed in units of Air Changes per Hour (ACH), calculated as the total air volume supplied in one hour divided by the room volume. Occupied laboratories often have ventilation rates on the order of 8 to 10 ACH, although it could be as low as 4 ACH when unoccupied (National Fire Protection Association, 2000). In comparison, offices usually see a much lower ventilation rate, often on the order of 4 ACH (ASHRAE, 2003).



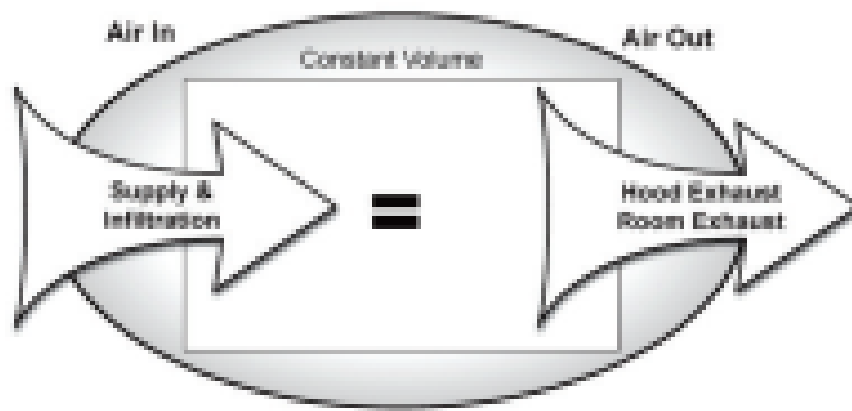
**Figure 3: High volumes of air exhausted from fume hoods, canopy hoods and other exhausts require a correspondingly large volume of air to be supplied to a laboratory**

### 3.3 Ventilation Control Types

The different types of ventilation control include:

## 1. Constant Volume (CV) Control

The first laboratories with mechanical ventilation, and many laboratories today, were designed as CV systems. As the name implies, the air volume supplied and exhausted from a CV laboratory does not change. Time of day, day of the week, holidays and building usage have no impact on operation of the HVAC system.



**Figure 4: Airflows in constant volume laboratories remain at consistently high levels at all times.**

Usually, in this control system, the supply volume remains less than the exhaust, maintaining a negative balance.

The CV control sequence has a number of advantages, even when used with laboratories driven by exhaust or cooling. These include:

1. Easy to design.
2. Minimizes cost of controls.
3. Few controls to maintain.

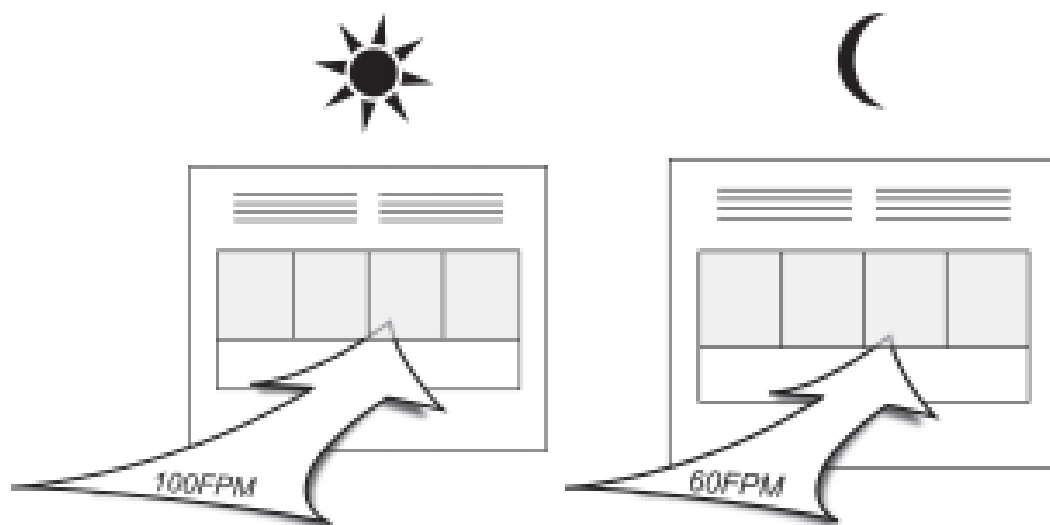
The CV sequence of operations has a number of potential disadvantages, including:

- Mechanical equipment must be sized for full-flows, increasing first and operating costs of the fans, chillers and other capital equipment.

- Difficulties relocating equipment. Moving equipment within a building may change the HVAC system pressure distribution, resulting in a requirement to rebalance systems without pressure-independent controls.
- Limited future expansion, because there may not be sufficient system capacity to allow additional equipment.
- Limited opportunities to warn users of unsafe system operations. Monitors and controllers are not inherent to CV systems. Adding monitors to comply with recent standards<sup>4</sup> adds cost. Older systems without monitors cannot warn users of unsafe conditions, potentially risking exposure.

## 2. 2-Position Control

Initial efforts to reduce the operating expenses associated with the high continuous operations of constant volume control focused on reducing airflow in unoccupied laboratories. Under this scenario, full airflows are present only when laboratory users are present. Reduced airflows are possible when the laboratory is unoccupied because users are not present to create airflow obstructions at fume hoods.



**Figure 5: 2-Position controls reduce airflows and therefore operating expenses when the space is unoccupied.**

The 2-Position control sequence offers similar advantages and disadvantages as the constant volume control sequence. Benefits include:

- Easy to design.
- Low cost of controls when compared to a variable air volume system.
- Decreased flows during unoccupied hours can reduce operating costs when compared to a constant volume system.

Weaknesses of the 2-Position control system mirror those of the constant volume control system:

- Mechanical equipment must be sized for full-flows, increasing first and operating costs of the fans, chillers and other capital equipment.
- Difficulties relocating equipment. Moving equipment within a building may change the HVAC system pressure distribution, resulting in a requirement to rebalance systems without pressure-independent controls.
- Limited future expansion, because there may not be sufficient system capacity to allow additional equipment.
- Limited opportunities to warn users of unsafe system operations. Monitors and controllers are not inherent to 2-Position systems. Adding monitors to comply with recent standards adds cost. Older systems without monitors cannot warn users of unsafe conditions, potentially risking exposure.

### **3. Variable Air Volume**

VAV laboratories reduce supply and air volumes to the minimum required to maintain temperature, ventilation and safe fume hood face velocities. When fume hood sashes are

lowered, VAV fume hood controls reduce the air exhausted to maintain constant velocity of air through the sash opening while VAV laboratory controls correspondingly reduce supply air to balance the space. Similarly, VAV room controls increase general exhaust volume to balance the laboratory when increasing the supply air volume to cool the space.

VAV laboratories offer a number of advantages and disadvantages when compared to constant volume and 2-position labs. Advantages include:

- Reduced energy costs because less air will be conditioned, supplied and exhausted when loads decrease.
- Use unoccupied mode to decrease supply and exhaust airflows to further save energy expenses.
- Applying diversity decreases maximum design airflows, resulting in smaller capital equipment such as fans, ductwork, and air handlers.
- Pressure-independent VAV controls adapt to system changes when equipment is moved or added, requiring rebalancing only in areas directly affected by the change as opposed to the entire building.
- VAV controls alarm if fume hood face velocity and room pressure differential or balances reach potentially unsafe levels.

Disadvantages of the VAV system include:

1. Reduced airflows are dependent on users closing fume hood sashes to reduce airflows. VAV system will not reduce airflows if sashes remain open.
2. Increased HVAC system complexity, including requirements for VAV controls on fans, air handlers and other capital equipment.

#### **4.0 Conclusion**



Proper ventilation is essential in maintaining the health of laboratory occupants and integrity of reagents and equipment. Poor ventilation can seriously affect efficiency and output in a laboratory.

### **5.0 Summary**

In this unit, we discussed the different systems of ventilation in laboratory construction.

### **6.0 Tutor-Marked Assignment**

1. How can you determine the air supply needed in constructing a laboratory?
2. List and explain the different ventilation control types? What are the advantages and disadvantages of these control types mentioned above?

### **7.0 References / Further Reading**

National Fire Protection Association, Standard NFPA 45 (2000). Section 6.3.3, p. 12.

ASHRAE 2003 Applications Handbook. Chapter 3, Commercial and Public Buildings, p. 3.2.

Laboratory Design Handbook (2014). TSI Incorporated. [www.tsi.com](http://www.tsi.com)

## **UNIT 3: REQUIREMENTS FOR ENVIRONMENTAL LABORATORIES**

### CONTENTS

1.0 Introduction

2.0 Objectives

3.0 Main content

3.1: Laboratory Design/Housing/Building

3.2: Laboratory Furniture

3.3: Electricity Supply and Electrical Services

3.4 Sample Digestion System/Hood System

3.5 Water Supply & Distilled/De-ionized Water

3.6 Environmental Laboratory Soundness

4.0 Conclusion

5.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

### **1.0 Introduction**

Environmental laboratories should have sufficient space, proper design, interior furnishing, proper ventilation, proper lighting, temperature control, dust free atmosphere etc. since all these factors influence the quality of analytical data generated in a laboratory and also may affect the production of reliable data. Regular and proper supply of some of the essential needs like water and electricity is to be ensured for smooth functioning of the laboratory. In addition to these,

other important factors like facilities for preservation of samples, weighing, cleaning of glassware, quality of chemicals, fire-fighting facilities, laboratory safety and first-aid facilities are also an essential requirements.

## **2.0 Objectives**

At the end of this unit, the students should be able to discuss the requirements for environmental laboratories.

## **3.0 Main content**

### **3.1: Laboratory Design/Housing/Building**

Laboratory design plays a major role for efficient functioning of laboratory activities. Laboratory design should incorporate good spacing, proper ventilation system, well-ventilated stack rooms, store rooms, laboratory hoods, sinks, miscellaneous safety equipment like eye wash fountain, safety showers and arrangement for safe disposal of wastes. Laboratory building must have proper space for carrying out following activities separately for environmental samples:

#### **A. Water Analysis Section**

- i. Physico-chemical analysis
- ii. Microbiological examination
- iii. Biological examination

#### **B. Air Analysis Section**

- i. Ambient Air Monitoring & Analysis
- ii. Source Emission Monitoring & Analysis
- iii. Vehicular Emission Monitoring & Analysis
- iv. Noise Monitoring

#### **C. Soil and Solid Waste (including hazardous wastes) Analysis Section**

#### **D. Instrumentation Section (Analysis requiring sophisticated instruments)**

i. Inorganic analysis

ii. Organic analysis

All Infrastructural supply and discharge facilities for water, power, gas and air must be easily accessible and serviceable without constructional changes. All materials selected and specified must be of high quality and suitable for their functional end use. Laboratory design, outlay and materials should be selected and specified to minimize maintenance and operational costs of the facilities. It is envisaged that newly assigned tasks to the laboratory or any changes with reference to new instrument/equipment methods, analysts etc. can be easily and vastly accommodated.

The laboratory should maintain the following work areas with adequate instrumentation and infrastructure: Analytical Lab, Balance room, Instrument room with adequate provision of Gas cylinders etc., Microbiology room, Sample pre-treatment, Digestion & extraction room for pesticides & metals, Sample receipt section, Sample storage, Conference room-cum-library, Staff room, Computer room, Store room, Maintenance room, Laboratory Record room, Field age room, monitoring equipment room, Waste storage room.

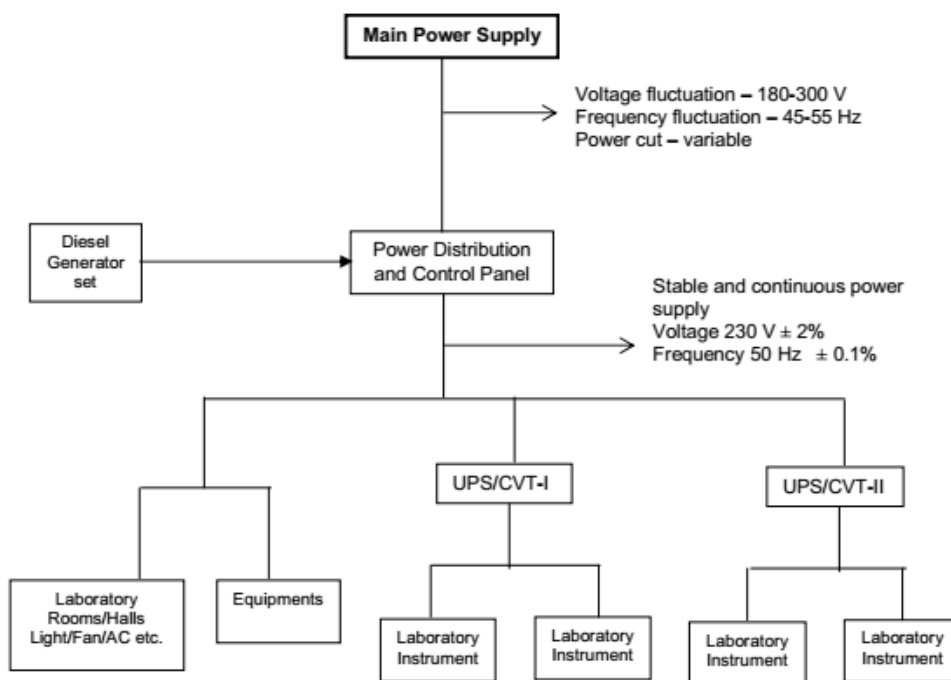
### **3.2 Laboratory Furniture**

The laboratory furniture and work benches with ergonomic designs should be given more emphasis to provide suitable laboratory work environment. The convenience and easiness of laboratories works depend upon the quality, dimension and placement of laboratory furniture as well as their ergonomic design. The laboratory working benches top surfaces are to be made up of acid and alkali resistant materials. The steel/aluminum frames used in furniture or any fittings is to be non-corrosive type. Wherever stainless steel materials are needed, the same is to be provided. The writing desks are also to be laminated and non-corrosive. The storage cupboard should be made of clipboard covered with melamine sheets. All the furniture is to be designed specifically according to the requirement of the laboratory so as to maximize the quality of work output.

### **3.3 Electricity Supply and Electrical Services**

Regular and stabilized electricity supply (220-230 volts) is essential for smooth functioning of the laboratory and its instruments. Necessary and adequate provisions should be made for continuous supply, constant voltage, adequate load, desired level of illumination, proper electrical fittings etc.

Because of the specialized nature of analytical work in laboratories, the lighting system are specific and different from those in other areas since laboratory works involve accurate readings of glassware-graduations, balance, venires, and other measuring lines, level of illumination, brightness, glass and location of light source should be controlled to facilitate ease in making these measurements and to provide maximum comfort for the laboratory analysts.



**Figure 1: Schematic diagram of the power supply system at Environmental Laboratory**

Some sophisticated instruments like Spectrophotometer, Flame photometer, Atomic Absorption Spectrophotometer, Gas chromatograph, Mercury Analyzer, Balance etc. requires constant voltage to maintain stabilized and drift-free instrument operation. The electric voltage regulation is therefore utmost necessary and can be achieved through use of voltage stabilizers and Uninterrupted Power Supply (UPS) system. Since the electrical supply for laboratory needs to be continuous, there should be the additional provision of Diesel Generator (D.G.) sets for continuous supply of power to equipment like BOD Incubator, Oven etc.

Care must be taken to ground all equipment that could constitute a shock hazard. The three-pronged plugs that incorporate grounds are best for this purpose.

### **3.4 Sample Digestion System/Hood System**

An efficient hood system is necessarily required at laboratories in order to remove various toxic and hazardous fumes from the work place generated during use of organic solvent/or during acid digestion.

## **Fume hood**

Fume hoods are safety devices, used to contain chemicals with long-term exposure hazards.

Fume hoods are not appropriate for protection from substances causing significant health consequences with only isolated, short-term exposures.

It is important to note that the technical term for a fume hood is a laboratory chemical hood.

However, industry convention remains to call them fume hoods. This primer will therefore call them fume hoods.

The exhaust system draws air through all openings in a fume hood, including the face. These entering airstreams prevent chemicals inside the air from escaping. Air in the fume hood is then exhausted, drawing vapors, gasses and particulates in the hood out.

Fume hoods come in different sizes and configurations. There are two basic configurations of fume hood. Bench top hoods, as the name suggests, are physically placed on the laboratory bench. Floor-mounted hoods, commonly called walk-in hoods, are therefore mounted on the floor. A floor-mounted hood cannot offer any protection to a user who actually enters the hood.



**Figure 2: Bench top fume hood. Photo: courtesy of Fisher Hamilton LLC.**

### **3.5 Water Supply & Distilled/De-ionized Water**

Water is an essential and basic need for laboratory operations, washing; cleaning etc. therefore the laboratory should have provision for continuous water supply either from a direct supply source or through storage tanks. It is preferred, if a dedicated water storage tank is made available exclusively for laboratory use. To avoid deterioration of stored water, frequent cleaning of storage tank is essential, particularly if the stored water is being used for drinking purpose also.

Distilled water is one of basic requirement of the laboratory and analytical errors are encountered because of improper quality of distilled water. Distilled or deionize water is used in the laboratory for preparation of reagents, dilution, and final rinsing of glass wares. Ordinary distilled water may be contaminated by dissolved gases from surrounding environment, materials leached from the container, in which it is prepared or stored. Hence for specific determinations, deionized/distilled water may require further purification.



Generally, distilled water with electrical conductivity of 2.0  $\mu$  Siemen/cm or less is considered reasonably ideal for routine work.

### **3.6 Environmental Laboratory Soundness**

The environmental laboratory facility during its operation should not itself be nuisance to the environment but it should be developed as state of the art facilities with environmental and energy considerations. In this respect it must be ensured that the environmental laboratory operations:

- does not unduly pollute air, water, soil or biota
- that hazards and accidents are prevented as far as possible
- that energy consumption at the laboratory is minimized as far as feasible.

For these requirements the environmental laboratory should adopt such work environment, which demonstrate appropriate solutions such as:

- review of analytical methods with reference to their environmental implications and replacement of extremely pollution methods, whenever feasible.
- Recovery and recycling of solvents and reagents.
- Treatment of laboratory effluents in a suitably designed effluent treatment facility.
- Separate collection of hazardous substances and their proper treatment and disposal
- Thermal insulation of the building to save energy costs for acclimatization
- Use of solar light and energy wherever possible. For example, the solar energy may be

utilized for following operation:

- Thermal heaters (in winter)
- Water distillation
- Power generation (photovoltaic)
- Hot water preparation
- Provision for natural lighting to rooms/halls whenever possible.

## **4.0 Conclusion**

There are basic requirements for that must be taken into consideration when planning an Environmental Laboratory. Different sections of the laboratories may also have different requirements which must be put into consideration in the construction process.

## **5.0 Summary**

In this unit, we discussed the requirements for the planning of Environmental Laboratory.

## **6.0 Tutor-Marked Assignment**

1. What are the different Environmental Analysis sections that must be provided for in the planning of an Environmental Laboratory building?
2. What is laboratory hood system?
3. Explain Environmental Laboratory soundness? How can it be achieved?

## **7.0 References / Further Reading**

American Conference of Governmental Industrial Hygienists (1998). *Industrial Ventilation*, 23<sup>rd</sup> Edition. ACGIH, Cincinnati, OH.

Klein, R.C., C. King, and A. Kosior (2011). Laboratory air quality and room ventilation rates: an update. *Journal of Chemical Health and Safety* 18(2):21-24.

National Research Council. (1995). *Prudent Practices in the Laboratory*. National Academy Press, Washington, DC.

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## **UNIT 4: SAFE LABORATORY PRACTICES**

### CONTENTS

1.0 Introduction

2.0 Objectives

3.0 Main content

3.1: Laboratory Work Environment

3.2: Safety Rules and Regulations in the Laboratory

4.0 Conclusion

5.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

### **1.0 Introduction**

It is essential that laboratory officials while working at laboratory must make every effort to protect themselves and their fellow workers by conscientiously adhering to the health and safety programme that has been developed and documented specifically for the laboratory.

The following facilities are essential to safeguard the health of laboratory staff:

- Multiple story buildings, especially laboratory should have adequate Exit doors in case of emergency.
- Adequate facilities for high toxic or highly inflammable materials or gases.
- Adequate number of exhaust fans for proper air circulation
- Cleanliness and good housekeeping reduces frequency of laboratory accidents.
- Adequate firefighting facilities and training to laboratory personnel in its operation.

Firefighting equipment shall be located at strategic points in the laboratories. Laboratory officials should also be trained regarding fire pertinent hazards with their work.

- Large gas cylinders should be securely fastened.
- Laboratory personnel may get exposure to toxic chemicals and solvents during laboratory analysis through following route:

- Contact with skin and eyes
- Inhalation
- Swallowing

To avoid this exposure, the safety gadgets like hand gloves, vacupets, dispensers, gas masks, goggles etc. must be used by the laboratory officials whenever necessary. Other protective equipment and / or apparel as appropriate may also be used.

### **3.1 Laboratory Work Environment**

Laboratory staff working in the laboratory is expected to develop healthy congenial environment for smooth analytical work. It is necessary to develop and encourage safe habits, avoid unnecessary exposures to chemicals by any route and avoid working alone at the laboratory as far as possible. The laboratory or other staff should not eat, drink, smoke, chew gum or apply cosmetics within laboratory work place since it is hazardous due to presence of all types of chemicals, solvents etc. Always wash hands before undertaking these activities. It is better to avoid storage, handling or consumption of food or beverage in storage area, laboratory refrigerators or glassware and utensils that are used for laboratory operations. Avoid practical jokes or other behaviour that might confuse, startle or distract other laboratory official. Do not use mouth suction for pipetting or starting a siphon. Handle and store laboratory glassware with care to avoid damage. Keep work area clean and uncluttered with chemicals and equipment properly labeled and stored. Clean up work area on completion of an operation and at the end of each day.

### **3.2 Safety Rules and Regulations in the Laboratory**

The rules and regulations that follow are universal for laboratories. In addition to becoming familiar with these, take note of safety warnings given with each specific experiment.

**General rules:** no person may work alone in the lab, supervision is needed all time. No work outside regular lab hours is permitted without specific permission. Visitors are not allowed in the labs.

**Clothing:** shorts and skirts should not be worn to the lab. Avoid wearing expensive clothes. Sandals or open-toe shoes are not acceptable. Confine long hair, or any loose clothing or accessories.

**Eye protection:** safety glasses are a required item to be worn in all areas of the laboratories. The wearing of contact lenses in the laboratory is strongly discouraged, even when eye protection is worn. There is a distinct possibility that chemicals may infuse under the contact lens and cause irreparable damage.

**Housekeeping:** All designated experimentation areas should be left in a neat orderly state at the conclusion of an experiment. The following items should be checked;

- a. All excess water should be removed from the floor.
- b. All loose paper should be picked up and deposited in trashcans.
- c. All working surfaces (tables, chairs, etc.) should be cleaned if needed.
- d. All miscellaneous items should be returned to their proper initial locations.
- e. All glassware should be washed prior to returning to the cabinet.
- f. All scales should have weights removed and scale arms locked.
- g. All manholes (sewers) should have their lids closed.
- h. All drums or containers used should be checked.
- i. Check all valves and electrical units. Turn off what is required.

**Chemicals:** In several of the experiments, chemicals are required, it is important to know what safety precautions, if any, are to be taken in conjunction with the use of these chemicals. It is very important to get the Material Safety Data Sheet (MSDS) for all chemicals.

1. In the case of gases being used, be sure you understand the nature of the hazards associated with the gas and do not deviate from the procedures as outlined, either oral or written, by the instructor.
2. Do not use mouth suction to fill pipettes.
3. Label all containers to avoid errors and read labels carefully.
4. Never remove shared chemicals from their original locations, others will need them.
5. Waste chemicals are placed in receivers and are not discharged in the drain, unless told otherwise.

**Electrical:** In many instances electrical extension cords are required for the operation of auxiliary equipment. Special precautions should be taken when using these cords.

When an electrical extension cord is checked out, be sure to examine its condition. If you find frayed or broken wires, insulation broken, prongs bent, no ground, etc., do not use but return to the stockroom, pointing out the faults to the Technical Assistants (or technicians). When using extension cords, be sure they do not lie on the floor, in particular, when the floor is wet, but are safely supported in such a fashion that they are not a bodily hazard. When making electrical connections, be sure the area you are standing in is dry.

**Accidents:** Even with the greatest safety precautions accidents DO happen. Be sure you are familiar with the locations of safety showers and medical first aid kits. If an accident happens, inform your instructor immediately. In the case of a serious accident, do not attempt first aid if you are not familiar with the proper technique, but do attempt to make the person comfortable until aid arrives. All chemicals spills are to be reported and directions must be followed for containment and cleanup.

Whenever your skin (hands, arms, face etc.) comes into contact with laboratory chemicals, wash it quickly and thoroughly with soap and warm water.

**Unauthorized Areas:** Do not touch unauthorized equipment, chemicals or experiments.

**Food or Drink:** Food and drink are forbidden in laboratories, that includes chewing gum and applying makeup. DO NOT taste chemicals, if instructed to smell chemicals do so by carefully fanning the top of test tube or bottle so that a little of the vapor is directed towards your nose.

**Smoking:** Smoking is not permitted.

**Ventilation:** Be sure that hoods are functioning, and that your work areas are properly ventilated.

**Safety Shower:** In the event of a chemical spill on your body, or if your clothes catch fire, quickly move to the safety shower, stand under it, and pull the chain. A large volume of water will fall onto your head. Get help immediately!

#### **4.0 Conclusion**

Safety in the laboratory must be taken seriously because most of the time we work with hazardous materials. Following the instructions for each experiment and adhering to safety guides is essential in maintaining safety.

#### **5.0 Summary**

In this unit, we discussed the safety rules and regulations in laboratory.

#### **6.0 Tutor Marked Assignments**

1. What are safe Laboratory practices?
2. What is the purpose of the Material Safety Data Sheet?

#### **7.0 References / Further Reading**

Water and Environmental Chemistry Laboratory Lab Manual (Part A). (2015). Department of Water and Environmental Engineering Water and Environmental Chemistry Laboratory.

Laboratory Design Guide (2019). University of Saskatchewan. Safety Resources. Pp 1-13.

Laboratory Design Handbook (2014). TSI Incorporated

## **MODULE 3: LABORATORY REAGENTS AND SOLUTIONS**

Unit 1: Preparation of reagents

Unit 2: Reagent preparation from stock standard solution

Unit 3: Converting between concentration units

Unit 4: Storage of laboratory reagents

### **UNIT 1: PREPARATION OF REAGENTS**

#### CONTENTS

1.0 Introduction

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3.0 Main content

3.1: Quality of Chemicals

3.2: Safety Symbols

4.0 Conclusion

5.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

#### **1.0 Introduction**

The chemistry reagents play an important role in the correctness of an experiment in the laboratory. If the composition of reagent is not correct, it often leads to errors in the observations and results. While preparing the chemistry reagents in a laboratory, correct proportion of chemicals and /or solvents as well as procedure is very crucial.

Safety is also very important, as Chemistry Labs are highly prone to accidents. While preparing reagents, one should be aware of correct handling, procedure, storage and safety hazards. With adequate knowledge and care, accidents can be avoided in the laboratory while preparing reagents.

#### **2.0 Objectives**



At the end of this unit, the students should be able to prepare reagents from stock solutions

### 3.0 Main Content

#### 3.1: Quality of Chemicals

The quality of chemicals/solvents used in the analytical laboratory may vary from laboratory grade to Analytical or Guaranteed Grade [Analar or AR or G.R.). The quality of chemical/solvents may become one of the causes of analytical errors, may affect the analytical instrument or may lead to interferences during determinations. Hence, selection of laboratory chemicals of an appropriate quality is most important factor for achieving result with desired accuracy.

For preparation of all standard solutions only “Analytical reagent grade (AR) or guaranteed reagent grade (GR) should be used, since their purity levels are known. Commercially produced chemical reagents such as acids and ammonia are highly concentrated solutions. For instance; commercial concentrated Tetraoxosulphate (VI) acid,  $\text{H}_2\text{SO}_4$  is approximately 18 – molar.

In order to prepare solutions of lower concentrations a calculated volume of the concentrated solution is taken from the stock solution and then added to a specified volume of distilled water. However the volume of such a solution to be taken depends on the information provided by the manufacturer on the label pasted on the stock bottle. For instance; the label on the stock bottle of concentrated  $\text{H}_2\text{SO}_4$ .

**Table 1: Concentrated Liquid Analytical Reagents**

REAGENT	% PURITY	R.M.M.	SPECIFIC GRAVITY (S.G)
$\text{CH}_3\text{COOH}$	99.5	60	1.05
$\text{NH}_3$	35	17	0.88
$\text{HCl}$	36	36.5	1.18
$\text{HF}$	48	20	1.18
$\text{HNO}_3$	70	63	1.42
$\text{HClO}_3$	60	84.5	1.53
$\text{H}_3\text{PO}_4$	85	98	1.69

H <sub>2</sub> SO <sub>4</sub>	98	98	1.80
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From Table 1, taking H<sub>2</sub>SO<sub>4</sub> for example:

The specific gravity of 1.80 means that the stock solution is 1.80 times heavier than an equal volume of water; i.e. 1cm<sup>3</sup> of the stock acid solution weighs 1.80g or 1000cm<sup>3</sup> (1dm<sup>3</sup>) of stock solution weighs 1.80 x 1000 = 1800g.

The 98% by mass means: 98g of the acid (solute) is in 100g of solution i.e. 100g of stock solution contains 98g of H<sub>2</sub>SO<sub>4</sub>. 1800g (mass of 1dm<sup>3</sup> of solution) contains  $(98/100) \times 1800 = 1764g$ . i.e. 1dm<sup>3</sup> of stock solution contains 1764g of pure H<sub>2</sub>SO<sub>4</sub>, therefore its mass concentration = 1764g/dm<sup>3</sup>. Hence, its molar concentration,  $C = 1764 \text{ per dm}^3 / 98g \text{ per mole} = 18.0 \text{ mole per dm}^3$  i.e. the concentration of the stock solution is 18.0 molar H<sub>2</sub>SO<sub>4</sub>

### 3.2 Safety Symbols

Labels are placed on chemicals to communicate to users and handlers the hazards that may be involved in using the chemicals. Some labels may carry information which tells the user if the chemical is harmful, explosive etc. Figures 1 below are some of the safety symbols on laboratory chemicals.

Symbol	Meaning	Symbol	Meaning
	Caution, Warning		Harmful, Irritant
	Dangerous, Toxic,		Corrosive
	Flammable		Explosive
	Oxidising agent		Unsafe for Environment
	Hygroscopic *		

**Figure 1: Safety symbols on laboratory chemicals**

*\* Not hazardous, but these compounds need to be stored in anhydrous conditions. While preparing reagents, due to their hygroscopic nature, their weight may vary and have effect on calculations.*

#### General Instructions in preparing laboratory reagents

It is highly recommended that the following precautions must be duly followed while preparing the reagents.

- Always wear a lab-coat and safety goggles.
- Wear disposable gloves while dealing with highly poisonous/irritant chemicals.
- Follow the specific procedure and amounts/volumes of chemicals and solvents to be used for the reagent to be prepared.
- Use clean and dry glass apparatus while preparing the reagents.
- Store the reagents after proper labeling (name of reagent, date of preparation, hazard warning) as per the instructions in clean apparatus.

- Dispose of any waste paper or any kind of solid waste properly.
- Maintain a safe distance while using the chemicals.
- Never touch, taste, or smell any chemical, whatever it may be.
- Make sure no flammable solvents are in the surrounding area when lighting a matchstick or a burner.
- Hold containers away from the body when transferring a chemical or solution from one container to another.
- Add concentrated acid to water slowly. Never add water to a concentrated acid.

#### **4.0 Conclusion**

Chemicals used in the preparation of reagents must be of analytical grade to have reliable results from experiments. Care should be taken to observe safety symbols while handling chemicals and reagents in the laboratory.

#### **5.0 Summary**

In this unit, we discussed the types of reagents used for analytical work and some safety signs that are found on chemicals used in the laboratory.

#### **6.0 Tutor-Marked Assignment**

1. What are the different qualities of chemicals/solvents used in the analytical laboratory? Which of these is best for preparation of standard solutions and why?
2. What precaution should be taken in mixing concentrated acid with water?

#### **7.0 References / Further Reading**

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## UNIT 2: REAGENT PREPARATION FROM STOCK STANDARD SOLUTION

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3.2: Expressing Concentration of Solute

3.3: Molarity and Formality

4.0 Conclusion

4.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

### 1.0 Introduction

Analysis in any laboratory is mainly based on reference materials like Stock standard solution. The accuracy in the preparation of stock standard reflects accuracy of the results.

### 2.0 Objectives

At the end of this unit, the students should be able to prepare dilute reagents from stocks of known concentrations.

### 3.0 Main content

#### 3.1: Definition of Some Terms

**Stock standard solution** is defined as a solution with high concentration of stable analyte(s) that can be stored at specific conditions in laboratory for long time and used as a standard reference material for analysis of the target analyte(s) in the daily use.

**Standard solution** is a solution of known concentration e.g. 0.5 Molar  $\text{H}_2\text{SO}_4$ .

**Molar solution** is a solution that contains 1 mole of solute per litre ( $1 \text{ dm}^3$ ) e.g. A molar solution of NaOH can be prepared by dissolving 1 mole (40 g) of NaOH (Na = 23, O = 16, H

=1) in 1 litre of distilled water. Therefore 80 g of NaOH will be required in 1 litre of distilled water to prepare 2 molar NaOH solution.

### 3.2 Expressing Concentration of Solute:

Concentration is a general measurement unit stating the amount of solute present in a known amount of solution:

$$\text{Concentration} = \frac{\text{Amount of Solute}}{\text{Amount of Solution}}$$

Although the terms “Solute” and “Solution” are often associated with liquid samples, they can be extended to gas-phase and solid-phase samples as well. The actual units for reporting concentration depend on how the amount of solute and solution are measured.

### 3.3 Molarity and Formality

Both molarity and formality express concentration as moles of solute per liter of solution. There is, however, a subtle difference between molarity and formality. Molarity is the concentration of a particular chemical species in solution. Formality, on the other hand, is a substance’s total concentration in solution without regard to its specific chemical form. There is no difference between a substance’s molarity and formality if it dissolves without dissociating into ions. The molar concentration of a solution of glucose, for example, is the same as its formality.

For substances that ionize in solution, such as NaCl, molarity and formality are different. For example, dissolving 0.1 mol of NaCl in 1 L of water gives a solution containing 0.1 mol of Na<sup>+</sup> and 0.1 mol of Cl<sup>-</sup>. The molarity of NaCl, therefore, is zero since there is essentially no undissociated NaCl in solution. The solution, instead, is 0.1 M in Na<sup>+</sup> and 0.1 M in Cl<sup>-</sup>. The formality of NaCl, however, is 0.1F because it represents the total amount of NaCl in solution. The rigorous definition of molarity, for better or worse is largely ignored in the current literature, as it is in this text. When we state that a solution is 0.1 M NaCl we

understand it to consist of Na<sup>+</sup> and Cl<sup>-</sup> ions. The unit of formality is used only when it provides a clearer description of solution chemistry.

Molar concentrations are used so frequently that a symbolic notation is often used to simplify its

expression in equations and writing. The use of square brackets around a species indicates that we are referring to that species' molar concentration. Thus, [Na<sup>+</sup>] is read as the "molar concentration of sodium ions".

$$\text{Molarity} = \frac{\text{Moles of solute}}{\text{Volume of solution (L)}}$$

$$\text{Moles of solute} = \text{Molarity} \times \text{Volume of solution (L)}$$

$$\text{Volume of solution} = \frac{\text{Moles of solute}}{\text{Molarity}}$$

$$\text{Moles of solute} = \frac{\text{Weight of solute (g)}}{\text{Molecular weight (g/mol)}}$$

Note that it is the final volume of the solution that is important, not the starting volume of the solvent used. The final volume of the solution might be a bit larger than the volume of the solvent because of the additional volume of the solute. In practice, a solution of known molarity is prepared by weighing an appropriate amount of solute and placing it in a volumetric flask. Enough solvent is added to dissolve the solute, and further solvent is added until an accurately calibrated final volume is reached.

**The solution is then shaken until it is uniformly mixed as show in Figure 1.**



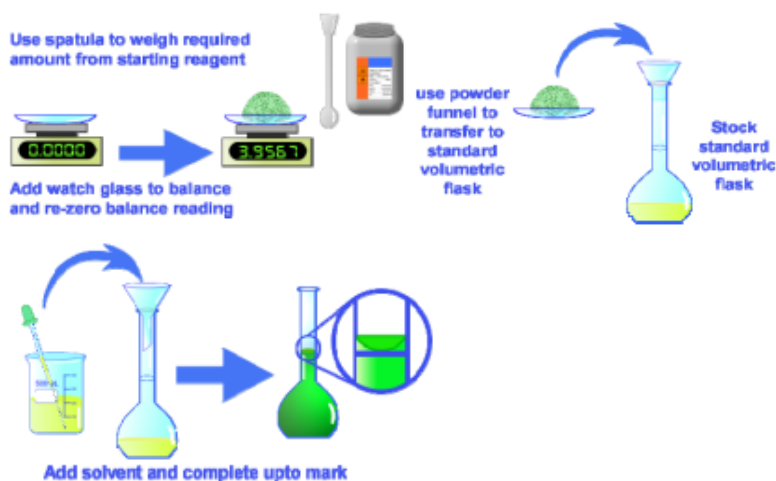


Figure 1: Preparing a stock solution of known molarity. (a) A measured number of moles of solute is weighed using analytical balance. (b) Solute is transferred in a volumetric flask. (c) Enough solvent is added to dissolve the solute by swirling and further solvent is carefully added until the calibration mark on the neck of the flask is reached, and the solution is then shaken until uniform.

Molarity can be used as a conversion factor to relate a solution's volume to the number of moles of solute. If we know the molarity and volume of a solution, we can calculate the number of moles of solute. If we know the number of moles of solute and the molarity of the solution, we can find the solution's volume.

### Examples

1. What is the molarity of a solution made by dissolving 2.355 g of sulfuric acid in water and diluting to a final volume of 50.0 mL?

### Solution

Molarity is the number of moles of solute per liter of solution. Thus it's necessary to find the number of moles of sulfuric acid in 2.355 g and then divide by the volume of the solution.

Molar mass of  $\text{H}_2\text{SO}_4 = (2 \times 1.0 \text{ g/mol}) + (1 \times 32.1 \text{ g/mol}) + (4 \times 16.0 \text{ g/mol}) = 98.1 \text{ g/mol}$

$$2.355 \text{ g H}_2\text{SO}_4 \times \frac{1 \text{ mol H}_2\text{SO}_4}{98.1 \text{ g H}_2\text{SO}_4} = 0.0240 \text{ mol H}_2\text{SO}_4$$

$$\text{Molarity} = \frac{\text{Moles of solute}}{\text{Volume of solution (L)}} = \frac{0.0240 \text{ mol H}_2\text{SO}_4}{0.0500\text{L}} = 0.480\text{M}$$

**The solution has a sulfuric acid concentration of 0.480 M**

2. Hydrochloric acid is sold commercially as a 12.0 M solution. How many moles of HCl are in 300.0 mL of 12.0 M solution?

**Solution**

The number of moles of solute is calculated by multiplying the molarity of the solution by its volume.

Moles of HCl= (Molarity of solution) × (Volume of solution L)

$$= \frac{12.0 \text{ mol HCl}}{1 \text{ L solution}} \times 0.300 \text{ L} = 3.60 \text{ mol HCl}$$

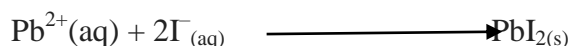
There are 3.60 mol of HCl in 300.0 mL of 12.0 M solution

Normality

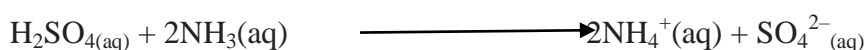
Normality is an older unit of concentration that, although once commonly used, is frequently ignored in today’s laboratories. Normality is still used in some handbooks of analytical methods, and, for this reason, it is helpful to understand its meaning. For example, normality is the concentration unit used in Standard Methods for the Examination of Water and Wastewater, and in some Standard EPA methods commonly used sources of analytical methods for environmental laboratories.

Normality makes use of the chemical equivalent, which is the amount of one chemical species reacting stoichiometrically with another chemical species. Note that this definition makes an equivalent, and thus normality, a function of the chemical reaction in which the species participates. Although a solution of H<sub>2</sub>SO<sub>4</sub> has a fixed molarity, its normality depends on how it reacts.

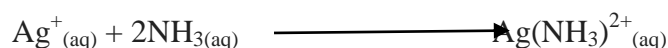
The number of equivalents,  $n$ , is based on a reaction unit, which is that part of a chemical species involved in a reaction. In a precipitation reaction, for example, the reaction unit is the charge of the cation or anion involved in the reaction; thus for the reaction



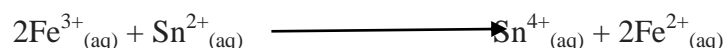
$n = 2$  for  $\text{Pb}^{2+}$  because each ion takes two electrons and  $n = 1$  for  $\text{I}^{-}$  because each ion donate only one electron. In an acid–base reaction, the reaction unit is the number of  $\text{H}^{+}$  ions donated by an acid or accepted by a base. For the reaction between sulfuric acid and ammonia



we find that  $n = 2$  for  $\text{H}_2\text{SO}_4$  because each molecule donate two ions of  $\text{H}^{+}$  and  $n = 1$  for  $\text{NH}_3$  because each ion accept one  $\text{H}^{+}$ . For a complexation reaction, the reaction unit is the number of electron pairs that can be accepted by the metal or donated by the ligand. In the reaction between  $\text{Ag}^{+}$  and  $\text{NH}_3$



the value of  $n$  for  $\text{Ag}^{+}$  is 2 because each ion accept pair of electrons in covalent bonds with Ammonia  $\text{NH}_3$  one electron from each covalent bond, for  $\text{NH}_3$  is  $n = 1$  because each molecule of ammonia donate one electron in each covalent bond it form with  $\text{Ag}^{+}$ . Finally, in an oxidation–reduction reaction there action unit is the number of electrons released by the reducing agent or accepted by the oxidizing agent; thus, for the reaction



$n = 1$  for  $\text{Fe}^{3+}$  because each ion accept one electron in the reduction step and  $n = 2$  for  $\text{Sn}^{2+}$  because each ion donate two electrons in the oxidation step. Clearly, determining the number of equivalents for a chemical species requires an understanding of how it reacts.

Normality is the number of equivalent weights (EW) per unit volume and, like formality, is independent of speciation. An equivalent weight is defined as the ratio of a chemical species' formula weight (FW) to the number of its equivalents

$$\text{Normality} = \frac{\text{Number of EWs solute}}{\text{litres of solution}}$$

$$\text{Number of EWs solute} = \frac{\text{Weight of solute}}{\text{Equivalent Weight (EW)}}$$

$$\text{Equivalent Weight (EW)} = \frac{\text{Formula Weight (g/mol)}}{n}$$

$$\text{Normality} = \frac{\text{Weight of solute}}{\text{Formula Weight} \times \text{liters of solution}}$$

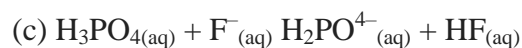
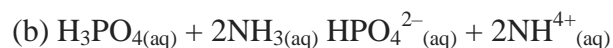
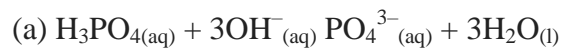
Consequently, the following simple relationship exists between normality and molarity.

$$N = n \times M$$

This equation is the simple form to fully understand the normality, you have to be able to determine the number of equivalents and calculate the molarity then use above equation to calculate the normality of the target analyte.

Examples

Calculate the equivalent weight and normality for a solution of 6.0 M H<sub>3</sub>PO<sub>4</sub> given the following reactions:



Solution

For phosphoric acid, the number of equivalents is the number of H<sup>+</sup> ion donated to the base.

For the reactions in (a), (b), and (c) the number of equivalents are 3, 2, and 1, respectively.

Thus, the calculated equivalent weights and normalities are

(a)  $EW = \frac{FW}{N} = \frac{97.994}{3} = 32.665 = \quad N = n \times M = 3 \times 6.0 = 18.0 \text{ N}$

(b)  $EW = \frac{FW}{N} = \frac{97.994}{2} = 48.997 = \quad N = n \times M = 2 \times 6.0 = 12.0 \text{ N}$

(c)  $EW = \frac{FW}{N} = \frac{97.994}{1} = 97.994 = \quad N = n \times M = 1 \times 6.0 = 6.0 \text{ N}$

## Molality

Molality is used in thermodynamic calculations where a temperature independent unit of concentration is needed. Molarity, formality and normality are based on the volume of solution in which the solute is dissolved. Since density is a temperature dependent property a solution's volume, and thus its molar, formal and normal concentrations, will change as a function of its temperature. By using the solvent's mass in place of its volume, the resulting concentration becomes independent of temperature.

Molality is defined as mole of solute in Kg of solvent as per equation:

$$\text{molality} = \frac{\text{moles of solute}}{\text{Kg of solvent}}$$

Example

**What is the molality of solution made by dissolve 25 g of NaCl in to 2.0 Liter of water.**

**Assume the density of water  $d = 1.0 \text{ g/mL} (= \text{kg/L})$ .**

Solution

$$\text{Molar mass of NaCl} = (1 \times 22.99 \text{ g/mol}) + (1 \times 35.45 \text{ g/mol}) = 58.44 \text{ g}$$

$$25 \text{ g of NaCl} \times \frac{1 \text{ mol NaCl}}{58.44 \text{ g NaCl}} = 0.428 \text{ mol of NaCl}$$

$$2.0 \text{ Litre water} \times \frac{1.0 \text{ kg}}{1.0 \text{ Litre water}} = 2.0 \text{ kg}$$

$$\text{molality} = \frac{\text{moles of solute}}{\text{Kg of solvent}} = \frac{0.428 \text{ mol NaCl}}{2.0 \text{ kg water}} = 0.214 \text{ m of NaCl}$$

The solution has concentration of NaCl equals of 0.214 m.

## Weight, Volume, and Weight-to-Volume Ratios

Weight percent (% w/w), volume percent (% v/v) and weight-to-volume percent (% w/v) express concentration as units of solute per 100 units of solution. A solution in which a solute has a concentration of 23% w/v contains 23 g of solute per 100 mL of solution. Parts per million (ppm) and parts per billion (ppb) are mass ratios of grams of solute to one million or

one billion grams of sample, respectively. For example, steel that is 450 ppm in Mn contains 450 mg of Mn for every kilogram of steel. If we approximate the density of an aqueous solution as 1.00 g/mL, then solution concentrations can be expressed in parts per million or parts per billion using the following relationships. For gases apart per million usually is a volume ratio. Thus, a helium concentration of 6.3 ppm means that one liter of air contains 6.3 mL of He.

Table 1 explains most common equations used in calculations of weight and volume ratios units. Note that the ppm or ppb units can be used as (w/w) or (w/v), for solutions if unit is not specified as (w/v) or (w/w) then (w/v) is assumed as default.

**Table 1.** Common equations used in calculations of weight and volume ratios units.

Concentration Unit	Equations
% w/w	$= \frac{g \text{ of solute}}{100 g \text{ of Solution}} = \frac{g \text{ of solute}}{g \text{ of Solution}} \times 100$
% v/v	$= \frac{mL \text{ of solute}}{100 mL \text{ of Solution}} = \frac{mL \text{ of solute}}{100 mL \text{ of Solution}} \times 100$
% w/v	$= \frac{g \text{ of solute}}{100 mL \text{ of Solution}} = \frac{g \text{ of solute}}{mL \text{ of Solution}} \times 100$
Part per million (ppm) w/v	$= \frac{mg \text{ of solute}}{L \text{ of Solution}} = \frac{\mu g \text{ of solute}}{mL \text{ of Solution}} = \frac{ng \text{ of solute}}{\mu L \text{ of Solution}}$
Part per million (ppm) w/w	$= \frac{mg \text{ of solute}}{Kg \text{ of Solution}} = \frac{\mu g \text{ of solute}}{g \text{ of Solution}} = \frac{ng \text{ of solute}}{mg \text{ of Solution}}$
Part per billion (ppb) w/v	$= \frac{\mu g \text{ of solute}}{L \text{ of Solution}} = \frac{ng \text{ of solute}}{mL \text{ of Solution}} = \frac{pg \text{ of solute}}{\mu L \text{ of Solution}}$
Part per billion (ppb) w/w	$= \frac{\mu g \text{ of solute}}{Kg \text{ of Solution}} = \frac{ng \text{ of solute}}{g \text{ of Solution}} = \frac{pg \text{ of solute}}{mg \text{ of Solution}}$

**w/w: Weight by weight**

**w/v: Weight by volume**

**v/v: Volume by volume**

Examples

How many grams of NaCl required preparing each of the following solutions?

a) 2500 ppm (w/v) NaCl 250 mL solution.

b) 10% (w/v) NaCl in 250 mL solution.

c) 20% (w/w) NaCl in 250 g solution.

Solution

a) 2500 ppm (w/v) NaCl

$$\text{Conc. (ppm)} = \frac{\text{mg of solute}}{\text{Volume of Solution (L)}}$$

$$\text{mg of solute} = \text{Conc. (ppm)} \times \text{Volume of Solution (L)}$$

$$\text{mg of NaCl} = 2500 \text{ ppm} \times 0.250 \text{ L} = 625 \text{ mg} = 0.625 \text{ g of NaCl}$$

b) 10% (w/v) NaCl

$$\text{Conc. \% (w/v)} = \frac{\text{g of solute}}{\text{mL of Solution}} \times 100\%$$

$$\text{g of solute} = \frac{\text{Conc. \% (w/v)} \times \text{mL of Solution}}{100\%}$$

$$\text{g of NaCl} = \frac{10\% \text{ (w/v)} \times 250 \text{ mL of Solution}}{100\%} = 25 \text{ g}$$

c) 20% (w/w) NaCl

$$\text{Conc. \% (w/w)} = \frac{\text{g of solute}}{\text{g of Solution}} \times 100\%$$

$$\text{g of solute} = \frac{\text{Conc. \% (w/w)} \times \text{g of Solution}}{100\%}$$

$$\text{g of NaCl} = \frac{20\% \text{ (w/w)} \times 250 \text{ g of Solution}}{100\%} = 50 \text{ g}$$

2. What is the concentration of  $\text{MgSO}_4$  the following prepared solution, express concentrations in ppm, % (w/v) and (w/w) concentrations units. Assume solution density is 1.0 g/mL 30 g of  $\text{MgSO}_4$  dissolved in 500 mL distilled water.

$$\text{Conc. (ppm)} = \frac{\text{mg of solute}}{\text{Volume of Solution (L)}} = \frac{30000 \text{ MgSO}_4}{0.500 \text{ L}} = 60000 \text{ ppm MgSO}_4$$

$$\text{Conc. \% (w/v)} = \frac{\text{g of solute}}{\text{mL of Solution}} \times 100\% = \frac{30 \text{ g MgSO}_4}{500 \text{ mL}} \times 100\% = 6\% \text{ (w/v) MgSO}_4$$

When solution density is 1.0 g/mL then

$$\text{Conc. \%}(w/v) = \frac{\text{g of solute}}{\text{g of Solution}} \times 100\% = \frac{30\text{g MgSO}_4}{500\text{g}} \times 100\% = 6\% (w/w)\text{MgSO}_4$$

#### 4.0 Conclusion

Preparation of reagents from stock solution is an essential skill for an Environmental Laboratory Scientist. In most cases reagents needed for an analysis may be prepared from stock solutions with higher concentration than what is required.

#### 5.0 Summary

In this unit, we discussed the methods of preparing solutions from stock reagents.

#### 6.0 Tutor-Marked Assignment

1. Define the following: Stock standard solution, Standard Solution, Molar Solution.
2. Differentiate between molarity and formality?
3. Calculate the molar concentration of commercial trioxonitrate (V) acid of specific gravity 1.42 and 70.0% of acid ( $\text{HNO}_3 = 63.0$ )
4. How would you prepare 2 molar  $(\text{NH}_4)_2\text{CO}_3$ ?

#### 5.0 References / Further Reading

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## UNIT 3: CONVERTING BETWEEN CONCENTRATION UNITS

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3.0 Main content

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3.2: Dilution of Concentrated Solutions

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### **1.0 Introduction**

Many scientific activities involve numerical calculations - measuring, weighing, preparing solutions, and so forth—and it's often necessary to convert a quantity from one unit to another. The simplest way to carry out calculations that involve different units is to use the dimensional-analysis method.

### **2.0 Objectives**

At the end of this unit, the students should be able to carry out conversions between concentration units.

### **3.0 Main content**

#### **3.1 Dimensional analysis method**

In this method, a quantity described in one unit is converted into an equivalent quantity with a different unit by using a conversion factor to express the relationship between units:

$$\textit{Original quantity} \times \textit{Conversion factor} = \textit{Equivalent quantity}$$

As an example, we know that 1 ppm equals 1000 ppb. Writing this relationship as a fraction restates it in the form of a conversion factor, either ppm per ppb or ppb per ppm:

Conversion factor between ppm and ppb

$$\frac{1 \text{ ppm}}{1000 \text{ ppb}} \text{ equals to } \frac{1000 \text{ ppb}}{1 \text{ ppm}} \text{ equals } 1$$

Note that this and all other conversion factors are numerically equal to 1 because the value of the quantity above the division line (the numerator) is equal to the value of the quantity below the division line (the denominator). Thus, multiplying by a conversion factor is equivalent to multiplying by 1 and so does not change the value of the quantity.

The key to the dimensional-analysis method of problem solving is that units are treated like numbers and can thus be multiplied and divided (though not added or subtracted) just as numbers can. The idea when solving a problem is to set up an equation so that unwanted units cancel, leaving only the desired units.

Usually it's best to start by writing what you know and then manipulating that known quantity. For example, if you know the volume is 153 mL and want to find it in deciliter (dL), you can write down the volume in mL and set up an equation multiplying the mL by the conversion factor in mL per dL:

$$153 \text{ mL} \times \frac{1 \text{ dL}}{100 \text{ mL}} = 1.53 \text{ dL}$$

The unit "mL" cancels from the left side of the equation because it appears both above and below the division line, and the only unit that remains is "dL" The dimensional-analysis method gives the right answer only if the equation is set up so that the unwanted units cancel.

The main drawback to using the dimensional-analysis method is that it's easy to get the right answer without really understanding what you're doing. It's therefore best after solving a problem to think through a rough estimate, or "ballpark" solution, as a check on your work. If your ballpark check isn't close to the detailed solution, there's a misunderstanding somewhere, and you should think the problem through again. Even if you don't make an estimate, it's important to be sure that your calculated answer makes sense. The dimensional-

analysis method and the use of ballpark checks are techniques that will help you solve problems of many kinds, not just unit conversions.

Conversion between concentration units is so easy once you understand the above conversion principles, the units of concentration most frequently encountered in analytical chemistry are molarity, weight percent, volume percent, weight-to-volume percent, parts per million, and parts per billion. By recognizing the general definition of concentration given, it is easy to convert between concentration units. Table 1 shows relations between most commonly volume units.

Table 1: Conversion between most commonly used volume units.

Unit	Symbol	Relation	Remark
Liter	L	1L = 1000 mL	
deciLiter	dL	1 dL = 100 mL 1 L = 10 dL	deci = $10^{-1}$ deciLiter = $10^{-1}$ L
milliLiter	mL	1 L = 1000 mL 1 dL = 100 mL	milli = $10^{-3}$ milliLiter = $10^{-3}$ L
Cubic meter	m <sup>3</sup>	1 m <sup>3</sup> = 1000 L 1 m <sup>3</sup> = 10,000 dL	
Cubic decimeter	dm <sup>3</sup>	1 m <sup>3</sup> = 1000 dm <sup>3</sup> 1 m <sup>3</sup> = 1000 L 1 dm <sup>3</sup> = 1 L	deci = $10^{-1}$ (deci...) <sup>3</sup> = $10^{-3}$ Cubic decimeter = $10^{-3}$ m <sup>3</sup>
Cubic Centimeter	cm <sup>3</sup>	1 m <sup>3</sup> = 1000 dm <sup>3</sup> 1 dm <sup>3</sup> = 1000 cm <sup>3</sup> 1 L = 1 dm <sup>3</sup> 1 L = 1000 mL 1 mL = 1 cm <sup>3</sup>	centi = $10^{-2}$ (centi...) <sup>3</sup> = $10^{-6}$ Cubic centimeter = $10^{-6}$ m <sup>3</sup>

### Example

A concentrated solution of aqueous ammonia is 28.0% w/w NH<sub>3</sub> and has a density of 0.899 g/mL. What is the molar concentration of NH<sub>3</sub> in this solution?

### Solution

We will use the general conversion equation starting from information we have, 28 % w/w means for every 100 g of solution it contain 28 g ammonia NH<sub>3</sub>, density of solution 0.899 g/mL and molecular weight of ammonia NH<sub>3</sub> is 17.03 g/mol. To convert to molarity we should reach the molarity expression moles per Litre:

$$\text{Molarity} = \frac{\text{Moles of solute}}{\text{Volume of solution (L)}}$$

To reach this expression we will put all possible conversion factors we will use, note Table 2 shows two types for each conversion factor (factor 1) and (factor 2). Both are same but you have to justify it in the way that units cancel each other until you reach units expression you want (mol/L).

**Table 2: All possible conversion factors we expect to use. Use factor 1 or factor 2 from each type:**

Factor Description	Factor 1	Factor 2
Solution Density (g/mL)	$\frac{0.899 \text{ g Solution}}{1 \text{ mL Solution}}$	$\frac{1 \text{ mL Solution}}{0.899 \text{ g Solution}}$
Molecular Weight (g/mol)	$\frac{1 \text{ mol NH}_3}{17.03 \text{ g NH}_3}$	$\frac{17.03 \text{ g NH}_3}{1 \text{ mol NH}_3}$
Volume Conversion (mL/L)	$\frac{1000 \text{ mL Solution}}{1 \text{ L Solution}}$	$\frac{1 \text{ L Solution}}{1000 \text{ mL Solution}}$

If we arrange above conversion factors the following expression will be:

$$\frac{28 \text{ g}}{100 \text{ g of solution}} \times \frac{0.899 \text{ g Solution}}{1 \text{ mL solution}} \times \frac{1 \text{ mol NH}_3}{17.03 \text{ g NH}_3} \times \frac{100 \text{ mL solution}}{1 \text{ L solution}} = 14.78 \text{ (mol/L)M NH}_3$$

The first part of the left side is the concentration that we have and the rest three are conversion factors.

To solve this problem using general molarity equations you have to assume you have 1 L of ammonia solution and continue you calculations based on this assumption and you will get the same result.

### 3.2 Dilution of Concentrated Solutions

Dilution is one of the main preparation processes which used daily in all laboratories; any chemist should be familiar with dilution calculations to prepare a correct diluted solution. For convenience, chemicals are sometimes bought and stored as concentrated solutions that must be diluted before use. Aqueous hydrochloric acid, for example, is sold commercially as a 12.0

M solution, yet it is most commonly used in the laboratory after dilution with water to a final concentration of 6.0 M or 1.0 M.

Concentrated Solution + Solvent Diluted Solution  $\longrightarrow$  Dilute solution

The key fact to remember when diluting a concentrated solution is that the number of moles of solute is constant; only the volume is changed by adding more solvent. Because the number of moles of solute can be calculated by multiplying molarity times volume, we can set up the following equation:

$$\begin{aligned} \text{Moles of solute (constant)} &= \text{Molarity} \times \text{Volume} \\ &= M_i \times V_i = M_f \times V_f \end{aligned}$$

Where  $M_i$  is the initial molarity,  $V_i$  is the initial volume,  $M_f$  is the final molarity, and  $V_f$  is the final volume after dilution. Rearranging this equation into a more useful form shows that the molar concentration after dilution ( $M_f$ ) can be found by multiplying the initial concentration ( $M_i$ ) by the ratio of initial and final volumes ( $V_i/V_f$ ):

$$M_f = M_i \times \frac{V_i}{V_f}$$

$V_f/V_i$  is called Dilution Factor:

$$M_f = \frac{M_i}{DF}$$

$$M_i = M_f \times DF$$

$$\text{Where } DF \text{ (Dilution Factor)} = \frac{V_f}{V_i} = \frac{M_i}{M_f}$$

Suppose, for example, that we dilute 50.0 mL of a solution of 2.00 M to a volume of 200.0 mL. The solution volume increases by a factor of four (from 50 mL to 200 mL), so the concentration of the solution must decrease by a factor of four (from 2.00 M to 0.500 M):

$$M_f = 2.00 \text{ M} \times \frac{50.0 \text{ mL}}{200.0 \text{ mL}} = 0.500 \text{ M}$$

## 4.0 Conclusion

Conversion between different concentration units and dilution techniques are important components of reagent preparation.

## 5.0 Summary

In this unit, we discussed the conversion between different concentration units and how to prepare dilute solutions.

## 6.0 Tutor Marked Assignment

1. How would you prepare 500.0 mL of 0.2500 M NaOH solution starting from a concentration of 1.000M?
2. How many grams of Sodium Persulfate ( $\text{Na}_2\text{S}_2\text{O}_8$ ) required to prepare a 1 L solution of Sodium Persulfate with concentration of 10% (w/v). This solution is widely used as oxidizing reagent for Total Organic Carbon analyzer (TOC).
3. What is the concentration of Hydrochloric Acid (HCl) in Molarity unit (M), if the following information written on the bottle: M.Wt = 36.46 g/mol,  $d = 1.18 \text{ g/cm}^3$ , 37% (w/w).
4. How do you prepare a 500 mL solution of 2.0 N Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) from concentrated bottle of Sulfuric acid. If the following information written on Sulfuric acid bottle: M. Wt = 98.08 g/mol,  $d = 1.84 \text{ g/cm}^3$ , 97% (w/w).

## 7.0 References/Further Reading

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## **UNIT 4: STORAGE OF LABORATORY REAGENTS**

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### **1.0 Introduction**

Proper storage of laboratory reagents is one way to maintain a good laboratory practice. Storage of laboratory reagents when not properly handled can lead to different types of accidents in the laboratory. The accidents can range from explosions and fire outbreaks.

### **2.0 Objectives**

At the end of this unit, the students should be able to understand and be conversant with the process of storage of laboratory reagents.

### **3.0 Main content**

#### **3.1 Glassware/Polyethenewares/Other Laboratory wares**

Generally, glassware of borosilicate glass, which is relatively inert, is used for the analytical work. Plastic bottles of polythene (PE) or polypropylene (PP) are suitable for collecting and transporting water samples. Unless instructed otherwise, borosilicate glass bottles may be used for the storage of reagents and standard solutions. Standard solutions of silica, boron and

alkali metals should be stored in polyethylene bottles. Whenever necessary, amber or dark coloured glass bottles must be used for storing photo-reactive chemical solutions.

### **3.2 Recommendations for Storage of Laboratory Chemicals**

The following general suggestions for safe storage of chemicals in the laboratory should be implemented.

1. The quantities of chemicals that are stored within a laboratory should be minimized, as specified by NFPA (National Fire Protection Association) 45 and OSHA (Occupation Safety and Health Administration). Many authorities recommend that the NFPA guidelines for maximum quantities and sizes of containers should be reduced to one-half or even one-third of the recommended values.
2. Bulk quantities of chemicals (i.e., larger than one-gallon) must be stored in a separate storage area.
3. Transfer of flammable liquid from 5 gallon or larger metal containers may not be done in the laboratory.
4. Chemicals must be stored at an appropriate temperature and humidity level. This can be especially problematic in hot, humid climates. As a rule, chemicals should not be stored near heat sources, such as steam pipes or laboratory ovens.
5. Chemicals should never be stored indirect sunlight.
6. Chemicals should be dated when received and when opened. If the chemical is one that degrades in quality or becomes unsafe after prolonged storage, the shelf-life expiration date should also be included.
7. Visual inspection of the material and its container should be conducted routinely. Indications for disposal include: cloudiness in liquids material changing color evidence of liquids in solids or solids in liquids "puddling" of material around outside of container o pressure build-up within bottle obvious deterioration of container.



8. Chemicals should not be routinely stored on the bench tops. In such locations they are unprotected from exposure and participation in a fire situation and are also more readily knocked over.
9. Each chemical should have a specific storage area and be returned there after use. Large quantities of flammable materials should not be stored in the laboratory. Only the amounts needed should be kept on bench tops, the remainder should be kept in flammable storage cabinets.
10. Laboratory shelves should have a raised lip along the outer edge to prevent containers from falling.
11. Never allow the container to hang off the edge of the shelf!
12. Liquid or corrosive chemicals should never be stored on shelves above eye-level.
13. Glass containers should not touch each other on the shelves.
14. Secondary containers or trays should be used for chemical storage whenever possible to minimize the flow of material should a spill or rupture occurs.
15. Round bottom flasks should always be supported properly in cork rings or by other means to keep them from tipping.
16. Adequate security must be provided so that unauthorized personnel do not have access to hazardous materials.
17. Chemicals must never be stored on the floor, not even temporarily!
18. Chemicals that are no longer to be used for research purposes should be properly disposed of or given to another research group that has a use for it.
19. Flammable materials must never be stored in domestic-type refrigerators. Only explosion- flammable material refrigerators should be used for storage of these chemicals within a laboratory environment.

20. All containers stored within the refrigerator should be tightly capped to keep vapors from interacting with each other and to alleviate "smell" problems.
21. Flasks with cork, rubber or glass stoppers should be avoided because of the potential for leaking.
22. All containers stored in the refrigerator must be properly labeled.
23. Inventory the materials in your refrigerator frequently to avoid overcrowding with materials that have long since been forgotten.
24. Also make it a point to defrost your refrigerator occasionally so that chemicals do not become trapped in unique ice formations!
25. Before flammable materials are stored in a refrigerator, it should be determined if keeping the material chilled will serve any purpose. No benefit is derived from refrigerating a chemical that has a flash point below the temperature of the refrigerator.
26. Never store peroxide formers (i.e., ether) in a refrigerator!
27. Fume hoods should not be used as general storage areas for chemicals. This may seriously impair the ventilating capacity of the hood.
28. Gas cylinders must be securely strapped to a permanent structure (wall, lab bench, etc.). When they are not in use they should be capped off.
29. On termination, graduation or transfer of any laboratory personnel, all hazardous materials must be properly disposed of, or arrangements made to transfer them to the laboratory supervisor.

### **3.3 Segregation of Chemicals Based on Hazard Classes**

In addition to general safe storage practices, segregated storage of incompatible materials is a must. As a minimum, laboratories should separate chemicals according to similar hazards,

such as flammability, and corrosivity, sensitivity to water or air, and toxicity. The following major categories of chemicals are strongly recommended:

1. Flammables
2. Oxidizers
3. Corrosives acids bases
4. Highly Reactive Extreme Toxics/Regulated Materials
5. Low Hazard

However, problems may arise with a general segregation of chemicals. Below, you will find a few of these potential problems.

**The actual identification of the hazards themselves:** Recent legislation has made this task somewhat easier since all chemical manufacturers are now required to list all hazards on outgoing chemical containers and each chemical must be accompanied by a Safety Data Sheet(SDS). The chemical label thus furnishes a quick method of determining whether the material is a fire hazard, health hazard or reactivity hazard. The SDS furnishes more detailed information regarding toxicity exposure levels, flashpoints, required safety equipment and recommended procedures for spill containment.

**Multiple hazards for chemicals:** Most chemicals have multiple hazards and a decision must be made as to which storage area would be most appropriate for each specific chemical. First you have to determine your priorities! When establishing a storage scheme, the number one consideration should be the flammability characteristics of the material. If the material is flammable, it should be stored in a flammable cabinet. If the material will contribute significantly to a fire (i.e., oxidizers), it should then be isolated from the flammables. If there were a fire in the lab and response to the fire with water would exaggerate the situation, isolate the water reactive material away from contact with water. Next look at the corrosivity of the material, and store accordingly.

Finally, consider the toxicity of the material, with particular attention paid to regulated materials. In some cases, this may mean that certain chemicals will be isolated within a storage area, for instance, a material that is an extreme poison but is also flammable, should be locked away in the flammable storage area to protect it against accidental release. There will always be some chemicals that will not fit neatly in one category or another, but with careful consideration of the hazards involved, most of these cases can be handled in a reasonable fashion. For the safety of all personnel and to protect the integrity of the facilities, hazardous materials must be segregated.

### **3.4 Peroxide-Forming Materials**

Peroxides are very unstable and some chemicals that can form them are commonly used in laboratories. This makes peroxide-forming materials some of the most hazardous substances found in a lab. Peroxide-forming materials are chemicals that react with air, moisture, or impurities to form peroxides. The tendency to form peroxides by most of these materials is greatly increased by evaporation or distillation. Organic peroxides are extremely sensitive to shock, sparks, heat, friction, impact, and light. Just the friction from unscrewing the cap of a container of an ether that has peroxides in it can provide enough energy to cause a severe explosion.

#### **Use and Storage of Peroxide-Formers**

Do not open the chemical container if peroxide formation is suspected. The act of opening the container could be sufficient to cause a severe explosion. Visually inspect liquid peroxide forming materials for crystals or unusual viscosity before opening. Pay special attention to the area around the cap. Peroxides usually form upon evaporation, so they will most likely be formed on the threads under the cap.

Date all peroxide forming materials with the date received, and the expected shelf life. Chemicals such as diisopropyl ether, divinyl acetylene, sodium amide, and vinylidene

chloride should be discarded after three months. Chemicals such as dioxane, diethyl ether, and tetrahydrofuran should be submitted to Waste Management Department for disposal after one year. Store all peroxide-forming materials away from heat, sunlight, and sources of ignition. Sunlight accelerates the formation of peroxides. Secure the lids and caps on these containers to discourage the evaporation and concentration of these chemicals.

Never store peroxide-forming materials in glass containers with screw cap lids or glass stoppers. Friction and grinding must be avoided. Also, never store these chemicals in a clear glass bottle where they would be exposed to light.

#### **4.0 Conclusion**

Requirements for the storage of reagents are based on the type of reagents. Some reagents are not stable and some are sensitive (e.g. to light), these have special requirements that must be followed for their storage. Some chemicals also cannot be stored together.

#### **5.0 Summary**

In this unit, we discussed the requirements for the storage of solutions and reagents.

#### **6.0 Tutor Marked Assignment**

1. Which type of glassware would you recommend for an analytical work? Give reasons.
2. Mention the different hazard classes of chemicals.
3. How can peroxides forming chemicals be safely stored?

#### **7.0 References/Further Reading**

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School Chemistry Laboratory Safety Guide, Oct 2006; p 6

[www.cpsc.gov/Page\\_Files/122344/NIOSH2007107.pdf](http://www.cpsc.gov/Page_Files/122344/NIOSH2007107.pdf)

[www.flinnsci.com/media/396468/safety\\_contract\\_hs.pdf](http://www.flinnsci.com/media/396468/safety_contract_hs.pdf)

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## **MODULE 4: INSTRUMENTATION IN ENVIRONMENTAL LABORATORY SCIENCE**

Unit 1: Instrumentation in Environmental Laboratory Science

Unit 2: Noise, sound and radiation measuring instruments

Unit 3: Polymerase Chain Reaction

Unit 4: Cryostat

### **UNIT 1: INSTRUMENTATION IN ENVIRONMENTAL LABORATORY SCIENCE**

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#### **1.0 Introduction**

Instrumentation is concerned with the techniques in Environmental Sciences, which involves data collection, manipulation and information presentation. Instrumentation is used in almost every activity and generating system, where consistent and reliable operations are required. Instrumentation provides the means of monitoring, recording and controlling a process to maintain it at a desired state. A typical environmental activity yields many process variables that have to be measured and manipulated. Variables such as boiler level, temperature and pressure, turbine speed, generator output and many others have to be controlled prudently to

ensure a safe and efficient station operation. Instrumentation is often referred to as the power behind scientific investigations. This unit introduces you to instrumentation used in Environmental Science.

## **2.0 Objectives**

At the end of this unit, the students should be able to understand instrumentation

## **3.0 Main content**

### **3.1: What is instrumentation?**

Instrumentation can be generally described as the study, development and manufacture of scientific instruments and equipment. But for the sake of this course, we will simply define instrumentation as instruments and procedures used in collecting and analyzing data in a study.

### **3.2 Types of Instrumentation for Environmental Studies**

Different types of data are required for different categories of environmental studies; hence different instruments and procedures for their data collection and analyses exist too. The different methods of data acquisition can be summarily described as the following:

#### **a. Instrumentation for field observations:**

Instrumentation for these includes land survey equipment, both analogue and digital. Instruments for analogue approach to field survey include tape, arrow, Günter chain, etc. Digital instruments include Total Stations, Global Positioning Systems (GPS).

There are also some indirect methods of extracting information, especially from aerial photographs and satellite imageries.

Other categories of instrument for field surveys include samplers, which are of various types depending on the parameter to sample; air, water, sediment and soil samplers. We also have testers or field equipment for direct observation, including pH and electrical conductivity meters, quadrats; for vegetation sampling, tape, ball of string, scissors, clipboard, pens and paper.



## **b. Instrumentation for laboratory analyses**

In a particular application the selection of a technique will be based on the particular requirements:

1. what species (parameters) are to be measured;
2. is the simultaneous determination of several parameters necessary; and
3. what are the required accuracy, time resolution, and spatial resolution?
4. logistic requirements like power consumption, mounting of light sources or retro-reflectors or accommodation of the instrument on mobile platforms.

### **3.3 Classification of laboratory equipment**

Laboratory techniques are both specialized and universal techniques. Specialized techniques allow only one parameter (specie) to be detected by an instrument, e.g. UV absorption detection. On the other hand universal techniques allow one to measure many species with one instrument.

Another fundamental property of instruments is the spatial range of the measurements, usually expressed in terms of in situ or remote sensing measurements. While in situ measurements come close to the ideal to determine specie concentrations in a 'spot' in space that is usually very close to the instrument, remote sensing techniques allow one to make measurements from a large distance, perhaps as far as from a satellite instrument in the earth's orbit. It thus usually give averages of the concentration over a relatively large area.

Remote sensing techniques always rely on the sensing of electromagnetic radiation.

Examples of laboratory instruments belonging to either category include

- (i) gas chromatographic (universal technique, in situ),
- (ii) optical spectroscopy (universal technique, in situ and remote sensing),
- (iii) mass spectrometry (MS),

(iv) any other (in situ) technique, where the most commonly employed principles include chemiluminescence (e.g. for the detection of NO or O), photoacoustic detection, electrochemical techniques, matrix isolation), and chromatography (IC)

It is important to note that field surveys equipment such as total stations, GPS and aerial and satellite sensors are also remote sensing devices. This is because they are a devices used to obtain information from the real world surface without being in physical contact with the object from which information is being obtained.

#### **4.0 Conclusion**

This unit has introduced you to the concept of instrumentation in Environmental Science. Environmental analyses depend on both the theoretical and technical principles of this important concept.

#### **5.0 Summary**

So far you have learnt that instrumentation is a term used to describe a collection of instruments and procedures used in collecting and analyzing data in a study. Instrumentation is used for field observations and laboratory analysis. It is based on results from these activities using required instrumentation that results could be obtained and presented.

#### **6.0 Tutor-Marked Assignment**

1. What is instrumentation?
2. Describe the two categories of instrumentation in Environmental Science.
3. List 5 examples of field and laboratory equipment that use remote sensing technique.

#### **7.0 References/Further Reading**

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## **Unit 2: NOISE, SOUND AND RADIATION MEASURING INSTRUMENTS**

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3.2: Sound Level Meter

3.3: Dosimeter: Noise Measurement

3.4: Dosimeter: Radiation Measurement

4.0 Conclusion

5.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

### **1.0 Introduction**

There is no single method or process for measuring noise. Hearing safety and health professionals can however use a variety of instruments to achieve this. The choice of a particular instrument and approach for measuring and analyzing noise depends on many factors, not the least of which will be the purpose for the measurement and the environment in which the measurement will be made.

### **2.0 Objectives**

At the end of this unit, the students should be able to know how to measure noise, sound and radiation using the measuring instruments.

### **3.0 Main content**

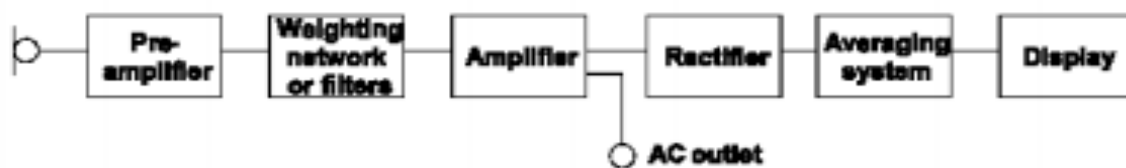
#### **3.1: Components of Sound Measuring System**

Many types of measuring systems can be used for the measurement of sound depending on the purpose of the study, the characteristics of sound and the extent of information that is desired about the sound.

The various elements in a measuring system are:

- the transducer; that is, the microphone;
- the electronic amplifier and calibrated attenuator for gain control;
- the frequency weighting or analyzing possibilities;
- the data storage facilities;
- the display.

Not all elements are used in every measuring system. The microphone can, for instance, be connected to a sound level meter or directly to a magnetic tape recorder for data storage and future measurement or reference. An example of the components of the sound level meter is shown in the Figure below



**Figure 1: Sound level meter block diagram**

The two main characteristics are:

- i. The frequency response: that is, the deviation between the measured value and the true value as a function of the frequency. As the ear is capable of hearing sounds between 20 Hz and 20 kHz, the frequency response of the sound level meter should be good, with variations smaller than 1 dB, over that range;
- ii. The dynamic range: that is, the range in dB over which the measured value is proportional to the true value, at a given frequency (usually 1000 Hz). This range is limited at low levels by the electrical background noise of the instrument and at high levels by the signal distortion caused by overloading the microphone or amplifiers.

### 3.2 Sound Level Meter

The sound level meter is the basic measuring instrument for noise exposures. It consists of a microphone, a frequency selective amplifier, and an indicator. At a minimum, it measures sound level in dB SPL. An integrating function may be included to automate the calculation of the noise dose.

### Audiometer

An audiometer is a machine used for evaluating hearing acuity. They usually consist of an embedded hardware unit connected to a pair of headphones and a test subject feedback button, sometimes controlled by a standard PC. Such systems can also be used with bone vibrators, to test conductive hearing mechanisms.



(a) Portable audiometer Tetra-Tone Model EB-46, circa 1975 (b) Portable audiometer Maico, circa 1960s-

Audiometers are standard equipment at ENT (ear, nose, and throat) clinics and in audiology centers. An alternative to hardware audiometers are software audiometers, which are available in many different configurations. Screening PC-based audiometers use a standard computer. Clinical PC-based audiometers are generally more expensive than software audiometers, but are much more accurate and efficient. They are most commonly used in

hospitals, audiology centers and research communities. These audiometers are also used to conduct industrial audiometric testing. Some audiometers even provide a software developer's kit that provides researchers with the capability to create their own diagnostic tests.

### **Functionality**

An audiometer typically transmits recorded sounds such as pure tones or speech to the headphones of the test subject at varying frequencies and intensities, and records the subject's responses to produce an audiogram of threshold sensitivity, or speech understanding profile.

### **Types**

Medical grade audiometers are usually an embedded hardware unit controlled from a PC. Software audiometers which run on a PC are also commercially available, but their accuracy and utility for evaluating hearing loss is questionable due to lack of a calibration standard.

The most common type of audiometer generates pure tones, or transmits parts of speech. Another kind of audiometer is the Bekesy audiometer, in which the subject follows a tone of increasing and decreasing amplitude as the tone is swept through the frequency range by depressing a button when the tone is heard and releasing it when it cannot be heard, crossing back and forth over the threshold of hearing. Bekesy audiometry typically yields lower thresholds and standard deviations than pure tone audiometry.

### **3.3 Dosimeter: Noise Measurement**

Measuring noise with a sound level meter is relatively simple when the noise levels are continuous and when the person remains essentially stationary during the work shift. A noise dosimeter is preferred for measuring a person's noise exposure when the noise levels are varying or intermittent, when they contain impulsive components, or when the worker moves around frequently during the work shift.

The noise dosimeter may be thought of as a sound level meter with an additional storage and computational function. It measures and stores the sound levels during an exposure period and computes the readout as the percent dose. Many dosimeters available today can provide an output in dose using various exchange rates (e.g., 3, 4, and 5 dB), 8-hr criterion levels (e.g., 80, 84, 85, and 90 dBA), and sound measurement ranges (e.g., 80 to 130dBA).

In noise dosimetry, the microphone is attached on the worker whose exposure is being measured. The microphone is usually located on the mid top of the person's more exposed shoulder and is oriented approximately parallel to the plane of this shoulder.

### 3.4 Dosimeter: Radiation Measurement

There are dosimeters used for the measurement of the amount of energy deposited by ionizing radiation. This measurement is used to estimate the effective dose received by the human body through exposure to external ionizing radiation.

The different types of dosimeter and systems include: personal dosimeters, operational dosimeters, extremities dosimeters (usually for hands, feet or eyes).



Figure (a) Personal dosimeter      (b) Operational dosimeters      (c) Extremity dosimeters

**Personal dosimeters** are required by people who work in Radiation Areas. This dosimeter is personal and not transferable. The personal dosimeter is sometimes referred to as: legal dosimeter, passive dosimeter, individual or DIS dosimeter. In the past, when a different technology was used, this dosimeter was called a 'film badge'.

The personal dosimeter actually includes two dosimeters: one to measure gamma and beta radiation and a second one to measure neutron radiation. When a dosimeter is read, only the gamma/beta dosimeter readout value is shown. The neutron dosimeter is evaluated on the



annual exchange of the dosimeter. The complete result of gamma/beta and neutron dose is usually displayed on official records of people who work in radiation zones after evaluation and validation scientists.

**Operational dosimeter** is required, in addition to personal dosimeter, for working in Limited Stay and High Radiation Controlled Radiation Areas.

The operational dosimeter is sometimes referred to as: active dosimeter, electronic dosimeter or DMC. The operational dosimeter features a direct dose display, audible indication of the radiation level and alarm functions when thresholds for dose or dose rates are exceeded. Operational dosimeter can either be assigned permanently to a person or can be used in 'pool' mode. For the latter, the dosimeter is not assigned to a specific person and can be shared amongst colleagues.

**Extremity dosimeters** are provided for interventions where higher doses to the extremities (hands, feet) and/or eyes are likely to occur. A typical extremity dosimeter would be a finger ring integrating a thermoluminescence-based detector. The finger ring dosimeter has a detection limit of about 0.2-0.3 mSv. Doses are reported for

#### **4.0 Conclusion**

This Unit has discussed what you need to know about the basis of noise, sound and radiation measurements.

#### **5.0 Summary**

Noise, sound and radiation probing equipment have been discussed. These include: audiometer, sound level meters, noise and radiation dosimeters.

#### **6.0 Tutor-Marked Assignment**

1. What are the components of a typical sound measuring system?
2. What are the characteristics of sound measuring system?
3. Different between dosimeters in the measurement of noise and radiation.

## **7.0 References / Further Reading**

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## **UNIT 3: POLYMERASE CHAIN REACTION (PCR)**

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1.0 Introduction

2.0 Objectives

3.0 Main content

3.1: Polymerase Chain Reaction (PCR) Techniques

3.2: Steps in carrying out PCR

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5.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

### **1.0 Introduction**

From the isolation of specific genes to the sequencing of entire genomes, the polymerase chain reaction (PCR) has become one of the most widely used technologies for conducting biological research. Advances have led to the development of specific and sensitive high-throughput PCR methods for the detection of a variety of microorganisms, and these methods are increasingly being applied to analysis of environmental samples.

### **2.0 Objectives**

At the end of this unit, the students should be able to know the concept of the PCR and its use in the Environmental Health Laboratory.

### **3.0 Main content**

#### **3.1 Polymerase Chain Reaction (PCR) Techniques**

Polymerase chain reaction (PCR) is a common laboratory technique used to make many copies (millions or billions!) of a particular region of DNA. Typically, the goal of PCR is to

copies (millions or billions!) of a particular region of DNA. Typically, the goal of PCR is to make enough of the target DNA region that it can be analyzed or used in some other way. For instance, DNA amplified by PCR may be sent for sequencing, visualized by gel electrophoresis, or cloned into a plasmid for further experiments. PCR is used in many areas of biology and medicine, including molecular biology research, medical diagnostics, and even some branches of ecology. PCR can be used for the detection of microorganisms in the environmental samples such as: detection of waterborne microbial pathogens e.g. *Legionella*, *Vibrio vulnificus* and *Giardia*; detection of microorganism in soil sample, wastewater and effluents.

Especially, the bacteriological safety of water supplies is tested by monitoring coliform bacteria whose presence in the water indicates potential human fecal contamination and the possibility of the presence of enteric pathogens. Coliform bacteria are traditionally detected by culturing on media such as MacConkey, m-Endo, Eosin methylene blue, or Brilliant-green-lactose-bile media. Bej *et al.* have developed a PCR gene probe-based method for the detection of coliform bacteria. Amplification of a portion of the *lacZ* gene detects *E. coli* and other coliform bacteria; including *Shigella spp.* Amplification of part of the *lamB* gene detects *E. coli*, *Sahnonella*, and *Shigella spp.*

The key ingredients of a PCR reaction are Taq polymerase, primers, template DNA, and nucleotides (DNA building blocks). The ingredients are assembled in a tube, along with cofactors needed by the enzyme, and are put through repeated cycles of heating and cooling that allow DNA to be synthesized.

### **3.2 Steps in carrying out PCR**

The basic steps are:

Denaturation (96°C): Heat the reaction strongly to separate, or denature, the DNA strands.

This provides single-stranded template for the next step.

Annealing (55 - 65°C): Cool the reaction so the primers can bind to their complementary sequences on the single-stranded template DNA.

Extension (72°C): Raise the reaction temperatures so Taq polymerase extends the primers, synthesizing new strands of DNA.

This cycle repeats 25 - 35 times in a typical PCR reaction, which generally takes 2 - 4 hours, depending on the length of the DNA region being copied. If the reaction is efficient (works well), the target region can go from just one or a few copies to billions.

The results of a PCR reaction are usually visualized (made visible) using gel electrophoresis. Gel electrophoresis is a technique in which fragments of DNA are pulled through a gel matrix by an electric current, and it separates DNA fragments according to size. A standard, or DNA ladder, is typically included so that the size of the fragments in the PCR sample can be determined.

DNA fragments of the same length form a "band" on the gel, which can be seen by eye if the gel is stained with a DNA-binding dye. For example, a PCR reaction producing a 400 base pair (bp) fragment would look like this on a gel:

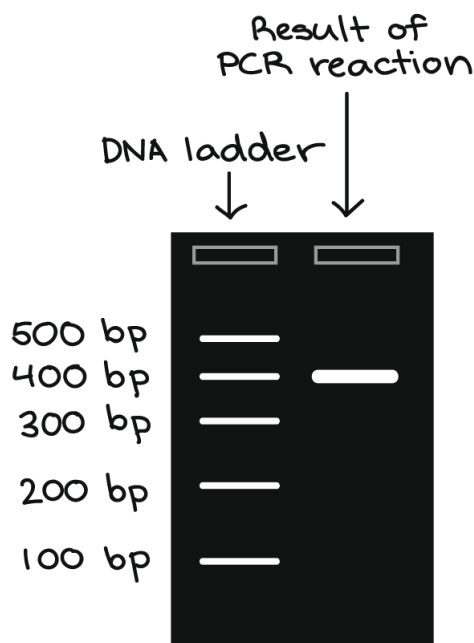


Figure 1: Electrophoregram from an electrophoresis

Results from PCR analysis can be used to confirm the presence of certain indicator microorganisms in environmental samples.

#### **4.0 Conclusion**

PCR is a widely used in molecular biology technique to amplify a single copy or a few copies of a segment of DNA across several orders of magnitude, generating thousands to millions of copies of a particular DNA sequence. This tool is useful in Environmental analysis to detect the presence of certain indicators microorganisms in environmental samples e.g. water analysis.

#### **5.0 Summary**

In this unit, we discussed the importance of Polymerase Chain Reaction in Environmental analysis. We also discussed the different steps involved in PCR analysis.

#### **6.0 Tutor-Marked Assignment**

1. What is Polymerase Chain Reaction?
2. What are the basic steps involved in a PCR reaction?
3. Mention the requirements for a successful PCR reaction.
4. How would you determine the presence of *Legionella* in a water sample?

#### **7.0 References/Further Reading**

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## **UNIT 4: THE CRYOSTAT**

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3.2: Applications of Cryostats

3.3: Biological Microtome Type

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4.0 References/Further Reading

### **1.0 Introduction**

A cryostat is a device used to maintain low and stable cryogenic temperatures of samples or devices mounted within the cryostat. Low temperatures may be maintained within a cryostat by using various refrigeration methods, most commonly using cryogenic fluid bath such as liquid helium (Frank, 2007). Hence it is usually assembled into a vessel, similar in construction to a vacuum flask or Dewar. Cryostats have numerous applications within science, engineering, and medicine.

### **2.0 Objectives**

At the end of this unit, the students should be able to know the concept of the Cryostat and its use in the Environmental Health Laboratory.

### **3.0 Main content**

#### **3.1 Types of Cryostats**



## **1. Closed-cycle cryostats**

Closed-cycle cryostats consist of a chamber through which cold helium vapour is pumped. An external mechanical refrigerator extracts the warmer helium exhaust vapour, which is cooled and recycled. Closed-cycle cryostats consume a relatively large amount of electrical power, but need not be refilled with helium and can run continuously for an indefinite period. Objects may be cooled by attaching them to a metallic cold plate inside a vacuum chamber which is in thermal contact with the helium vapour chamber.

## **2. Continuous-flow cryostats**

Continuous-flow cryostats are cooled by liquid cryogens (typically liquid helium or nitrogen) from a storage Dewar. As the cryogen boils within the cryostat, it is continuously replenished by a steady flow from the storage Dewar. Temperature control of the sample within the cryostat is typically performed by controlling the flow rate of cryogen into the cryostat together with a heating wire attached to a PID temperature control loop. The length of time over which cooling may be maintained is dictated by the volume of cryogens available.

Owing to the scarcity of liquid helium, some laboratories have facilities to capture and recover helium as it escapes from the cryostat, although these facilities are also costly to operate.

## **3. Bath cryostats**

Bath cryostats are similar in construction to vacuum flasks filled with liquid helium. A cold plate is placed in thermal contact with the liquid helium bath. The liquid helium may be replenished as it boils away, at intervals between a few hours and several months, depending on the volume and construction of the cryostat. The boil-off rate is minimized by shielding the bath with either cold helium vapour, or vacuum shield with walls constructed from so-called super insulator material. The helium vapour which boils away from the bath very

effectively cools thermal shields around the outside of the bath. In the older designs there may be additional liquid nitrogen bath, or several concentric layers of shielding, with gradually increasing temperatures. However, the invention of super insulator materials has made this technology obsolete.



Figure 1: A non-metallic, tilt able bath cryostat for liquid nitrogen

#### **4. Multistage cryostats**

In order to achieve temperature lower than liquid helium, additional cooler stages may be added to the cryostat. Temperatures down to 1K can be reached by attaching the cold plate to a 1-K pot, which is a container of He-3 isotope which is connected to vacuum pump. Temperatures down to 1mK can be reached by employing dilution refrigerator or dry dilution refrigerator typically in addition to the main stage and 1K pot. Temperatures below that can be reached using magnetic refrigeration.

### **3.2 Applications of Cryostats**

#### **Magnetic resonance imaging and research magnet types**

Cryostats used in MRI machines are designed to hold a cryogen, typically helium, in a liquid state with minimal evaporation (boil-off). The liquid helium bath is designed to keep the superconducting magnets bobbin of superconductive wire in its superconductive state. In this state the wire has no electrical resistance and very large currents are maintained with a low power input. To maintain superconductivity, the bobbin must be kept below its transition temperature by being immersed in the liquid helium. If, for any reason, the wire becomes

resistive, i.e. loses superconductivity, a condition known as a "quench", the liquid helium evaporates, instantly raising pressure within the vessel. A burst disk, usually made of carbon, is placed within the chimney or vent pipe so that during a pressure excursion, the gaseous helium can be safely vented out of the MRI suite. Modern MRI cryostats use a mechanical refrigerator (cryocooler) to re-condense the helium gas and return it to the bath, to maintain cryogenic conditions and to conserve helium.

Typically cryostats are manufactured with two vessels, one inside the other. The outer vessel is evacuated with the vacuum acting as a thermal insulator. The inner vessel contains the cryogen and is supported within the outer vessel by structures made from low-conductivity materials. An intermediate shield between the outer and inner vessels intercepts the heat radiated from the outer vessel. This heat is removed by a cryocooler. Older helium cryostats used a liquid nitrogen vessel as this radiation shield and had the liquid helium in an inner, third, vessel. Nowadays few units using multiple cryogens are made with the trend being towards 'cryogen-free' cryostats in which all heat loads are removed by cryocooler.

### **3.3 Biological microtome type**



**Figure 2: Cryostat-microtome**

Cryostats are used in medicine to cut histological slides. They are usually used in a process called frozen section histology. The cryostat is essentially an ultrafine "deli-slicer", called a microtome, placed in a freezer. The cryostat is usually a stationary upright freezer, with an external wheel for rotating the microtome. The temperature can be varied, depending on the

tissue being cut - usually from minus 20 to minus 30 degree Celsius. The freezer is either powered by electricity, or by a refrigerant like liquid nitrogen. To minimize unnecessary warming all necessary mechanical movements of the microtome can be achieved by hand via a wheel mounted outside the chamber. Newer microtomes have electric push button advancement of the tissue. The precision of the cutting is in micrometres.

#### **4.0 Conclusion**

With the use of cryostat, environmental samples can be maintained at low and stable cryogenic temperatures. Also components of some laboratories equipment that needs to be kept at low temperature can be mounted within the cryostat to achieve low and stable temperature.

#### **5.0 Summary**

In this unit, we discussed the several applications of cryostat in Environmental Laboratory.

#### **6.0 Tutor-Marked Assignment**

1. What is a cryostat?
2. How is the bath cryostat different from multistage cryostat?
3. Mention two applications of cryostats?

#### **7.0 References/Further Reading**

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## **MODULE 5: QUALITY ASSESMENT AND QUALITY CONTROL PRACTICE**

Unit 1: Quality Assurance

Unit 2: Quality Control

Unit 3: Analytical Quality Control Procedures

Unit 4: Sample Collection and Handling

**UNIT 1: Quality Assurance**

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3.4: Standard Operating Procedure

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6.0 Tutor-Marked Assignment

7.0 References/Further Reading

### **1.0 Introduction**

Quality assurance/ quality control measures are those activities that are undertaken to demonstrate the accuracy (how close to the result is to the real result) and precision (how reproducible the results are) of results from analysis in a laboratory. Quality Assurance (QA) generally refers to a broad plan for maintaining quality in all aspects of a programme. This plan should describe how a monitoring effort should be undertaken: proper documentation of all procedures, training of volunteers, study design, data management and analysis, and specific quality control measures.

### **2.0 Objectives**

At the end of this unit, the students should be able to understand quality assurance and quality assessment.

### **3.0 Main Content**

#### **3.1: Laboratory quality system**

Quality assurance is the definite programme for laboratory operation that specifies the measures required to produce reliable data of known precision and accuracy. This programme is required to be defined in documented laboratory quality system.

The laboratory quality system consists of a quality assurance manual, operating procedures, work instructions and records. The manual includes a quality policy that defines the statistical level of confidence include to express the precision and bias of data, as well as the method detection limits.

#### **3.2 Data Quality Objectives**

Data Quality Objectives (DQOs) are defined as follows:

Qualitative and quantitative statements of the overall level of uncertainty that a decision-maker will accept in results or decisions based on environmental data. They provide the statistical frame work for planning and managing environmental data operations consistent with user's needs (U.S. EPA,1997).The DQOs contain a specific set of objectives for bias, precision, completeness, detection limits, and calibrations.

#### **3.3 Quality Assurance Project Plans**

The Quality Assurance Plan “integrates all technical and quality aspects of a project, including planning, implementation, and assessment. The purpose of the QA Project Plan is to document planning results for environmental data operations and to provide a project-specific “blueprint” for obtaining the type and quality of environmental data needed for a specific decision or use. The QA Project Plan documents how quality assurance (QA) and

quality control (QC) are applied to an environmental data operation to assure that the results obtained are of the type and quality needed and expected” (U.S. EPA, 1998).

A laboratory which plan to conduct quality assurance should organize in such a way that it will apply the conditions, listed below but not limited to:

### **Quality Assurance Unit (QAU)**

QAU must be designated to audit the laboratory studies and the accompanying data. It serves as an internal control function. It is responsible for monitoring each study to assure management that the facilities, equipment, personnel, methods, practices, records, controls, SOPS, final reports.

### **3.4 Standard Operating Procedure**

Standard Operating Procedures (SOPs) are written procedures for a laboratories programme. They define how to undertake protocol, specified activities. SOP's include chronological listing of action steps with respect to:

- Routine inspection, cleaning, maintenance, testing, calibration and standardization of instruments.
- Action to be taken in response to equipment failure
- Analytical methods
- Definition of raw data
- Data handling, storage and retrieval
- Health and safety precautions.
- Receipt, identification, storage, mixing and method sampling of test and control articles.
- Record keeping, reporting, storage and retrieval of data
- Coding of studies, handling of data
- Operation of quality assurance personnel in performing and reporting study audits, inspections and final study report review.

## **Personnel**

Each individual engaged in the conduct of or responsible for the supervision of a study should have education, training and experience or combination. This will enable the individual to perform the assigned function. Personnel must be qualified to do the work. Operators of instrument should have sufficient training and or experience to operate the instrument properly and to identify a malfunction of the instrument.

The laboratory may maintain a current summary of training and experience and job description for each individual engaged in or supervising the laboratory, Job description and participation in training courses. The documentation needs to be regularly updated, retained and archived.

## **Reagents and Solutions**

All reagents and solutions in laboratory areas should be labeled to indicate identity, titer or concentration, storage requirement and expiry date. Certified standards are recommended which can be purchased from appropriate suppliers.

## **Raw Data**

Raw data refers to any laboratory worksheets, records, memoranda, notes or exact copies that are the results of original observations and activities of a study. For raw data entries, it is recommended to use a laboratory notebook for each study. This should be robust, bound and numbered with pages. All entries should be made in indelible ink.

## **Equipment**

Equipment used in generation, measurement or assessment of data and equipment used for environmental analysis should be assessed and certified to be appropriate design and adequate. Equipment / instruments should have the capacity to function according to the protocol and should be suitably located for operation, inspection, cleaning and maintenance.



The equipment should undergo a validation process to ensure that it will constantly function as intended.

Equipment should be adequately inspected, cleaned and maintained. Equipment used for generation, measurement or assessment of data shall be adequately tested, calibrated and or standardized. A laboratory should establish schedules for such operations based on manufacturer's recommendations and laboratory experience.

Written records should be maintained of all inspection, maintenance, testing, calibration and or standardizing operations. These records, containing the date of inspection, shall describe whether the maintenance operation; followed written SOPs.

### **3.5 Quality Assurance Reporting**

It is important that Environmental Health Laboratories assess the quality of their data against the Data Quality Objectives. The process of analyzing the quality of the data is known as Data Quality Assessment, which is described in detail in a U.S. EPA (2000).

It is recommended that an annual or biannual Quality Assurance Report be prepared that includes a description of the QA/QC procedures used in laboratory projects and research and a formal assessment of data quality relative to the Data Quality Objectives. The report should focus, in particular, on the accuracy, precision, completeness, comparability and representativeness of the data. All QA Reports should be submitted to the Quality Assurance Department for archiving.

### **4.0 Conclusion**

Quality assurance is an integral part of laboratory operations. This system states the requirements needed to produce reliable results of known precision and accuracy in laboratory operations.

### **5.0 Summary**

In this unit, we discussed quality assurance in a laboratory and the different components of a quality assurance system.

### **6.0 Tutor Marked Assignment**

1. Define quality assurance
2. What are the conditions for quality assurance?
3. Explain Standard Operating Procedures (SOPs)?

### **7.0 References / Further Reading**

Areskoug, H. (1988) In Expert Meeting on Sampling, Chemical Analysis and Quality Assurance (edited by Nodop, K. and Leyendecker, W.), Arona, Italy 11-14. Published by Norwegian Institute of Air Research, Kjeller, Norway.

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<http://www.epa.gov/trs/>.

## **UNIT 2: QUALITY CONTROL**

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7.0 References/Further Reading

### **1.0 Introduction**

Quality Control (QC) consists of steps taken to determine the validity of sampling and analytical procedures. Quality assessment is the assessment of the overall precision and accuracy of the data, after analysis have been run. Quality assurance and quality control are extremely important to the collection and reporting of high quality environmental data. Quality Objectives are set to define specific levels of qualities that must be achieved. This unit describes specific QA/QC procedures in Environmental Laboratories.

Quality control includes all technical activities that measure the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer. It also includes operational techniques and activities that are used to fulfill requirements for quality.

QC may not always detect blunders. Good laboratory practices, in addition to adherence to standard operating procedures (SOPs), are part of the overall QA/QC aspects needed to check the laboratory's performance. To monitor and control quality, laboratories use performance indicators, which are instrument- or protocol-related parameters that are routinely monitored

to assess the laboratory's estimate of measurement uncertainty, precision, bias, etc. Initially, these parameters are used to maintain or demonstrate control over the analytical process. The performance indicators should be tracked by appropriate personnel. If the performance indicator control limits are exceeded, management should be informed and corrective action should be initiated.

## **2.0 Objectives**

At the end of this unit, the students should be able to know the importance of quality control in Environmental Laboratory

## **3.0 Main Content**

### **3.1: Use of Reference Materials (RMS)/Certified Reference Materials (CRMS)**

Accurate and precise measurement of environmental pollutants such as metals and organics is becoming more and more important with increase in industrial, agricultural and other human activities. Quantitative and qualitative analysis of such pollutants i.e. metals, pesticides, Polynuclear aromatic hydrocarbon (PAH's), Polychlorinated Biphenyls (PCB's), Dioxins & Furnaces, Benzene, Toluene, Xylene etc. some of which are very toxic and carcinogenic at very low levels, requires sophisticated instruments like AAS, ICP, GC, GCMS, HPLC, BTX Analyzer etc. Further, analysis of any such pollutants on these instruments need proper calibration of the involved instruments using standard solution of the appropriate chemicals of required purity as instrument is used to compare an unknown sample with that of known composition. The sample of known composition is known as Reference Material or RM and if the known composition has been obtained through the internationally accepted certification procedures, the material is called as "Certified Reference Material" (CRM).

### **Reference Material (RM)**

A material or a substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for

assigning values to materials. The RM may be in the form of pure or mixed gases, liquid or solid or even a simple manufactured object.

### **Certified Reference Material (CRM)**

A reference material is one or more of whose property values are certified by the technically valid procedure, traceable to a certificate or other documentation which is issued by the certifying body. A CRM may consist of units, which are certified individually or certified by examination of representative samples from a batch.

Use of CRM may not be essential as long as Reference Materials with purity details are available, and the purity provided is adequate in context of the parameters to be measured. CRM's should be preferred over RM's for better assurance of quality to eradicate matrix interference and to enhance credibility of the results.

### **3.2 Analytical Quality Control (AQC)**

Analytical Quality Control (AQC) is one of the main components of a quality assurance (QA) system, wherein the quality of analytical data being generated in the laboratory is controlled through minimizing or controlling errors to achieve a accuracy. A particular Water Quality study or any organized water quality monitoring program involves the collection, comparison and interpretation of analytical data, which leads to a decision for the management and use of the water resource. The correctness of decision or action depends largely upon the accuracy of the analytical results. If the errors of the analytical results were high, the Manpower, Material and Money spent on any monitoring programme or study would be futile and further lead to wrong decision and improper action plans. In order to generate good and reliable environmental data, the following features are required:

- Suitable laboratory facilities;
- Laboratory instruments, sampling equipment, glassware reagents;

- Standardized analytical procedures covering the desired variables ranges of concentration;
- Well-trained and experienced laboratory personnel;
- Well-maintained equipment and facilities;
- Adequate filing and reporting system; and
- Systematic analytical quality control programme.

### 3.3 Levels of Analytical Quality Control Programme

The AQC scheme is taken up at two levels:

**1. Internal AQC or within laboratory AQC:** It is necessary basis for checking the precision and accuracy of analytical results within laboratory. It is initial demonstration of capability of the laboratory analytical functions. Various sequential stages involved for each parameter are:

- a. Choosing an analytical method suitably free from bias and ensuring the complete and unambiguous description of that method.
- b. Checking that satisfactory precision is obtained with the method.
- c. Establishing a control chart as a continuing check on precision and some sources of bias.
- d. Ensuring accuracy of standard solution.

**2. External AQC or between laboratory AQC:** A group of laboratory has to achieve comparability of results by controlling the precision and accuracy of each laboratory.

AQC tests between laboratories are necessary for the following reasons:

- i. To test for possible errors caused by sources not already checked in within-laboratory AQC and
- ii. To provide direct evidence that the required comparability of results between laboratories has been achieved.

- iii. Accuracy may deteriorate with time and hence subsequent regular tests are required as a continuing check on between laboratories bias.
- iv. The procedure to convert a method to standard status is done through collaborative test (inter-laboratory studies).

#### **4.0 Conclusion**

Quality Control allows minimization of errors in the laboratory. QC which is composed of internal and external AQC is a vital part of a QA plan.

#### **5.0 Summary**

In this unit, we discussed the concept of quality control and the different component of QC used to achieve quality in the laboratory.

#### **6.0 Tutor Marked Assignment**

1. What is quality control and analytical quality control?
2. What are the levels of Analytical Quality Control Programme?
3. Give four reasons why QAC test within laboratories is important.

#### **7.0 References / Further Reading**

Laboratory Analytical Techniques Series LATS/9/2008-2009. (2009). Guidelines for Recognition of Environmental Laboratories under the Environment (Protection) ACT, 1986 (Revised & Updated Version)

Taylor, J. K. (1987). Quality Assurance of Chemical Measurements. Lewis Publishers, Inc., Chelsea, MI, USA.

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## **UNIT 3: ANALYTICAL QUALITY CONTROL PROCEDURES**

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1.0 Introduction

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3.0 Main content

3.1: Intra-Laboratory (Within Laboratory) AQC Procedures

3.2: Laboratory – Fortified Matrix (LFM) / Laboratory-Fortified Matrix Duplicate (LFMD)

3.3: Checking Recovery in Analytical Procedures

4.0 Conclusion

5.0 Summary

6.0 Tutor-Marked Assignment

7.0 References/Further Reading

### **1.0 Introduction**

As a first step of within laboratory AQC procedures, the analytical team must check standards or unknown concentrations and evaluate the results. This practice can uncover weakness of analytical procedures, which can then be improved. These weaknesses can be accounted to faulty sample treatment, improper elimination of interference, poor calibration practices, sloppy experimental technique, impure or incorrectly standardized reagents, defective instrumentation, or even mistakes in arithmetic.

### **2.0 Objectives**

At the end of this unit, the students should be able to understand analytical control procedures.

### **3.0 Main Content**

#### **3.1: Intra-Laboratory (Within Laboratory) AQC Procedures**

A periodic reassessment should be made of available analytical methods, in view of appropriateness and applicability. In addition, each method selected should be evaluated for sensitivity, precision and accuracy. The benefits of intra-laboratory as well as inter-laboratory

Analytical quality evaluations should be known to the analytical team. These procedures should not be interpreted as an examination of their abilities by their superiors, but as a normal quality assessment procedure.

As a first step of within laboratory AQC procedures, the analytical team must check standards or unknown concentrations and evaluate the results. This practice can uncover weakness of analytical procedures, which can then be improved. These weaknesses can be accounted to faulty sample treatment, improper elimination of interference, poor calibration practices, sloppy experimental technique, impure or incorrectly standardized reagents, defective instrumentation, or even mistakes in arithmetic.

### **Method blank/Reagent blank**

The method blank/Reagent blank is a quality control sample that consists of reagent water and all reagents that are normally added in the sample during analytical procedure. The method blank is used to identify any interferences or contamination of the analytes that may lead to the reporting of elevated concentrations or false positive data. Potential sources of contamination include solvent, reagents, glassware, other sample processing hardware and the laboratory environment etc. As a minimum, one reagent blank may be included with each sample set (batch) or on a 5% basis, whichever is more frequent.

The result of this analysis is one of the quality control measures used to assess batch acceptance. The source of method blank contamination should be investigated and measures taken to correct, minimize or eliminate the problem.

### **Laboratory – Fortified Blank (LFB)**

A laboratory-fortified blank is a reagent water sample, to which known concentration of analysis of interest has been added. A LFB is used to evaluate laboratory performance and analyte recovery in a blank matrix. One LFB is included with each sample set (batch) or on a 5% basis, whichever is more frequent except for analyte, for which spiking solutions are not

available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, colour, odor, temperature, dissolved oxygen or turbidity. The LFB may be evaluated for percent recovery of the added analyte. If LFB results are out of control, corrective action should be taken by re-preparation and reanalysis of associated samples.

### **3.2 Laboratory – Fortified Matrix (LFM) / Laboratory-Fortified Matrix Duplicate (LFMD)**

Laboratory-Fortified Matrix (LFM) is an additional portion of a sample, to which known amount of the analyte of interest are added before sample preparation. The LFM is used to evaluate analyte recovery in a sample matrix. The concentrations same as for the LFB is used to allow the analyst to separate the effect of matrix from laboratory performance.

A laboratory – fortified matrix duplicate is second aliquot of a sample that is spiked with the selected target analyte(s) and analyzed with associated sample LFM sample. The results of the LFM and LFMD are used together to determine the bias introduced by the sample matrix and precision of the analytical process. The acceptance criteria of the LFM are expressed as the percent recovery of the known concentration, spiked to the environmental sample. This is identical in principle to the calculation of the percent recovery of LFB.

The acceptance criteria for the LF/LFMD analysis are based on the relative percent difference (RPD) between the samples, provided both samples meet the acceptance criteria for spike recovery.

#### **Sample Duplicate**

A laboratory sample duplicate is a second aliquot of an environmental sample taken from the same sample container that has to be processed identically along with the first aliquot. Sample duplicates are processed as independent samples, within the same analytical batch. Sample duplicate may be used to assess sample homogeneity and the precision of the analytical process.

### **Internal Standard**

Internal Standards (IS) are used for organic analysis by GC-MS, certain GC analysis and some metal analysis by ICP-MS. An internal standard is an analyte included in each standard and added to each sample or sample extract/ digestate just before sample analysis. Internal standards usually mimic the analytes of interest, but not interfere with analysis. If internal standard results are out of control, corrective action should be taken by the laboratory, including reanalysis of samples.

### **Surrogate Standard**

A surrogate standard is a compound of a known amount added to each sample before extraction, surrogate mimic the analytes of interest and are compared unlikely to be found in environmental sample. Surrogate are used specifically for organic analysis.

Poor surrogate recovery may indicate problem with the sample composition or problem with the isolation of target analyte from the sample matrix prior to chromatographic analysis.

### **Post Digestion Spike (PDS)**

A Post Digestion Spike (PDS) is an analytical spike, created by adding known concentrations of target analytes into prepared portion (i.e. post digestion) of sample, just before the analysis. It is used frequently in inorganic instrumental analysis. It provides information on matrix effects encountered during analysis, such as suppression or enhancement of instrument signal levels. It is used in elemental analysis involving various forms of atomic emission or atomic absorption spectroscopy, to ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values. A single analytical spike serves as a single point application of the method of standard addition.

### **Dilution Test**

A dilution test (serial dilution) is performed, if the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution). The analysis of 1:5 dilutions should agree within  $\pm 10\%$  of the original determination. If not, a chemical or

physical interference effect is suspected. It is most often used in elemental analysis involving various forms of atomic emission or atomic absorption spectroscopy to ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort accuracy of the reported values.

The above mentioned within laboratory analytical tests may not be possible all the time, while analyzing various types of samples collected from various sources. Therefore, it is advised that relevant tests be selected by the analyst as per their convenience and performed at regular intervals.

### **Quality Control Chart**

Quality control charts are statistical tools used to ensure that a measurement system is in control. Analytical quality can be violated by random and systematic errors during determination. Random errors are recognized to produce reduction in precision, while systematic errors give rise to reduction in accuracy. To check on both types of errors, quality control chart should be made for each variable. The application of quality control chart is based upon the assumption that the experimental data have normally distributed errors. The quality control chart should be plotted from the results repeatedly obtained from standard samples of known concentration against time.

In order to prepare the quality control chart, the mean and standard deviation are calculated based on initial calibration study (analysis of at least 20 replicate standards of realistic concentration). The control chart is then constructed with the calibration mean as its center line. Warning and rejection areas are added at  $\pm$  two times and  $\pm$  three times the standard deviation from the mean respectively. The analysis is performed each day for the same standard. If the analytical result of standard analysis falls outside the  $\pm 3$  standard deviation line, the analysis is said to be out of control and an immediate check is required to know the cause for this gross analytical errors. After corrective measures the analysis should be

repeated. The occurrence of an unduly high percentage of results exceeding the warning limits (2 standard deviation) is an indication that laboratory precision may not be as good as expected or that the frequency distribution of the results are not normal. The control limits may be recalculated periodically as experience is gained with the technique. Preferably use several standards that span a range of concentrations to ascertain linearity and to avoid unintentional bias due to familiarity with the fixed results.

If internal quality control data are available, it is possible to indicate the accuracy of the data. Accuracy is a measure of the systematic nature of the errors in the analysis, and can be calculated as the difference between the mean of series of subsequent standard determinations and the true concentration of the standard sample. It is expressed as mean error:

$$\text{Mean Error} = X - TV$$

Where: X = mean; TV = true value

If the difference between the true value and the standard mean is greater than one standard deviation, the method, reagents, glassware, and instrumental are to be checked for deviations for corrective actions to be taken.

During series of observations it is often observed that one or more values of the results deviate from the mean, whereas the other values are in close agreement with it. At this point, decide whether to reject disagreeing values. Theoretically no result should be rejected, because the presence of large percentage of disagreeing results indicate faulty techniques and may doubt the acceptance of analytical results.

### **3.3 Checking Recovery in Analytical Procedures**

Recovery methods are tools to remove doubt about the applicability of method to a sample. The recovery procedure involves applying the analytical method to a reagent blank; to a series of known standards covering the expected range of concentration of the environmental sample; to the environmental sample itself; and to recovery samples composed by adding a

range of known quantities of the analyte to separate aliquots of the environmental sample. The analyte should be added in sufficient quantity to overcome the limits of error of the analytical method, but not to cause the total in the sample to exceed the concentration range of the standards used. Correct the analytical results by subtracting the reading from the reagent blank from all other readings. Make a calibration graph from the corrected results of standard determinations. Use this graph to convert the readings of the spiked and non-spiked environmental samples to the actual concentrations. Subtract the concentration of the unspiked sample from each of the concentrations of sample plus known concentration of added substance. The resulting concentration of substance divided by the actually added concentration, multiplied by 100, gives the percentage recovery.

Rigid rules regarding the percentage of recovery required for the acceptance of results cannot be stipulated, because the recoveries in the range of sensitivity may vary. In general, intricate and exacting procedures for trace substances that have inherent errors due to their complexity may give recoveries that could be considered very poor. Yet, from a practical point of view these analysis may be considered useful (no alternative).

### **Inter-Laboratory (Between Laboratories) AQC**

To confirm the comparability of the results produced by different laboratories taking part in a monitoring programme, aliquots of the same reference samples should be analyzed once or twice a year by all laboratories performing the same types of analysis. These reference samples may be the same as the standard samples. In evaluating proficiency testing results, the laboratory issuing the reference samples may use control charts. The rejection and warning limits are generally set larger than those used in internal quality control because the variation between laboratories is normally larger than the variation within one laboratory as a result of the difference in instruments, glassware etc.

### **Sources of Errors**



Errors in results may also arise through non-representative sampling and through changes in the sample between sampling and analysis. The first source of error must be controlled through the choice of appropriate time, location and procedure of sample collection. The second source of error must be controlled by using appropriate technique of sample preservation and by analyzing samples without undue delay. The specification of appropriate sample preservation techniques must be an integral part of any analytical method and any effective AQC scheme must include all necessary precautions required for sample preservation. Besides analysis laboratory staff must be acquainted with sampling, preservation and transportation of environmental samples.

#### **4.0 Conclusion**

Analytical Quality Control procedures provide us with tools with which the correctness and validity of laboratory data can be tested. These tools may be use intra- or inter-laboratory.

#### **5.0 Summary**

We have discussed how the quality control chart can be used as a statistical tool to ensure the validity of data generated in the laboratory; we also discussed the different sources of errors.

#### **6.0 Tutor Marked Assignment**

1. What is a quality control chart?
2. Identify the different sources of errors that may interfere with quality in laboratory operations
3. What are internal and external surrogate standards?
4. What are the reasons for using reagent blank and sample duplicates in quality control?
5. How would you check recovery in analytical procedures?

#### **7.0 References / Further Reading**

- Areskoug, H. (1988). In Expert Meeting on Sampling, Chemical Analysis and Quality Assurance (edited by Nodop, K. and Leyendecker, W.), Arona, Italy 11-14. Published by Norwegian Institute of Air Research, Kjeller, Norway.
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## **UNIT 4: SAMPLE COLLECTION AND HANDLING**

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7.0 References/Further Reading

### **1.0 Introduction**

Sample collection is a vital aspect of any laboratory analysis. Improper sample collection leads to wrong laboratory results. This in turn could lead to wrong conclusions.

### **2.0 Objectives**

At the end of this unit, the students should be able to understand the concept of sample collection and handling.

### **3.0 Main Content**

#### **3.1: Sample Container Testing**

Sample containers (e.g., buckets, bags, funnel-and-bottles) used in environmental samplers must be tested for chemical inertness before being deployed to the field. Specific testing must be done to ensure that the collection vessel material does not adversely affect the integrity of the sample through the adsorption/desorption of major ions to/from the vessel walls.

Old collection vessels should be tested in the foregoing manner at least every 5 years to ensure that absorption/desorption effects have not accumulated or changed with time and use.

### **Sample Container Cleaning**

All sample containers (including funnels and bottles) must be cleaned with de-ionized water between uses – preferably at the analytical laboratory but, if necessary, at the field sites. Where the collection vessels are cleaned on-site, the deionized water used in the cleaning process must be tested to ensure that it meets predetermined quality specifications, both before and after the sample containers are cleaned. Methods must include careful cleaning and rinsing of the bucket and/or funnel rims since they are in direct contact with the collector hood. After being cleaned, sample containers that are not used immediately must be air dried in a clean location, sealed or covered in plastic, and stored in a clean area.

### **3.2 Sample Collection**

Specific quality control procedures that prevent contamination from occurring during sample collection and handling include:

- Inspecting the collection vessels for visible signs of contamination before they are placed in the collector
- Standing on the downwind side of the precipitation chemistry samplers when collecting samples in order to reduce the potential for contamination
- Wearing disposable plastic gloves whenever handling precipitation sample containers and transferring samples
- Instructing operators never to touch the inside of sample containers
- Replacing dirty gaskets on the underside of the sampler hood at frequent and regular intervals (every few months depending on the dirtiness of the site)
- Checking for, and documenting, sample leaks in the field, during shipping, and upon receipt at the laboratory.

### **3.3 Sample Storage and Shipping**

Proper storage and shipping methods must be used to preserve the chemical and physical integrity of the environmental laboratory samples. Quality control procedures for this purpose include:

- Maintaining sample temperature at 4°C during periods of storage on-site, in the laboratory and in shipment.
- Weighing samples to determine sample volume at the collection site and at the laboratory in order to detect leaks in transit
- Instituting procedures to eliminate leaks during transit.

#### **4.0 Conclusion**

Improper sampling method or tools may introduce error in analysis. It is important to collect samples at the right time and at the right location using the right procedure. Samples collected also must be preserved to retain its integrity before and during analysis to minimize errors. Analysis of samples should not be delayed.

#### **5.0 Summary**

This unit has summarized the importance of sampling and sample handling in quality control.

#### **6.0 Tutor Marked Assignment**

1. Discuss the importance of sampling in quality control.

#### **7.0 References / Further Reading**

- Areskoug, H. (1988). In Expert Meeting on Sampling, Chemical Analysis and Quality Assurance (edited by Nodop, K. and Leyendecker, W.), Arona, Italy 11-14 Oct. Published by Norwegian Institute of Air Research, Kjeller, Norway.
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