

National University –SUDAN

Faculty of Clinical and Industrial Pharmacy

Second Year (**Batch-PA-14**)-Semester Four

Professional Skills-2- Laboratory Skills-1

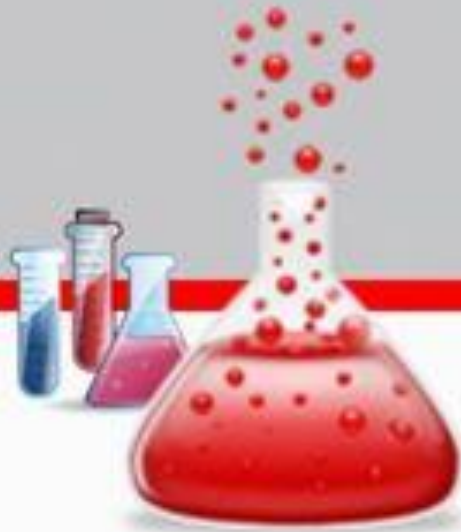
(**PA-SKILL-221**)

Course Coordinator: **Us.** Maria M. Elamin Hamid,

M.Sc. Pharmaceutical Analysis and Quality Control

Email: chocoroy991@gmail.com

Phone Number (**00249-913714903**)



Quality Control for Sampling and Chemical Analysis

Objectives

By the end of this lesson the student is expected to understand

1. Write practical report
2. The definition of Quality control
3. The Purpose of QA/QC
4. Importance of the tools for the analyst
5. Errors in measurements

LAB REPORT FORMAT

A descriptive of the component parts of the lab report.

TITLE SHEET

- This sheet identifies the lab activity. It contains the name of the student, class and period, date and instructors' name.
- It may contain some art that is relevant to the activity that the report will describe.

PAGE 1

- **Purpose:** The reason for performing the lab activity. (sentence)
- **Materials:** A list of the items used to carry out the lab activity. (list)
- **Procedure:** Step by step, what was done to execute the lab exercise. (paragraph)
- **Conclusion:** Quantitative and/or qualitative observations of the activity based on the data gathered or results from the activity. (paragraph)

PAGE 2

- **Data:** Numerical or statistical information that can be put into words in the conclusion. Usually comes in the form of charts, graphs, and tables.
- **Plates:** Illustrations that depict what was observed during the exercise. Mostly used in Biology Labs.

SCIENTIFIC METHOD

- **Statement of the Problem.** (Question)
- **Collection of Information.** (Research)
- **Formation of Hypothesis.** (Educated Guess)
- **Test for Hypothesis.** (Experiment)
- **Accept or Reject Hypothesis.** (Data Analysis)
- **Report the Results.** (Conclusion)

What is a good Hypothesis?

- **HYPOTHESIS-** is a statement that gives the best possible response to a question and should be based on already known facts.
- **Educated Guess...**
- **Examples:** If I do such and such, then this or that will occur.
- **Or another example:** I predict that such and such will happen.

VARIABLES

- **Independent Variable-** the factor that is changed or manipulated by the researchers, in order to determine the effect of the change.
- **Dependent Variable-** the factor that the experimenter is measuring or counting. This changes in response to the independent variable.
- **Control Variable-** all other factors in the experiment. These things the researcher attempts to control, with the goal of keeping them the same for all samples.

Concepts

- The purpose of the **Control Variable** is to have an element of the experiment kept at a constant state for the entire experiment to make some comparisons of what variable affects an experiment.
- **Qualitative Observations**- use of senses. Example: That pie smells good!
- **Quantitative Observations**- use of numbers. Example: There are 12 dollars in the bank.

Writing a fundamentally sound paper.

- **Introductory paragraph:** Gives the reader an idea of what you are writing about. Try to mention three things that you can fluently and expertly discuss. Let last sentence of this paragraph be your transition into the next paragraph.

The Body

- **Three paragraphs:** These paragraphs should discuss the three things mentioned in your introductory paragraph. Each of these three paragraphs should end with a transition sentence for the next paragraph.

The Conclusion

- **Final paragraph:** It should be a summary of what was written and should include one statement of **Universal Truth** that the reader can relate to about your topic. Most conclusive paragraphs may begin with:
 - I must conclude by saying...
 - In conclusion...
 - It can be concluded that...

THE FINAL WORD

- Whatever you do, whatever you write, proofread what you've written. Let it make sense and have consistency of thought.
- **Keep it real!**

LABORATORY SAFETY

- Safety procedures are designed to protect you and others from injury.
- Use common sense.
- Read all directions before working in the lab.
- Wear appropriate clothing.
- Know where the Safety Shower, Eyewash Station, and Fire Extinguisher are located.
- Report ALL accidents to TEACHER immediately.

GLASSWARE SAFETY

- Never use broken or chipped glassware. Dispose of in container specified by Teacher.
- Never heat glassware not thoroughly dry.
- Never pick up HOT glassware. Use tongs.
- Do not put hot glassware in cold water or on any cold surface.

SHARP INSTRUMENT SAFETY

- Always use single edged razors.
- Handle any sharp instrument with extreme care.
- Never cut any material toward you. Always cut away from you.
- Dispose of used instruments in container specified by teacher.

FIRE and HEAT SAFETY

- Never use an open flame without safety goggles.
- Never heat anything unless instructed to do so.
- Never heat anything in a closed container.
- Never reach across a flame.
- Always use tongs to handle hot objects.
- When heating objects move in and out of flame.
Never leave object in flame.

ELECTRICAL SAFETY

- If an extension cord is needed to plug in an electrical device, use the shortest extension cord possible.
- Do not use socket multipliers to overload an electrical outlet.
- Never touch an electrical appliance or outlet with wet hands.

CHEMICAL SAFETY

- Always wear a safety apron and protective gloves when handling chemicals to protect yourself from chemical spills. If a chemical contacts your skin rinse immediately and notify your instructor and seek emergency care.
- If instructed by your teacher to smell a chemical, never do so by sniffing directly from the container. Instead, hold the container away from your face and use your hand to waft some of the chemical odor towards your nose.

More CHEMICAL SAFETY

- Use proper ventilation in the lab through use of chemical fume hood.
- Keep all lids closed when chemicals are not in use.
- Dispose of all chemicals as instructed by your teachers.

EYE AND FACE SAFETY

- Wear safety goggles when handling chemicals.
- When you are heating a test tube or bottle, always point it away from you and others.
- Remember, chemicals can splash or boil out of a heated test tube.
- If a chemical comes in contact with your eyes, use the eyewash fountain immediately, and seek emergency care.

PROPER DRESS

- Wear long-sleeved blouses, shirts, and pants rather than shorts.
- Tie back long hair to prevent it from coming into contact with chemicals or an open flame.
- Wear shoes without open ends.
- Remove or tie back any dangling jewelry or loose clothing to prevent them from getting caught on any equipment.

What is QA/QC?

- Quality Assurance (QA)
 - Are we doing the right things?
 - Management
- Quality Control (QC)
 - Are we doing things right?
 - Technical

Quality ~~Control~~

Are we doing things right?

A technical concept:

- Did we do the test?
- Did the results pass or fail the criteria?
- If the results failed did we perform a corrective action?

Purpose of QA/QC

- Determine precision and accuracy,
- Demonstrate absence of interferences,
- Demonstrate absence of contamination (from sampling equipment, glassware, and reagents)

Quality

Assurance

Are we doing the right things?

A management concept:

- Planning
- Assessment
- Continued improvement

What Qualities do We Want to Control?

- Accuracy - closeness to the “true” value
- Precision - repeatability

Accuracy

- Composed of precision and bias
- Measure of the overall agreement of a measurement to a known value
 - when random errors are tightly controlled, bias dominates the overall accuracy
 - when random errors predominate, variance (imprecision) dominates the overall accuracy
- Use bias and precision as separate measures rather than accuracy

Precision

- Precision is the measure of agreement among repeated measurements under identical conditions
- A precision QC is a quantitative indicator of the random errors or fluctuations in the measurement process
 - e.g., standard deviation or variance

Sensitivity

- Usually regarded as detection limit
- Capability of a method or instrument to discriminate between measurement responses
 - but this term is often used without defining what is intended (minimum detection or quantitation)
- A sensitivity QC describes the capability of measuring a constituent at low levels
 - a Practical Quantitation Level describes the ability to quantify a constituent with known certainty
 - e.g., a PQL of 0.05 mg/L for mercury represents the level where a precision of +/- 15% can be obtained

Data Verification

- The procedures needed to ensure that a set of data is a faithful reflection of all the processes and procedures used to generate the data
 - verification entails the examination of objective evidence that the specified method, procedures, and contractual requirements were fulfilled

Data Validation

- Analyte and sample matrix-specific process to determine the analytical quality of a data set
 - inspection of data handling practices for deviations from consistency,
 - review of quality control (QC) information for deviations,
 - assessment of deviations,
 - assignment of data qualification codes
- Validation can entail the examination of the data with respect to the QA Project Plan

Accuracy

Closeness to the “true” value

Types of QC to assess accuracy:

- Matrix Spike
- Laboratory Control Sample
- Laboratory Fortified Blank
- Standard Reference Material

Bias

Error in a specific direction

Blanks are used as an indication of bias

- Field Blank
 - analyte contamination - positive bias
- Method Blank
 - analyte contamination - positive bias
- Calibration Blank
 - analyte contamination - negative bias
 - Matrix mismatch – positive or negative bias
- Calibration Check Blank or Instrument Blank
 - Instrumental drift over time - positive or negative bias

Precision

Precision = repeatability

Types of QCs to assess precision:

- Duplicate Field Sample
- Duplicate Lab Sample
- Duplicate Matrix Spike
- Duplicate Laboratory Control Sample
- Duplicate Laboratory Fortified Blank

QC – Tools for the Analyst

1. Use Quality Control samples to identify accuracy and precision problems,
1. AND to isolate the problems,
1. AND to address the problems

Why Measurements?

Measurements are basic tools in S & T. Said a German philosopher: If I can define it, I can measure it.

If I can measure it, I can analyze it. If I can analyze it, I can control it.

If I can control it, I can improve it.

Measurements are needed to

Choose ,develop, and validate models used to predict, control or Improve various phenomena. Measurements provide the very basis Of all control actions.

What are Measurements?

Measurement :

A Process that follows a defined sequence of steps/activities involves physical, material and technological resources;and results in a numerical value/a set of numerical values that is assigned to an item in respect of a defined property/ parameter/ characteristic.

Measurement : The output of a
(measurement) process.

- a numerical value

Errors in Measurements:

Errors in the observed results of a measurements (process) give rise to uncertainty about the true value of the measured as is obtained (estimated) from those results. Both systematic and random errors affecting the observed results (measurements) contribute to this uncertainty.

Random errors presumably arise from unpredictable
Variations of influence quantities ,for example.

- the way connections are made or the measurement method employed
- uncontrolled environmental conditions or their influences
- inherent instability of the measuring equipment
- personal judgment of the observer or operator, etc.

These cannot be eliminated totally but can be reduced by exercising appropriate controls.

Basic requirements of a Calibration Lab

- ❖ Instruments(Standards)
- ❖ Personnel
- ❖ Documentation

ADVANTAGES OF CALIBRATION

1. Insures that all tools & test standards properly agree
2. Verifies compatibility of parts, systems & assemblies
3. Obtain peak performance of all systems in use
4. Insures that everyone in the company stays working to the same standard

contd . . .

5. Ensures performance after recalibration
6. Avoids damage during performance checking because of improperly calibrated or functioning instruments

ERRORS IN MEASUREMENT

Types of error

Gross error

Random error

Systematic error

ERRORS IN MEASUREMENT

Contd. . .

Gross errors

Are strictly under the control of individual (independent of instrumentation)

Examples-

- Misreading of instrument
- Making incorrect adjustments
- Improper application of instruments
- Recording interpolated data

Can be avoided by-

- Care and attention
- Close supervision
- More systematic training

ERRORS IN MEASUREMENT

contd...

Random errors

A scatter about an average when a multiple no. of measurements are taken

As a result of:

**Variations in measuring system Changes
in quality being measured**

Detectable when:

**Repeated measurements are taken with a seemingly
constant set-up and consistent technique**

"Chemistry states the more energy you put into a bond, the harder it is to break."



Thank you