الجامعة الألمانية الأردنية
German Jordanian University
GJU

WEEM 3510

Water and Wastewater Unit Operation Lab Manual

Spring 2015

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Syllabus

Water and Wastewater Unit Operation Lab
WEEM 3510
Tuesday & Wednesday 14:00-17:00

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Course text: None required. This manual is work in progress and will be handed out a week ahead of conducting each experiment. In addition some experiment will undergo modification and updating. Please be patient as the manual and our laboratory evolve. You are encouraged to check your GJU mail a head of each lab. Notes and changes will be sent via e-mail.

Reference texts: Same text as the WEEM 351

Course objective: This course is designed to introduce third-year water and environment engineering students to experiments that demonstrate principles that are learned in WEEM351 course. Student will learn technical skills, how to use and calibrate measurement devices, how to conduct experiment that test samples for physical and chemical characteristics, and how to write a report.

Grading:

***Attendance/participation**	**17**
Written lab reports	**23**
Lab quizzes & Pre Lab	**20**
Final	**40**
TOTAL	**100**

Reports:
Reports on experiments will be due **ONE WEEK** after the experiment is completed, at the beginning of each class. Reports submitted after 14:00 on will be counted as one day late. Late reports are accepted for only 2 days and will be penalized 35 % per day. No report after that day will be accepted at all.

Schedule:

Week Order	Date	List of Experiment**	
1st Week	3rd & 4th March		Orientation and Safety
		Exp 1	Sampling
2nd Week	10th & 11th March		Total solids
		Exp 2	Volatile solids
3rd Week	17th & 18th March	Exp 3	Sludge Volume Index
4th Week	24th & 25th March	Exp 4	Settling Zones / Kinetics
5th Week	31st March & 1st April	Exp 5	Jar Test
6th Week	7th & 8th April	Exp 6	Reverse Osmoses
7th Week	14th & 15th April	Exp 7	Aerobic digestion
8th Week	21st & 22nd April	Exp 8	Anaerobic digestion
9th Week	28th & 29th April	Exp 9	Adsorption
10th Week	6th & 7th May	Exp 10	Ion Exchange
11th Week	13th & 14th May	Exp 11	Absorbance / Flame photometer
12th Week	20th & 21st May	Exp 12	Filtration
13th Week	27th & 28th May		Free Lab
14th Week	3rd & 4th June		FINALS

***** Note that experiment order or even can be change according to the instructor.***

Course Policies: The following course policies must be followed. At the discretion of the instructor or teaching assistances, non-complacence with course policy may negatively impact your final course grade.

- Preparation: Before coming to the laboratory, read the description of the experiment and relevant background material must be read. For each experiment, be prepared to complete a short quiz covering the objective of work, theory and procedure, the quiz may also cover the previous experiment.

- Attendance and work: Attendance is compulsory for all labs during the semester. If an emergency arises, you must inform the instructor and the TA before class and arrangements will be made since there will be no remake of any lab. Unexcused absence count as zero on the quiz, experimental work and lab reports. **No** written report will be accepted **without attending** the experiment.
  Exceeding one lab absence will mean expelling from the lab.
  The experimental work is an individual effort, unless told otherwise. When laboratory work is completed, you are to write your own reports with NO SHARING OF WRITTEN WORK.

- Integrity: Each student work and behavior is assumed to hold the highest standards of honesty. Cheating, fabrication, plagiarism, and helping the others to commit these actions are all forms of dishonesty and they are wrong. You are prohibited from using old reports and files from previous years. You are prohibited from copying sections from any resources including other students' work, text books, and websites. All resources, including figures downloaded

from the internet must be cited. First instances of plagiarism will negatively impact your final course grad; in other words, no warning will be given for plagiarism cases and a NEGATIVE grade will be given to such cases.

- Midterm and Final: There will be no midterm exam. The final test will be a written one.

- Safety: Safety in laboratory is tantamount to good laboratory practice. No rule, unless augmented with safety awareness and good sense, will protect you from accidents.

Bottom line

Maintain high standards for your own work. Start work early so the temptation to cut corners does not arise. If you have questions or concerns, do not hesitate to ask the instructor or TAs.

The following practices will be exercised in the laboratory:

1. On laboratory days, long pants must be worn, and shoes must be closed-toe (no sandals). If students dressed inappropriately, they will be asked to change before the beginning of the experiment. If not, they will be prohibited from the conducting the experiment and will lose marks based on that.
2. In the laboratory, safety glasses, white apron are required as well as gloves.
3. You are to bring only your manual, calculator and pen into the lab. Your back bags, hand bags will all be gathered away and not allowed to be put on the lab benches
4. Smoking, eating or drinking are not allowed in the laboratory. GUM chewing is included!
5. Cell phones are to be shut down during the lab, or they will be confiscated during other labs for students who break the rule.
6. Report all injuries and accidents to the instructor or TAs immediately, no matter how minor.
7. Each student is responsible of cleaning his/her station all the time. Marks will be deducted from your work if the station and glassware were left dirty.

PLEASE REFER TO THE SHEETS CONCERNING THE SAFETY RULES THAT MUST BE CONDUCTED IN THE LAB.

STUDENTS MUST AFFIRM THAT THEY HAVE READ AND UNDERSTOOD THE LABORATORY SAFETY MANUAL AND SIGN THE AGREEMENT GIVEN BY THE LAB SUPERVISOR.

Safety Rules and Regulation

The rules and regulations that follow are universal for the 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. Students who consistently violate the eye protection policy are subject to dismissal from the lab .

There are EYE-WASH stations located in lab. If chemicals entire your eyes, flush them immediately at the station. Water might leak out onto the floor from the wash station – ignore it, while trying not to slip on the water.

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 to perform the experiment. Students should check with their instructor as to where to get these chemicals and what safety precautions, if any, are to be taken in conjunction with the use of these chemicals. They should be able to get the Material Safety Data Sheet (MSDS) for all chemicals.

Horseplay

Repeated incidents are unprofessional, and will result in a grade penalty.

i. 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.
ii. Do not use mouth suction to fill pipettes.
iii. Label all containers to avoid errors and read labels carefully.
iv. Never remove shared chemicals from their original locations, others will need them.
v. 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 TAs. 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 and TA 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…) 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!!

Obligation: Each student has a professional obligation to contribute a full and honest effort in the group execution of experiments and reports. Consistent failure to observe this rule is considered unprofessional behavior, and will be penalized.

Agreement

Sign and date this as a reminder of procedures you will be practicing during water and environmental chemistry lab.

- I have read and agree to follow all of the safety rules set forth in this contract. I realize that I must obey these rules to insure my own safety, and that of my fellow students and instructors. I will corporate to the fullest extent with my instructor and fellow students to maintain a safe lab environment. I will also closely follow the oral and written instructions provided by the instructor. I am aware that any violation of this safety contract that results in unsafe conduct in the laboratory or misbehavior on my part may result in being removed from the laboratory.
-

No.	Student Name	Reg. No.	Date	Signature

LAB WARE AND GLASSWARE

Bring:

Notebook
Safety Glasses
Calculator
Lab Coat
Lab Manual
Pen
Soap, And Your
Own Towel

Lab Equipment

The measurement of trace constituents in water demands methods capable of maximum sensitivity. In addition to sensitive methods, however, there are other areas that require special consideration. One such area is that of the cleanliness of laboratory glassware. Obviously, the very sensitive analytical systems are more sensitive to errors resulting from the improper use or choice of apparatus, as well as to contamination effects due to an improper method of cleaning the apparatus.

Types of Glassware

Laboratory vessels serve three functions:

1. Storage of reagents
2. Measurement of solution volumes
3. Confinement of reactions.

For special purposes, vessels made from materials such as porcelain, nickel, iron, aluminum, platinum, stainless steel, and plastic may be employed to advantage. Glass, however, is the most widely used material of construction.

There are many grades and types of glassware from which to choose, ranging from student grade to others possessing specific properties such as super strength, low boron content, and resistance to thermal shock or alkali. The mainstay of the modem analytical laboratory is a highly resistant borosilicate glass, such as that manufactured by Coming Glass Works under the name "Pyrex".

The use of plastic vessels, containers, and other apparatus made of Teflon, polyethylene, polystyrene, and polypropylene has increased markedly. Some of these materials, such as Teflon, are quite expensive. However, Teflon stopcock plugs have practically replaced glass plugs in burettes, separatory funnels, etc., because lubrication to avoid sticking or "freezing" is not required. Polypropylene, a methylpentene polymer, is available as laboratory bottles, graduates, beakers, and even volumetric flasks. It is crystal clear, shatterproof, autoclavable, and chemically resistant.

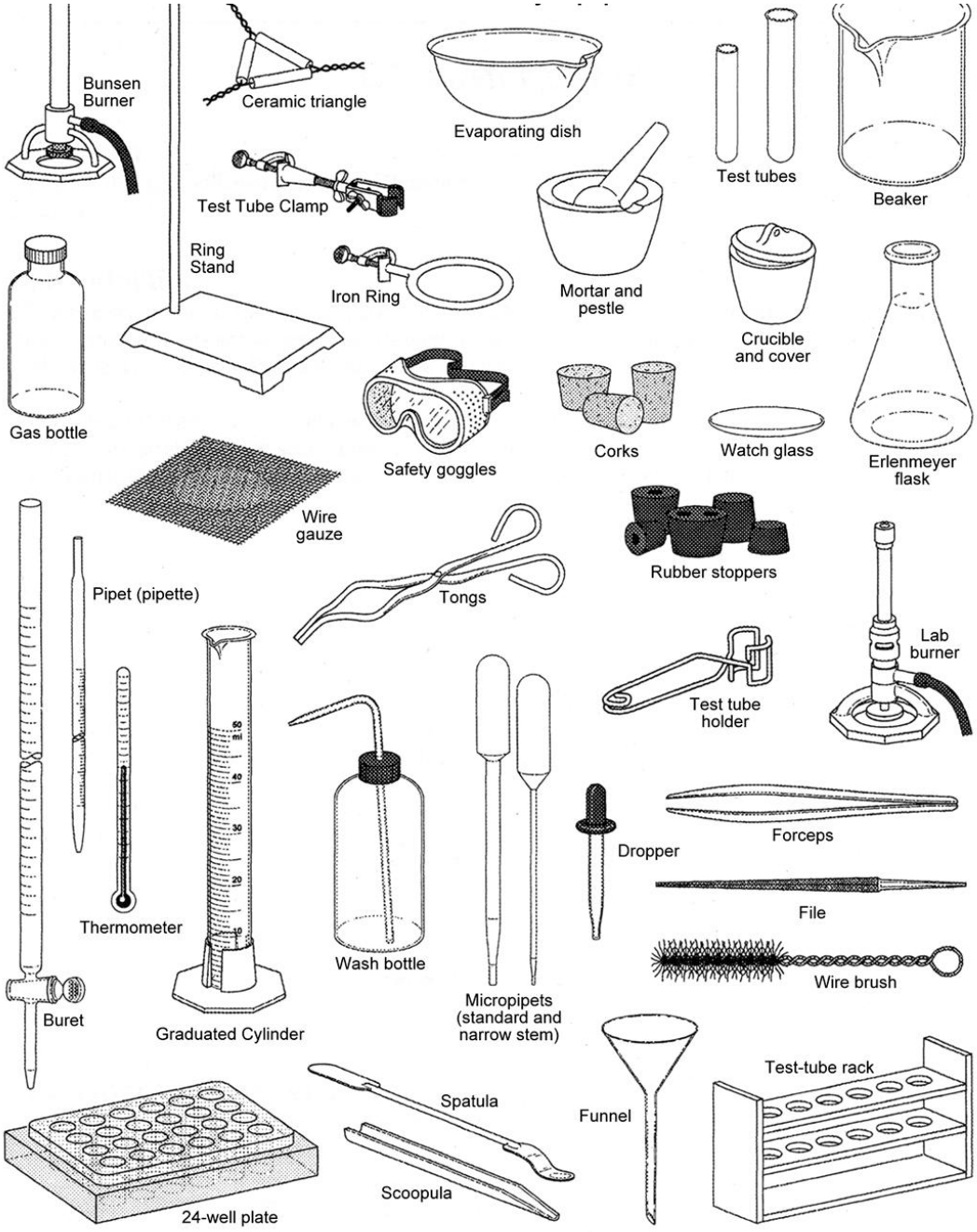
The following are some points to consider in choosing glassware or plasticware:

a. Unless instructed otherwise, borosilicate or polyethylene bottles may be used for the storage of reagents and standard solutions.
b. Dilute metal solutions are prone to plate out on container walls over long periods of storage. Thus, dilute metal standard solutions must be prepared fresh at the time of analysis.
c. For some operations, disposable glassware is entirely satisfactory.
d. Plastic bottles of polyethylene and Teflon have been found satisfactory for the shipment of water samples. Strong mineral acids (such as

sulfuric acid) and organic solvents will readily attack polyethylene and are to be avoided.

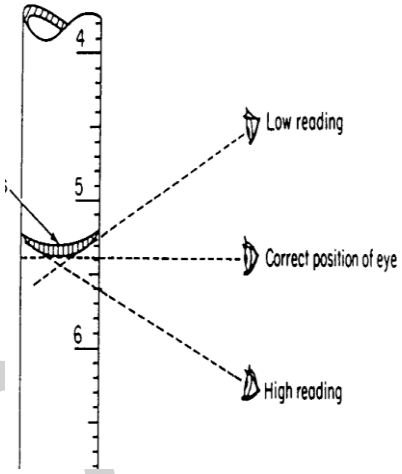
e. Borosilicate glassware is not completely inert, particularly to alkalies. Therefore; standard solutions of silica, boron, and the alkali metals are usually stored in polyethylene bottles.

Volumetric Analyses

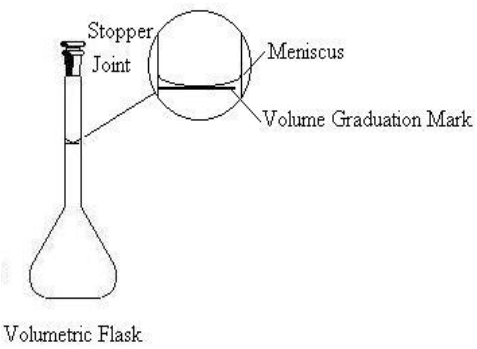


By common usage, accurately calibrated glassware for precise measurements of volume has become known as volumetric glassware. This group includes volumetric flasks, volumetric pipettes, and accurately calibrated burettes. Less accurate types of glassware including graduated cylinders and serological and measuring pipettes also have specific uses in the analytical laboratory when exact volumes are unnecessary.

The precision of volumetric work depends in part upon the accuracy with which volumes of solutions can be measured. There are certain sources of error that must be carefully considered. The volumetric apparatus must be read correctly; that is, the bottom of the meniscus should be tangent to the calibration mark.



There are other sources of error, however, such as changes in temperature, which result in changes in the actual capacity of glass apparatus and in the volume of the solutions.



Volumetric Flask

The capacity of an ordinary glass flask of 1000-ml volume increases 0.025 ml/deg with rise in temperature, but if the flask is made of borosilicate glass, the increase is much less. One thousand milliliters of water or of most 0.1N solutions increases in volume by approximately 0.20 ml/deg increase at room temperature. Thus solutions must be measured at the temperature at which the apparatus was calibrated. This temperature (**usually 20°C**) will be indicated on all volumetric ware.

There may also be errors of calibration of the apparatus; that is, the volume marked on the apparatus may not be the true volume. Such errors can be eliminated only by recalibrating the apparatus or by replacing it.

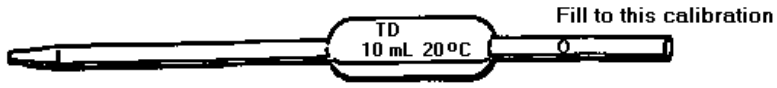
Volumetric apparatus is calibrated to contain or to deliver a definite volume of liquid. This will be indicated on the apparatus with the letters "TC" (TO CONTAIN) or "TD" (TO DELIVER). Volumetric flasks are calibrated to contain a given volume and are available in various shapes and sizes.

A. Volumetric pipettes are calibrated to deliver a fixed volume. The usual capacities are 1 through 100 ml although micropipettes are also available.

In emptying volumetric pipettes, they should be held in a vertical position and the outflow should be unrestricted. The tip of the pipette is kept in contact with the wall of the receiving vessel for a second or two after the free flow has stopped. *The liquid remaining in the tip is not removed*; this is most important.

B. Measuring and serological pipettes should also be held in a vertical position for dispensing liquids; however, the tip of the pipette is only touched to the wet surface of the receiving vessel after the outflow has ceased. For those pipettes where the small amount of liquid remaining in the tip is to be blown out and added, indication is made by a frosted band near the top.

Volumetric Pipets



Measuring Pipets

Mohr pipet

Serological pipet

C. Burettes are used to deliver definite volumes. The more common types are usually of 25- or 50-ml capacity, graduated to tenths of a milliliter, and are provided with stopcocks. For precise analytical methods in microchemistry, microburettes are also used.

Automatic burettes with reservoirs are also available ranging in capacity from 10 to 100 ml. Reservoir capacity ranges from 100 to 4,000 ml.

General Rules regarding the manipulation of a burette:

i. Do not attempt to dry a burette that has been cleaned for use, but rinse it two or three times with a small volume of the solution with which it is to be filled.

ii. Do not allow alkaline solutions to stand in a burette because the glass will be attacked, and the stopcock, unless made of Teflon, will tend to freeze.

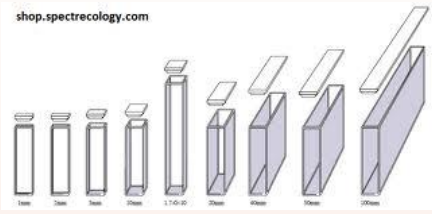
iii. A 50-ml burette should not be emptied faster than 0.7 ml/s, otherwise too much liquid will adhere to the walls and as the solution drains down, the meniscus will gradually rise, giving a high false reading.

It should be emphasized that improper use or reading of burettes can result in serious calculation errors.

iv. In the case of all apparatus for delivering liquids, the glass must be absolutely clean so that the film of liquid never breaks at any point. Careful attention must be paid to this fact or the required amount of solution will not be delivered.

Special Cleaning Requirements

Absorption cells, used in spectrophotometers, should be kept scrupulously clean, free of scratches, fingerprints, smudges, and evaporated film residues.



Glassware to be used for phosphate determinations should not be washed with detergents containing phosphates. This glassware must be thoroughly rinsed with tap water and distilled water.

Bottles to be used for the collection of samples for organic analyses should be rinsed successively with chromic acid cleaning solution, tap water, distilled water, and, finally, several times with a redistilled solvent such as acetone . Caps are washed with detergent, rinsed with tap water, distilled water, and solvent. Liners are treated in the same way as the bottles and are stored in a sealed container.

Cleaning of Glass and Porcelain

The method of cleaning should be adapted to both the substances that are to be removed, and the determination to be performed.

Water-soluble substances are simply washed out with hot or cold water, and the vessel is finally rinsed with successive small amounts of distilled water. Other substances more difficult to remove may require the use of a detergent, organic solvent, dichromate cleaning solution, nitric acid, or **AQUA REGIA** (25 percent by volume concentrated HNO_3 in concentrated HCl).

In all cases it is good practice to rinse a vessel with tap water as soon as possible after use. Material allowed to dry on glassware is much more difficult to remove.

Dichromate cleaning solution (chromic acid) is a powerful cleaning agent; however, because of its destructive nature upon clothing and upon laboratory furniture, extreme care must be taken when using this mixture. If any of the solution is spilled, it must be cleaned up immediately.

A persistent greasy layer or spot may be removed by acetone or by allowing a warm solution of sodium hydroxide, about 1 g per 50 ml of water, to stand in the vessel for 10 to 15 min; after rinsing with water, dilute hydrochloric acid, and water again, the vessel is usually clean. Alcoholic potassium hydroxide is also effective in removing grease. To dry glass apparatus, rinse with acetone and blow or draw air through it.

Glassware may be dried for immediate use by rinsing with redistilled acetone. Otherwise glassware may be oven dried or drip dried. Glassware should be stored immediately after drying to prevent any accumulation of dust and stored inverted or with mouth of glassware covered with foil.

Disposable Glassware

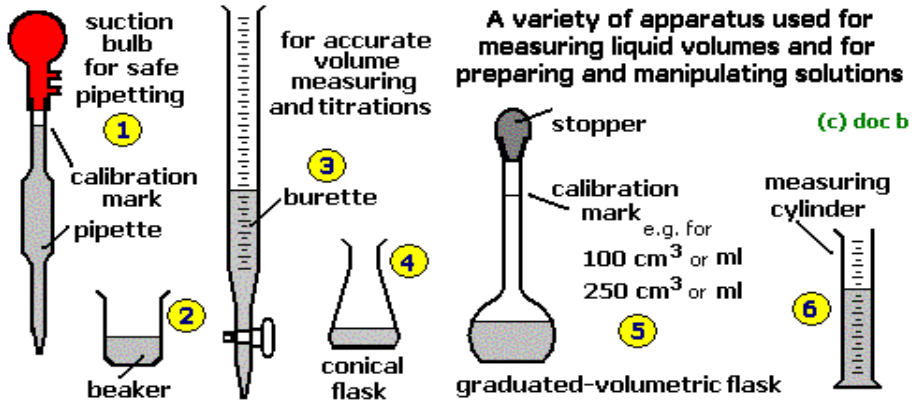
When the risk of washing a pipette for reuse becomes too great, as in the case of use with toxic

materials, or when the cost of washing glassware becomes prohibitive, disposable vessels may be the answer, provided they meet the necessary specification. Various types are available including bacteriological, serological, and microdilution pipettes. Disposable glassware generally is made of soft glass although plastic vessels and pipettes are also available.

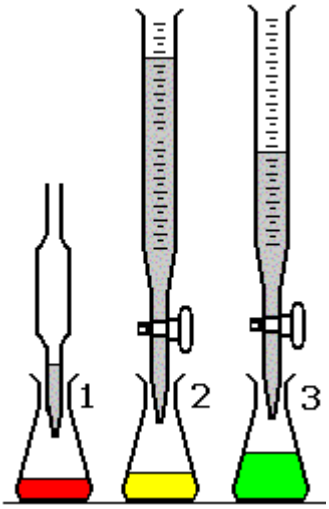
Glassware Proper Usage:

1. ***Graduated cylinders*** are used to measure small volume of liquids and solution for experiments.

2. ***Pipette***s (moher/ graduated) are used to deliver any precise volume within its range. A detailed way of using the pipette is described next:
   a. Prepare the pipette: clean the pipette with soap solution; rinse with several portion of tap water then with deionized water. No water droplets should adhere to the inner wall of the pipette. Transfer the liquid or solution that you intend to pipette from the reagent bottle to a beaker. Dry the pipette tip with a clean, dust free towel or tissue. Rinse through the pipette tip into a waste beaker. Using the pipette pump (never use your OWN MOUTH) draw 2-3 ml volumes into the pipette as rinse. Roll each rinse around in the pipette so that the solution washes the entire surface of the inner wall. Deliver each rinse through the pipette tip into a waste beaker.
   b. Fill and operate the pipette: to fill the pipette, place the tip well below the surface of the solution in the beaker. Then using the pipette pump, draw the solution into the pipette until its level is 3-5 mm above the mark.
   c. Deliver the solution: remove the tip from the solution, dry the tip with a dust free towel, and holding the pipette in a vertical position over a waste beaker, control the delivery of the excess solution from the pipette by lightly pressing the release bottom until the meniscus is at the mark. Remove any suspended droplet on the pipette tip by touching the inside wall of the waste beaker. Deliver the solution to the receiving vessel; keep the tip above the level of liquid and against the wall of the receiving vessel. Do not blow or shake out the Last bit of solution that remains in the tip; this liquid has been included in the calibration of the pipette.
   d. Clean the pipette: once the use of pipette is complete, rinse the pipette several times with deionized water. Roll each rinse to flush the inner wall of the pipette and drain through the tip.



3. ***Burettes*:** It may be used when samples of various sizes must be dispensed or measured precisely.
   The burette consists of a narrow calibrated glass tube, fitted at the bottom with a valve for controlling the flow of liquid. The valve is commonly called stopcock.
   A burette must be cleaned before use. If the burette is not completely clean, the level of precision is not attained.
   a. Clean the burette with soap and water, using a special long handed burette brush to scrub the interior of the glass. Then rinse the burette with tap water.
   b. Do not attempt to admit water directly to the burette from the water tap. Fill a beaker with tap water, and pour from the beaker into the burette.
   c. Finally, rinse the burette several times with distilled water. Before use, the burette should again rinse with distilled water.



   Many of the reagent solutions used in burette may attack the glass of the burette if they are not removed. This would destroy the calibration.

   A common mistake made by junior students is to fill the burette with the reagents solution to be dispensed to exactly the 0.00 mark. This is not necessary or desirable in most cases and is a waste of time. The burette should be filled to a level that is comfortable for you to read (based on your height). The precise initial liquid level reading of the burette should be taken before the solution is dispensed and again after the liquid is dispensed. The reading should be made to the exact and precise ml. The volume of the liquid dispensed is then obtained by simple subtraction of the two volume readings.

Techniques in Preparation Solutions

There are five units of concentration that are particularly useful to chemists. The first three: Molality, Molarity and Normality are dependent upon the mole unit. The last two: percent by volume and percent by weight have nothing to do with mole, only weight or volume of the solute or substance to be diluted, versus the weight or volume of the solvent or substance in which the solute is diluted. Percentages can also be determined for solids within solids.

1. **Molarity**"M"

The molar unit is probably the most commonly used chemical unit of measurement. Molarity is the number of moles of a solute dissolved in a liter of solution. A molar solution of sodium chloride is made by placing 1 mole of a solute into a 1-liter volumetric flask.

$$\boldsymbol{M = \frac{\#\ moles\ of\ sloute}{1\ L\ of\ solvent}}$$

2. **Molality "m"**

The molal unit is not used nearly as frequently as the molar unit. A molality is the number of moles of solute dissolved in one kilogram of solvent. Be careful not to confuse molality and molarity. Note that the solvent must be weighed unless it is water.

$$\boldsymbol{m = \frac{\#\ moles\ of\ sloute}{1\ Kg\ of\ solvent}}$$

3. **Normality"N"**

A measure of concentration that is equal to the gram equivalent weight per liter of solution. Gram equivalent weight is a measure of the reactive capacitity of a molecule.

$$\boldsymbol{N = \frac{\#\ eq.wt.}{1\ L\ of\ solvent}}$$

For acid reactions, a 1 M H_2SO_4 solution will have normality (N) of 2 N because 2 moles of H+ ions are present per liter of solution. For sulfide precipitation reactions, where the SO_4^- ion is the important part, the same 1 M H_2SO_4 solution will have a normality of 1 N.

4. **Percent by weight**: To make up a solution based on percentage by weight, one would simply determine what percentage was desired.

For example, a 20% by weight aqueous solution of sodium chloride, and the total quantity to be prepared.
If the total quantity needed is 1 kg, then it would simply be a matter of calculating 20% of 1 kg which, of course is:

0.20 NaCl * 1000 g/kg = 200 g NaCl/kg.

In order to bring the total quantity to 1 kg, it would be necessary to add 800g water.

5. **Percent by volume**: Solutions based on percent by volume are calculated the same as for percent by weight, except that calculations are based on volume. Thus one would simply determine what percentage was desired (for example, a 20% by volume aqueous solution of sodium chloride) and the total quantity to be prepared.

If the total quantity needed is 1 liter, then it would simply be a matter of calculating 20% of 1 liter which, of course is:

0.20 NaCl * 1000 ml/l = 200 ml NaCl/l.

Percentages are used more in the technological fields of chemistry (such as environmental technologies) than they are in pure chemistry.

Dilution

When preparing a dilution, decide the volume and molar concentration of the resulting solution you require. Use the following equation to determine how much of the concentrated reagent is needed to prepare the diluted solution,

No. of moles (reagent) = No. of moles (dilution)

$$\mathbf{M_{Reagent} \times V_{Reagent} = M_{Dilution} \times V_{Dilution}}$$

Where M is molarity and V is volume.
Slowly add the calculated volume of concentrated reagent to the proper-size volumetric flask half filled with distilled or de-ionized water and swirl the flask to mix. Once the solution is at room temperature, dilute to the mark with water and invert the flask several times to mix.

Dilution Factor (DF)

Dilution: is the mixing of a small accurately measured sample with a large volume of sterile water or normal saline called (diluents or dilution blank)

$$\boldsymbol{Dilution} = \frac{\boldsymbol{Volume\ of\ Sample}}{\boldsymbol{Total\ Volume\ of\ (sample\ +\ diluent)}}$$

$$\boldsymbol{Dilution\ Factor\ DF} = \frac{\boldsymbol{Total\ Volume\ of\ (sample\ +\ diluent)}}{\boldsymbol{Volume\ of\ sample}}$$

Writing a Lab Report

Cover Page

Write the Name of your University, School, Department, Subject name then Identify yourself and your partner/s, write you ID number, Date of Performing the experiment, Date of Submitting the report.

Purpose/ Objective

This gives the objective of the Experiment. What concept or skill was highlighted by this Experiment. Ask yourself "Why did we do this activity? What was I supposed to learn or practice?" Sometimes the purpose can be stated in one sentence. Other times it may be necessary to add some extra information to narrow the scope of the activity.

Introduction/ General Information

Based on your text books, write about the subject of the experiment, an introduction has been already included so you may either add to it or you can write a new one. You need to paraphrase any material you take from the manual.

Materials & Reagents

This should be a sentence or two that lists the materials that were needed to carry out this activity. This could also be in the form of a table.

Procedure

This is probably one of the most difficult parts of the report for the beginner. Most of the writing you have done up to this point has included a lot of descriptive language. Technical writing is very "cut and dried" by comparison. All you are trying to convey is a mental picture of what you did. Ordinal phrases are not necessary. The order of events is conveyed by the sentence order in the description. Remember that your audience should be able to repeat your procedure if they wish to do so. Write your description of what was done so that the reader can visualize the set-up. Be sure to include reference to any equipment that you used (The mass was taken on a balance.) A diagram or picture of the apparatus may be helpful but should not replace a good verbal description. Be very specific in your instructions. Emotions (This was hard. or this was fun.) are not necessary and detract from the purpose of this section.

Observations & Data

This section should include only those things that you saw, heard, touched, or smelled (taste is out since we never taste anything in a science lab). This includes both quantitative (numerical) and qualitative (sensual, not emotional) observations. Quantitative observations are best presented in data tables.

Qualitative observations may be organized in table form or paragraph form.

The goal of this section is to present the data that was collected in the activity in a clear and easily understood format. Units are necessary for any measurement. If you are unsure about whether something should be included in the data section, ask yourself "How did I get this piece of information? What instrument did I use to collect this information?" If you are giving a value that you did not measure directly (such as density) it should not be included as data.

Analysis of Data/ Results

This is the section where you will show any calculations that you made using the data you collected. Give the formula you will use for each type of calculation. You should show which measurements you are plugging into each calculation and then show the solution. Once you have shown **a sample calculation**, you may use a data table to show other calculated values of the same type. This is also the appropriate place to explain how the measurements relate to each other. This is the proper place to discuss anything that happened during the activity that may have affected your measurements.

You should also, mention the sources of error, and their effect on the results that were attained during the experiment.

Conclusions

This is the section of your report where you discuss how the purpose of the activity relates to the analysis of your data. In other words, what did you learn. Stick to the facts; do not comment on whether or not you enjoyed the activity. Be specific in your statements. If the results of the activity were not satisfactory, suggest how the activity could be improved to give better data. Did the activity raise questions that cannot be answered with the data you collected? This is the place to mention them. Remember, conclusions are connections that are not obvious on the surface.

Attachment

You have to attach the lab report that was signed by the RTA so that the report can be accepted. A penalty of deducting three marks will be given for each report missing the data sheet.

Finishing touches

1. Be sure you have filled in the information in the header. Give your name, your partner's name and the date you did the activity.
2. Proofread one last time to be sure that you have used correct grammar and punctuation. Remember that Spell Check on the computer will not catch a word that is used incorrectly if it is spelled correctly (there-their).

(1) Sampling

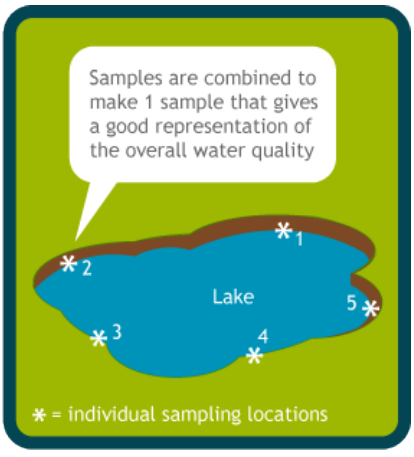
The analytical results of a sample are only as accurate as the quality of the sample taken. If your technique for collecting samples is poor, then no matter how accurate your lab procedures are, the results will be poor. By sampling according to set procedures, you reduce the chance of error and increase the accuracy of your sample results.
This draft will cover the proper methods of sampling, sample preparation, documentation and sampler cleaning.

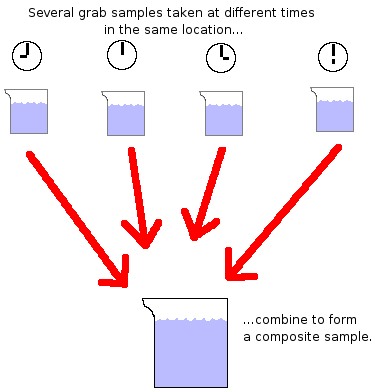
Sample Types.

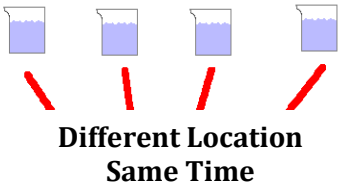
There are mainly three types of Water/Wastewater samples:

1. ***Grab samples***: Grab sample shows the characteristics of the water at the time of sampling only and should not exceed a sampling time of 15 minutes. Grab sampling is done for such procedures as batch discharge, constant waste stream characteristics and when the parameter tested deteriorates rapidly such as cyanides, volatile organic compounds and phenols .

2. ***Composite Samples*:** These are individual samples taken and deposited in the same collection bottle. There are two methods that are most common to collecting composite samples.
   a. Time paced is when samples are collected at set increments of time.
   b. Flow paced samples: which are taken when a measured volume of water flows over the sensor of a flow meter, which is more preferred; since it gives the most representative sample. Metals, Base/Neutral/Acid Organics, BOD and TSS samples may be collected by this method.







3. ***Integrated samples***: Those are combination of grab samples collected at the same time but at different locations. Integrated samples are required when the knowledge of the volume, movement, and composition of the various parts of the water being sampled usually is required. Collecting integrated samples is a complicated and specialized process that must be described adequately in a sampling plan for each test.

Sampling Locations:

1. **Surface water** is what is seen on the Earth's surface. It can be either flowing water, like oceans, rivers and streams, or stored in natural depressions, like lakes and water holes.

Surface water can be:

- Permanent: flowing or held in natural depressions throughout the year
- Semi–permanent: flowing or held for only part of the year
- Constructed: held in structures ranging from dams to a water tank that catches rain.

2.**Groundwater** is water stored in–between the particles of soil underground. The soil acts like a sponge eventually forming a groundwater reservoir, called an aquifier.

There are two main forms of groundwater.

- Superficial: usually between 3 to 20 meters down. It is the most accessible because it is near the surface.
- Confined: deep down below the surface of the Earth. Special equipment is needed to access this water.

Groundwater always becomes surface water sooner or later.

3. **Water distribution systems:** are ways of controlling the flow and direction of both surface water and groundwater. They are the link between the water supply source and the consumer. They include:

- Irrigation systems – used in agriculture
- bore lines – used to deliver bore water
- Scheme water systems – used to deliver water to households.

Some of these systems have been in use for decades, so sampling and testing is used to determine the health of the pipes and water.

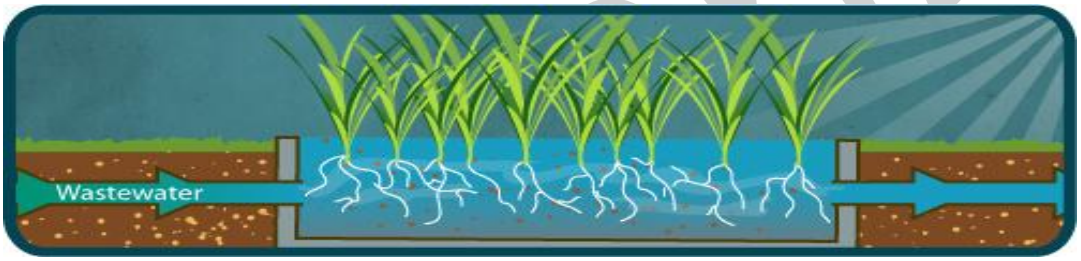
4. **Water treatment system:** Water is used for many purposes. Water treatment systems function to make untreated water suitable for a particular purpose. For example, as drinking water or for industrial processes.

   Examples of water treatment systems

   A basic reed bed treatment system

   A reed bed system (also called an artificial wetland) is used to filter the contaminants out of wastewater naturally. The systems are designed to make the water flow slowly through a series of specially chosen plants that take up contaminants as they grow. As a result, the wastewater is cleaned naturally.

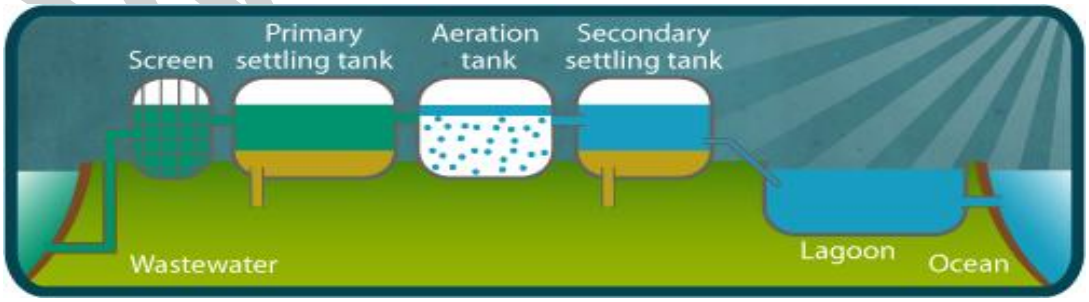
   The water isn't drinkable but sometimes it can be discharged into natural water systems or be reused for some purposes.



Wastewater treatment systems

Wastewater treatment systems work in a similar way to reed bed systems. But they don't do it naturally. Instead they use chemicals to clean the wastewater. These systems are used on a large scale to reduce the organic matter in wastewater before it is discharged into a water body (usually the ocean).

The wastewater is filtered and then disinfected with chemicals like chlorine to



make it suitable for human use or to be discharged into the environment.

Sampling Equipment:

Typical sampling equipment when taking water samples it's important to:

- Use the correct sampling equipment
- Use the correct personal protective equipment (PPE)
- Record the necessary information correctly
- Check all equipment before carrying out sampling.

There are many different types of equipment used for sampling water.

Groundwater sampling is conducted using groundwater bores and low flow pumps. Using this kind of equipment requires advanced knowledge. You may have an opportunity to observe this sort of water sampling.
Equipment typically used when sampling surface water includes:

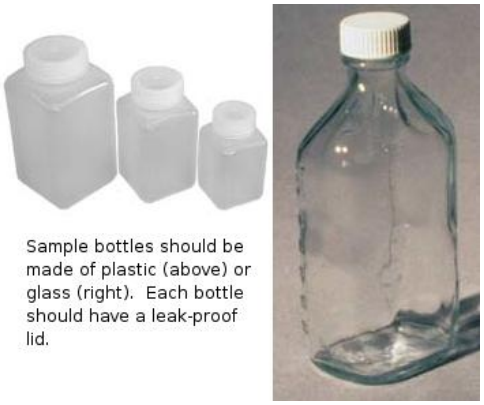
1. New, prepared plastic and glass sampling bottles, clearly labelled with a marker pen.
2. Sterile disposable gloves to avoid contamination.
3. A cooler for storing and transporting filled sample bottles.
4. Ice or dry ice to maintain samples at the correct temperature.
5. Any equipment needed for taking on-site tests (for example: thermometer, conductivity meter, pH meter)
6. Communication equipment (for example: mobile phone, walkie-talkie)
7. Any maps needed

Collection Sample Container Cleaning

1) Plastic (polyethylene)
- Wash with hot water (detergent optional).
- Rinse with acid (nitric for metals).
- Rinse with tap water, then three times with Distilled water.

2) Glass
- Wash with hot water (detergent optional).
- Rinse with acid (nitric for metals).
- Rinse with tap water, then three times with Distilled water.
- Dry in contamination-free area.
- Rinse glass containers with an interference-free, redistilled solvent (e.g., acetone, isopropanol or methylene chloride) for extractable organics.
- Rinse glassware for volatile organics with isopropanol.
- Rinse with tap water, and then at least three times with deionize water.
- Dry in contaminant-free area.

3) Ice Chests



Sample bottles should be made of plastic (above) or glass (right). Each bottle should have a leak-proof lid.

Sample Labelling:

Correct labelling of samples is essential. They need to be easily identified at all times. Without proper labelling, all samples can look alike and mistakes can happen.

Site Name: _____
Sample ID Number: _____
Date: _____ Time: _____
Location: _____
Container Size: _____
Container Type: _____
Sample Type (e.g., grab, composite): _____
Analysis: _____
Preservative: _____
Dechlorination: _____
Collected by (initials): _____

Water sample labels must include:

a) A unique identifying code for cross-referencing
b) Date of sampling.
c) They can also include:
d) The location and name of the sampling site
e) The name of the sampler
f) The time of sampling
g) The type of sample
h) Any observations that might affect test results.

You must always keep a record of your activity when sampling water. This is done using field sheets. Field sheets are forms used to record data relating to each sample.

Sampling Event Report Form

Collection Information	Date:		Site Name:	
Sample Owner and/or Collector:	Signature:			
Level of PPE Used:	Weather Conditions:			
Additional Agencies Involved:	Agency Contact Information:			
Signature of Agency Representative(s):				
Site and Sample Description				
Sample ID	**Sample Location**	**Time**	**Sample Amount (volume or weight)**	**Sample Type (Matrix)**
Matrix: DW = Drinking Water, RW = Reservoir Water, UW=Untreated Water, SD = Sediment, SL = Sludge, SO = Soil, SM = Misc. Solid Material				
Incident Details				
Describe the number of people exposed and the types of symptoms they are experiencing:				
General conditions of exposed flora and fauna (if available):				
Describe the event and reason for sample collection:				

It will have all the information relating to a single sample.

Chain of Custody (COC)

If the samples you take need to be sent to a laboratory for testing or if they need to be stored, a Chain of Custody (COC) must also be used.

REMEMBER:
Never sample alone and always be able to contact someone!

A Chain of Custody (COC) is a form that outlines:

1. The location a sample was taken
2. The date and time of the sampling

Chain of Custody Form

Site Name:			Sample Owner/Collector:				
Contact Information:			Signature				
Sample ID	Collection Date/Time (24 h)	Sample Location	Sample Type (Matrix)	Grab/ Composite	Preservative(s)	No./Type of Bottles	Comments
Matrix: DW = Drinking Water, RW = Reservoir Water, UW=Untreated Water, SD = Sediment, SL = Sludge, SO = Soil, SM = Misc. Solid Material							
Relinquished By:		Received by:		Date/Time:			
Relinquished By:		Received by:		Date/Time:			
Relinquished By:		Received by:		Date/Time:			
Relinquished By:		Received by:		Date/Time:			
Relinquished By:		Received by:		Date/Time:			
Dispatched by:	Date/Time:	Received by:	Date/Time				
Method of Sample Transport							
Shipper:	Phone No.:	Tracking No.:					

3. The sampling equipment used
4. The type of laboratory testing required
5. An analysis of the samples taken.

A Chain of Custody is a legal document used to trace who has been in control of the samples from the time of collection through analysis, reporting and disposal.
When you submit your samples to a laboratory you must give them a COC. The lab will make a copy of the COC to give you and the results will be faxed or emailed to a manager, usually within two weeks.

Sampling Plan

All equipment needs to be properly checked before sampling takes place. This includes communication equipment and transport.

All sampling plans include basic information about who is doing the sampling, when it is done and a job number.

1. **Job brief:** clearly describe why sampling needs to be done. What evidence is there to justify the project?

2. **Types of sample:** note whether you will be taking grab or composite samples, or both. What kind of containers will you need?
3. **Location and access:** include where the sampling will take place and what you will need to access it. What will you need to access this location?
4. **Sampling equipment:** decide what equipment you will need to carry out the sampling. List everything you need including Personal Protective Equipment (PPE) and records sheets.
5. **Transport needs:** decide what you will need to take to transport any samples needed for analysis.
6. **Types of test:** list what tests you will be sampling for. Include any tests you will be doing on site, as well as any tests that will be done in a laboratory.
7. **Testing equipment:** list any equipment you will need to do the onsite tests.

Sampling Methods

a. Manual sampling: Manual sampling involves minimal equipment but may be unduly costly and time-consuming for routine or large-scale sampling programs. It requires trained field technicians and is often necessary for regulatory and research investigations; for which critical appraisal of field conditions and complex sample collection techniques are essential. Manually collect certain samples, such as waters containing oil and grease.

b. Automatic sampling: Automatic samplers can eliminate human errors in manual sampling, can reduce labor costs, may provide the means for more frequent sampling, and are used increasingly

Number of Samples

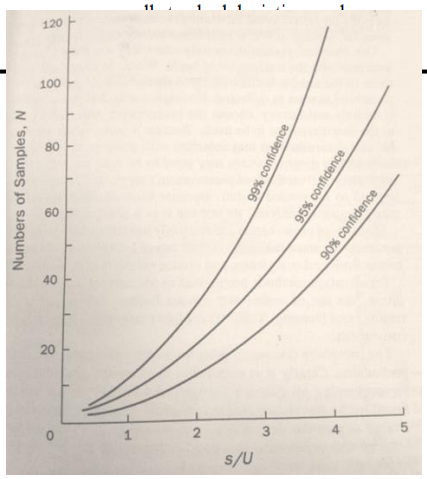
Because of variability from analytical and sampling procedures (i.e., population variability), a single sample is insufficient to reach any reasonable desired level of confidence. If an overall standard deviation (i.e., the standard deviation of combined sampling and analysis) is known, the required number of samples for a mobile matrix such as water may be estimated as shown

$$N \geq \left(\frac{ts}{U}\right)^2$$

where:

N = number of samples,

t = Student-t statistic for a given confidence level,



Sample Volumes

Collect a 1-L sample for most physical and chemical analyses. For certain determinations, larger samples may be necessary. Table 1060:I

(Included) lists volumes ordinarily required for analyses. It is strongly recommended that the laboratory that will conduct the analyses also be consulted to verify the analytical needs of sampling procedures.
Do not use samples from the same container for multiple testing requirements (e.g., organic, inorganic, radiological, bacteriological, and microscopic examinations) because methods of collecting and handling are different for each type of test.

Sample Storage and Preservation

Complete and unequivocal preservation of samples, whether domestic wastewater, industrial wastes, or natural waters, is a practical impossibility because complete stability for every constituent never can be achieved. At best, preservation techniques only retard chemical and biological changes that inevitably continue after sample collection.

Sample Storage before Analysis

a. Nature of sample changes: Some determinations are more affected by sample storage than others.

- Certain cations are subject to loss by adsorption on, or ion exchange with, the walls of glass containers. These include aluminum, cadmium, chromium, copper, iron, lead, manganese, silver, and zinc, which are best collected in a separate clean bottle and acidified with nitric acid to a pH below 2.0; to minimize precipitation and adsorption on container walls.
- Temperature changes quickly; pH may change significantly in a matter of minutes; dissolved gases (oxygen, carbon dioxide) may be lost. Because changes in such basic water quality properties may occur so quickly, determine temperature, reduction-oxidation potential, and dissolved gases in situ and pH, specific conductance, turbidity, and alkalinity immediately after sample collection. Many organic compounds are sensitive to changes in pH and/or temperature resulting in reduced concentrations during storage.
- Changes in the pH-alkalinity-carbon dioxide balance may cause calcium carbonate to precipitate, decreasing the values for calcium and total hardness. Microbiological activity may affect the BOD concentration. Color, odor, and turbidity may increase, decrease, or change in quality.
- Zero head-space is important in preservation of samples with volatile organic compounds and radon. After capping or sealing bottle, check for air bubbles by inverting and gently tapping it; if one or more air bubbles are observed then, if practical, discard the sample and repeat refilling bottle with new sample until no air bubbles are observed (this cannot be done if bottle contained preservatives before it was filled).

Preservation Techniques

To minimize the potential for volatilization or biodegradation between sampling and analysis, keep samples as cool as possible without freezing. Preferably pack samples in crushed or cubed ice or commercial ice substitutes before shipment.

◊ Avoid using dry ice because it will freeze samples and may cause glass containers to break.

◊ Dry ice also may effect a pH change in samples.

◊ Keep composite samples cool with ice or a refrigeration system set at 4°C during compositing.

◊ Analyze samples as quickly as possible on arrival at the laboratory. If immediate analysis is not possible, preferably store at 4°C.

◊ No single method of preservation is entirely satisfactory; choose the preservative with due regard to the determinations to be made.

◊ Use chemical preservatives only when they do not interfere with the analysis being made.

Preservation methods are limited to pH control, chemical addition, the use of amber and opaque bottles, refrigeration, filtration, and freezing. Table ( ) lists preservation methods by constituent.

PARAMETERS	CONTAINERS	SAMPLE VOLUME (mL)	PRESERVATION	MAXIMUM HOLDING TIME
		WATER		
		ROUTINE WATER SAMPLE		
Alkalinity	Cubitainer or Glass	100	Cool to 4 °C, dark	14 days
Total Suspended Solids/Suspended Solids	Cubitainer or Glass	400	Cool to 4 °C, dark	7 days
Chloride (Cl)	Cubitainer or Glass	100	None required	28 days
Sulfate (SO_4)	Cubitainer or Glass	100	Cool to 4 °C, dark	28 days
Orthophosphate (OPO_4)	Cubitainer or Glass	150	Filter ASAP; Cool to 4 °C, dark	48 hours
Nitrate + Nitrite ($NO_3 + NO_2$)	Cubitainer or Glass	150	1-2 mL conc. H_2SO_4 to pH <2, and Cool to 4 °C, dark	28 days
Ammonia (NH_3)	Cubitainer or Glass	150	1-2 mL conc. H_2SO_4 to pH <2, and Cool to 4 °C, dark	28 days
Total Phosphorus (TPO_4)	Cubitainer or Glass	150	1-2 mL conc. H_2SO_4 to pH <2, and Cool to 4 °C, dark	28 days
Total Organic Carbon (TOC)	Cubitainer or Glass	100	1-2 mL conc. H_2SO_4 to pH <2, and Cool to 4 °C, dark	28 days
Chlorophyll *a*	Quart cubitainer	1,000	Cool to 4 °C, dark	Filter 48 hours Filters may be stored frozen up to 30 days
Nitrite	Quart cubitainer	50	Cool to 4 °C, dark	48 hours
Total Dissolved Solids	Quart cubitainer	250	Cool to 4 °C, dark	7 days
Hardness	Quart cubitainer	250	2 mL conc. HNO_3 to pH<2; Cool to 4 °C, dark **OR** 2 mL conc. H_2SO_4 to pH <2; Cool to 4 °C, dark	6 months
		ROUTINE WATER SAMPLE COLLECTION PROCEDURE		

- Label container before collection with a unique sample identifier number, Station Location, Date and Sample Type
- Place an **X** on the container lid to identify the acidified sample.
- Open containers by pulling apart. Pre-rinsing cubitainers with ambient water is not necessary.
- Fill each container with ambient water by submerging container approximately one foot below the surface mid-stream until filled.
- Place sample on ice immediately. Acidify the **X** container as soon as possible.
- Place on ice and ship as soon as possible.

Table 3. Summary of grab sample collection methods, preservation, storage and handling requirements–Continued

PARAMETERS	CONTAINERS	SAMPLE VOLUME (mL)	PRESERVATION	MAXIMUM HOLDING TIME
WATER				
NON-ROUTINE WATER SAMPLES				
OIL AND GREASE	Glass container with teflon lined lid rinsed with hexane or methylene chloride	1,000	2 mL conc. H_2SO_4 to pH <2; cool to 4 °C, dark	28 days
PHENOLS	Glass container with teflon lined lid	1,000	2 mL conc. H_2SO_4 to pH <2; cool to 4 °C, dark	28 days
BIOCHEMICAL OXYGEN DEMAND	Gallon cubitainer	> 4,000	Cool to 4 °C; add 1g FAS crystals per liter if residual chlorine present	48 hours
CHEMICAL OXYGEN DEMAND	Quart cubitainer	110	2 mL conc. H_2SO_4 to pH <2; cool to 4 °C, dark	28 days
METALS-IN-WATER				
DISSOLVED (except Hg)	HNO_3 cleaned quart plastic container	1,000	Filter at sample site with 0.45 micron in-line filter[1] into ultra-pure[2] HNO_3 preacidified container to pH<2	6 months
DISSOLVED MERCURY	HNO_3 cleaned quart plastic container	1,000	Filter at sample site with 0.45 micron in-line filter[1] into ultra-pure[2] HNO_3 preacidified container to pH<2	28 days
TOTAL (except Hg)	HNO_3 cleaned quart plastic container	1,000	Preacidified container with 5 mL ultra-pure[2] HNO_3 to pH<2	6 months
TOTAL MERCURY (Hg)	HNO_3 cleaned quart plastic container	600	Preacidified container with 5 mL ultra-pure[2] HNO_3 to pH<2	28 days
HEXAVALENT CHROMIUM (filtered)	Plastic or glass	600	Cool to 4 °C, dark	24 hours; must notify lab in advance

METALS-IN-WATER SAMPLE COLLECTION PROCEDURES

DISSOLVED METALS (includes Hexavalent Chromium)
- Put on ***powder-free*** latex, polyethylene, or vinyl gloves using Clean Hands/Dirty Hands technique.
- Assemble pump[3], tubing, and filter.
- Immerse intake tubing directly into water 1ft. and pump approx. 500 mL of ambient water to flush tubing and filter.
- Fill precleaned, preacidified container with 600-1,000 mL of filtrate leaving some head space.

TOTAL METALS
- Put on ***powder-free*** latex, polyethylene, or vinyl gloves using Clean Hands/Dirty Hands technique.
- Assemble pump, and tubing without filter.
- Immerse intake tubing directly into water 1ft. and pump approx. 500 mL of ambient water to flush tubing
- Fill precleaned, preacidified container with 600-1,000 mL of filtrate leaving some head space.

NOTES
[1]Capsule Filter: 15 mm diameter or larger, tortuous path capsule filters, Gelman Supor™ 12175, or equivalent (Ref. EPA Method 1669).
[2]Nitric Acid, Ultra-pure, commercially known as Ultrex™, Ultrapure Reagent.
[3]Pump and pump apparatus–Required for use with the container method. Peristaltic pump–115 a.c., 12 volt d.c., internal battery, variable speed, single head, Cole-Parmer, portable, Masterflex L/S™, Catalog No. H-07570-10 drive with Quick Load pump head, Cat. No. H-07021-24, or equivalent (Ref. EPA Method 1669).

Time required:

An hour or less depending on the number of samples grabbed

Equipment and Reagents:

Ice box, sampling bottles, portable meters

Significant Experimental Hazards

- Student should be aware of hazards associated with the sampling waste water that contains many sorts of pathogens, personal protective equipment should be used.

Procedure:

This part should be written as it PERFORMED

Issues to consider for your practical report

- Determine the number of samples needed to be taken to measure a certain water constituent knowing that:
  - Case A
    - S = 0.5 mg/l
    - U = 95%
    - t= ±0.2
  - Case B
    - S = 0.5 mg/l
    - U = 99%
    - t= ± 0.1

- The link below shows a video that you need to watch and write a short report that summarizes the content. Screen shots are encouraged

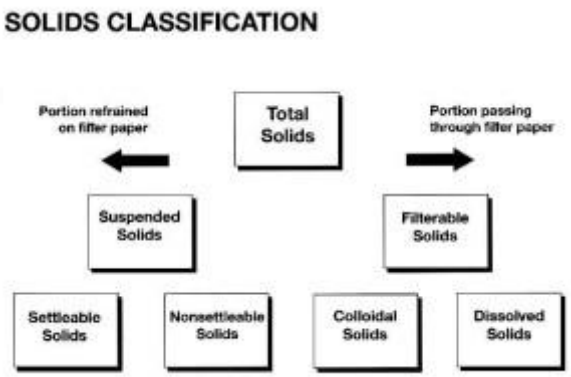
Sampling Wastewater at a Wastewater Treatment Facility
http://www.youtube.com/watch?v=exyVfFIn7TQ

(2) Solids in Water (A)

Solids (referred to as Total Solids, TS) is the matter that remains as residue upon evaporation at 103-105˚ C. Total solids include the Total Suspended Solids (TSS) and total dissolved solids (TDS).

Total Suspended Solids (TSS) are the amount of filterable solids in a water sample; usually the suspended solids contain, silt, stirred bottom sediment, organic matter; decaying plant matter, sewage treatment effluent. The filters are dried and weighed to determine the amount of total suspended solids in mg/l of sample.

Total Dissolved Solids (TDS) are those solids that pass through the filter with a pore size of 2µm or smaller, they are said to be non-filterable and includes mainly inorganic salts that yields ions such as (Na, Ca, Cl^-…), small amount of organic matter and dissolved gases. After filtration the filtrate (liquid) is dried at 103-105 °C and the remaining residue is weighed and calculated as mg/l of TDS.



The total solids content of wastewater are used in the design and process control of wastewater treatment facilities. Total dissolved solids are used to evaluate the suitability of water for both domestic supplies and industrial purposes. The total suspended solids (including the volatile fraction) are commonly monitored to evaluate the degree of pollution degree in natural waters and serves as a key process control parameter for wastewater treatment operation.

Most of the impurities in potable waters are in the dissolved state, principle as organic salts. Thus, the parameters TS and especially TDS are of primary importance here. Waters containing high concentrations of inorganic salts are not suitable as sources of drinking water, because such materials are often difficult to remove during treatment. Finished drinking waters containing more than 100 mg/l TDS are generally considered unacceptable. Waters of this type may also be unsuitable for agricultural purposes due to the harmful effects of high ionic concentrations on plants. TS also affect the clarity of water. Higher solids decrease the passage of light through water, thereby slowing photosynthesis by aquatic plants. Water will heat up more rapidly and hold more heat; this, in turn, might adversely affect aquatic life that has adapted to a lower temperature regime.

In the realm of municipal wastewater, suspended solids analysis is by far the most important gravimetric method. It is used to evaluate the strength of the raw wastewaters well as the overall efficiency of treatment. Furthermore, most wastewater treatment plants have effluent standard of 10-30 mg/l SS.

Time required:

Three hour or less depending on the number of samples determined

Equipment and Reagents:

Evaporating dishs
Crucibles
Measuring cylinder
Filter paper No 42
Balance
Oven
Funnel

Significant Experimental Hazards

- Student should be aware of hazards associated with the use of all glassware (cuts.)

Procedure:

I. Total solids (TS)

1. Take a clear dry Evaporating dish (which was kept at 103˚ C in an oven for 1 hour) of 400 ml and put an appropriate identification mark on it, using a pencil.
2. Weigh the Evaporating dish.
3. Pour **50 ml** of the thoroughly mixed sample, measured by the measuring cylinder, in the beaker.
4. Place the Evaporating dish in an oven maintained at 103˚ C for 2 hours, then remove it from the oven and place it in a desiccator to cool to room temperature.
5. After cooling the Evaporating dish, weigh it.
6. Find out the weight of solids in the beaker by subtracting the weight of the clean beaker determined in step(1)
7. Calculator total solids (TS) as follows:

$$\text{Total solids, TS (mg/l)} = \frac{\text{mg of solids in the Evaporating dish x 1000}}{\text{volume of sample, ml}}$$

Sources of TDS

Hard-water ions
- Ca^{2+}
- Mg^{2+}
- HCO_3^-

Fertilizers in agricultural runoff
- NH_4^+
- NO_3^-
- PO_4^{3-}
- $SO_4^=$

Urban runoff
- Na^+
- Cl^-

Salinity from tidal mixing, minerals, or returned irrigation water
- Na^+
- K^+
- Cl^-

Acidic rainfall
- H^-
- $NO3^-$

II. Total Dissolved Solids

1. Take a clear dry glass beaker (which was kept at 103˚ C in an oven for 1 hour) of 400 ml and put an appropriate identification mark on it.
2. Weight the beaker and note the weight.
3. Weigh a filter paper and write the weight.
4. Take a 100 ml of sample and filter it through a filter paper and collect the filtrate in a Evaporating dish.
5. Place the beaker in an oven maintained at 103˚ C for 2 hours, then remove it from the oven and place it in a desiccator to cool to room temperature.
6. After cooling the beaker, weigh it. Find out the weight of TDS in the beaker by subtracting the weight of the clean beaker determined in step(1)

$$\text{Total Dissolved Solids, TDS (mg/l)} = \frac{\text{mg of solids in the Evaporating dish} \times 1000}{\text{volume of sample, ml}}$$

If the suspended material clogs the filter and prolongs filtration, it may be necessary to increase the diameter of the filter or decrease the sample volume.

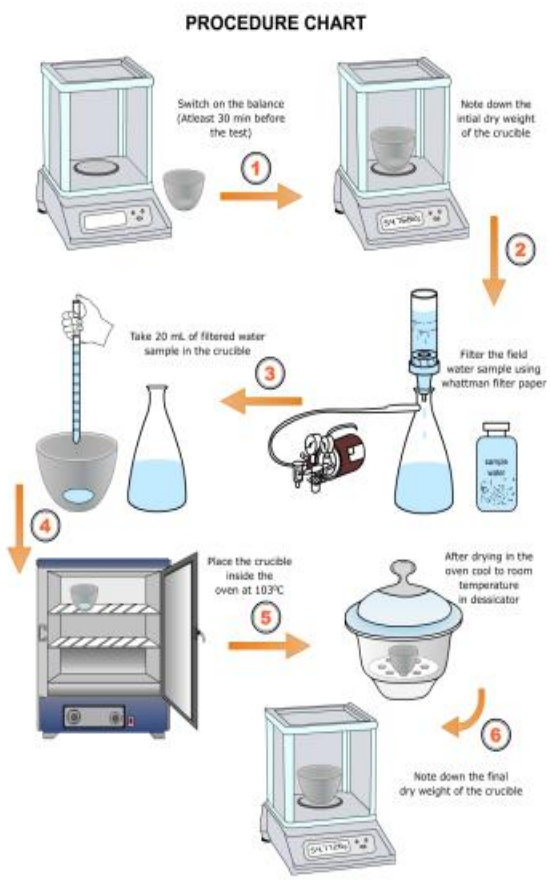
III. Total Suspended Solids

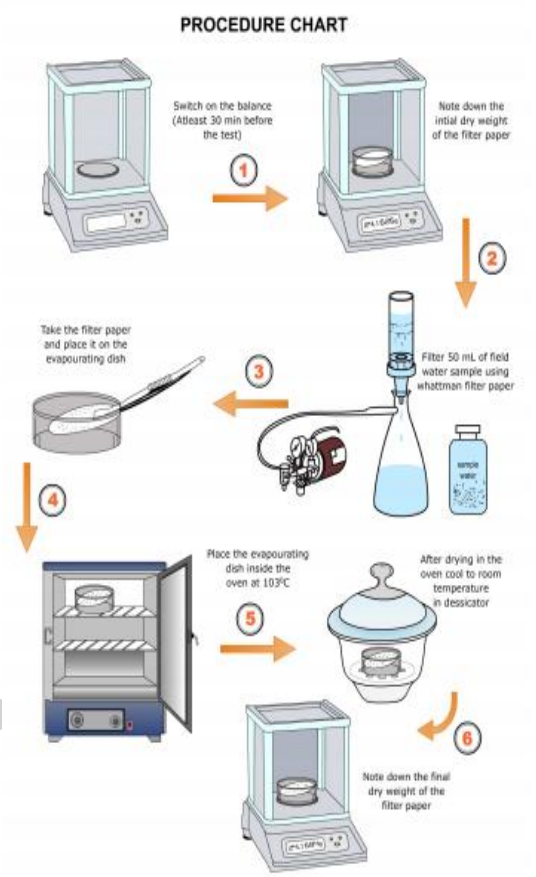
7. Place the filter paper that contains the suspended solids in a crucible and put it an oven maintained at 103˚ C for 1.0 hour, then remove it from the oven and place it in a desiccator to cool to room temperature.
8. After cooling the filter paper, weigh it. Find out the weight of suspended solids in the filter paper by subtracting the weight of the filter paper.

$$\text{Total Suspended Solids, TSS (mg/l)} = \frac{\text{mg of solids on filter paper} \times 1000}{\text{volume of sample, ml}}$$

Note that most of the time you will not get a precise measurement for the solids; since usually either TDS or TSS is the major part of the sample. In such cases calculation is more suitable.

TS (mg/l) = TSS (mg/l) + TDS (mg/l)





Issues to consider for your practical report

- What are the potential sources of error in this analytical determination? How could they be overcome?
- What are alternative methods for determining the TS, TSS and TDS of samples? How do they compare to this method?
- What are typical TS, TSS and TDS values of water and wastewater in Jordan? How do your data compare with these values?
- What are the main sources of TS, TSS and TDS in ground water, surface water and wastewater?
- What are the potential health and environmental effects if any of extreme TS, TSS and TDS values?
- Explain "ground water usually has higher TDS and surface water usually has higher suspended solids".
- Why the water is evaporated at 103˚C in assessment of solid of water.

Experiment (2): Solids in Water (A) Experimental Results	
Name	**Date**
ID No.	**Group**

Part (I) Total Solids (TS)						
ID	**Mass of Empty dish (g)**	**Volume of Sample (ml)**	**Mass of dish & solids after 105°C (g)**	**Mass of solids (mg)**	**Total Solids concentration (mg/l)**	**Notes**

Part (II) Total Suspended Solids (TSS)						
ID	**Mass of Empty dish+ filter paper (g)**	**Volume of Sample (ml)**	**Mass of dish & solids after 105°C (g)**	**Mass of solids (mg)**	**Total Suspended Solids concentration (mg/l)**	**Notes**

Part (III) Total Dissolved Solids (TDS)						
ID	**Mass of Empty dish (g)**	**Volume of *Filtrate* Sample (ml)**	**Mass of dish & solids after 105°C (g)**	**Mass of solids (mg)**	**Total Dissolved Solids concentration (mg/l)**	**Notes**

Observations & Calculations:

(3) Solids in Water (B)

Volatile solids (TVS) refer to materials that are completely volatilized from water at high temperature (550°C). These solids are often referred to the organic content of the water.

The term Total Fixed Solids (TFS) can be referred to materials which are not volatilized from water at high temperature (550°C). These solids are often referred to the inorganic content of the water.

Environmental significance:

The water which consists of high volatile solids is not suitable for drinking purpose and indicates that the water may have been polluted by domestic wastes or other organic wastes.

Volatile solids test is normally applied to sludge. It is indispensable in the design and operation of sludge digest. Vacuum filter and incineration plants.

Before the development of the COD test, it is used to find out the strength of industrial and domestic wastewater. It is helpful in assessing the amount biologically inert organic matter, such as lignin in case of wood pulping waste liquors.

The determination of volatile and fixed components in the residue is useful in the control of wastewater plant operation because it offers an approximate amount of organic matter present in the solid fraction of wastewater.

Measurement of TVS, TVDS and TVSS

The measurement of solids is by means of the gravimetric procedure. The principle of this experiment is that the sample is evaporated in a weighed dish in an oven and is dried to a constant mass at 103-105˚ C. The residue obtained is ignited to a constant weight at 550˚ C (the remaining solids represent the total fixed solids and the weight lost during the ignition represents the total volatile solids). The remaining forms, TDS and TSS require filtration of the sample. For liquid samples, all these solids levels are reported in mg/l.

Sample handling and preservation

Preservation of sample is not practical; because biological activity will continue after a sample has been taken, changes may occur during handling and storage. Both the characteristics and the amount of solids may change. To reduce these changes in sample taken for solids determination, keep all samples at 4. Do not allow sample to freeze. Analysis should begin as soon as possible.

Time required:

Three hour or less depending on the number of samples determined.

Equipment and Reagents:

Crucibles
Measuring cylinder
Filter paper No 42
Balance
Oven
Funnel
Tong

Significant Experimental Hazards

- Student should be aware of hazards associated with the use of all glassware (cuts) and during using the oven and furnace (burns)

Procedure:

I. Total Volatile Solids (TVS)

1. Take a clear dry crucible (which was kept at 103˚ C in an oven for 1 hour and ignited n an oven at 550˚ C also for an hour) of 100 ml and put an appropriate identification mark on it (*USE A PENCIL*).
2. Weight the crucible and note the weight. (W1)
3. Pour **25ml** of the thoroughly mixed sample, measured by the measuring cylinder, in the crucible.
4. Place the crucible in an oven maintained at 103˚ C for 2 hours, then remove it from the oven and place it in a desiccator to cool to room temperature.
5. After cooling the crucible, weigh it as soon as possible to avoid absorption of moisture. (W2)
6. Place the same crucible in the furnace at 550˚C and allow it to ignite for at least 20 min to 2 hr (until you get a constant weight).
7. Remove the crucible from the oven and place it in a desiccator to cool to room temperature.
8. After cooling the crucible, weigh it as soon as possible to avoid absorption of moisture. (W3)

Total Volatile Solids, TVS (mg/l) = TS (mg/l) – TFS (mg/l)

TFS (mg/l) = [W3-W1] mg / Volume of sample (l)

II. Total Volatile Dissolved Solids (TVDS)

1. Take a clear dry crucible (which was kept at 103˚ C in an oven for 1 hour and ignited n an oven at 550˚ C also for an hour) of 100 ml and put an appropriate identification mark on it (use a pencil).
2. Weight the crucible and note the weight. (W1)
3. Weigh a filter paper and write the weight.(F1)
4. Take a 25 ml of sample and filter it through a filter paper and collect the filtrate in a crucible.
5. Place the crucible in an oven maintained at 103˚ C for 2 hours, then remove it from the oven and place it in a desiccator to cool to room temperature.
6. After cooling the crucible, weigh it as soon as possible to avoid absorption of moisture. (W2)

7. Place the same crucible in the furnace at 550˚C and allow it to ignite for at least 20 min to 2 hr (until you get a constant weight).
8. Remove the crucible from the oven and place it in a desiccator to cool to room temperature.
9. After cooling the crucible, weigh it as soon as possible to avoid absorption of moisture. (W3)

Total Volatile Dissolved Solids, TVDS (mg/l) = TDS (mg/l)- TFDS mg

TFDS (mg/l) = [W3-W1] mg / Volume of sample (l)

III. Total Volatile Suspended Solids (TVSS)

1. Place the filter paper that contains the suspended solids in an oven maintained at 103˚ C for 2 hours, then remove it from the oven and place it in a desiccator to cool to room temperature.
2. After cooling the filter paper, weigh it. Find out the weight of suspended solids in the filter paper by subtracting the weight of the filter paper.(F2)
3. Place the filter paper in a weighed crucible and place it in an oven and ignite the sample at 550˚C and allow it to ignite for at least 20 min to 2 hr (until you get a constant weight).
4. Remove the crucible from the oven and place it in a desiccator to cool to room temperature.
5. After cooling the crucible, weigh it as soon as possible to avoid absorption of moisture and subtract the initial weight of the crucible to get the remaining solids weight. (F3)

Total Volatile Suspended Solids, TVSS (mg/l) = TSS (mg/l)- TFSS (mg/l)

TFSS (mg/l) = (F3-F1)mg /(volume of sample, l)

Note that most of the time you will not get a precise measurement for the solids; since usually either TVDS or TVSS is the major part of the sample. In such cases calculation is more suitable.

TVS (mg/l) = TVSS (mg/l) + TVDS (mg/l)

Issues to consider for your practical report

- What are the potential sources of error in this analytical determination? How could they be overcome?
- What are alternative methods for determining the TVS, TVSS and TVDS of samples? How do they compare to this method?
- What are typical TVS, TVSS and TVDS values of water and wastewater in Jordan? How do your data compare with these values?
- What are the main sources of TVS, TVSS and TVDS in ground water, surface water and wastewater?
- What are the potential health and environmental effects if any of extreme TVS, TVSS and TVDS values?
- Why the solids are ignited 550˚C in assessment of solid of water.

Experiment (3): Solids in Water (B)
Experimental Results

Name **Date**

ID No. **Group**

Part (I) Total Volatile Solids (TVS)							
ID	Mass of Empty dish (g)	Volume of Sample (ml)	Mass of dish & solids after 105°C (g)	Mass of dish & solids after 550°C (g)	TS (mg/l)	TFS (mg/l)	TVS (mg/l)

Part (II) Total Suspended Solids (TVSS)							
ID	Mass of Empty dish+ Ashless filter paper (g)	Volume of Sample (ml)	Mass of dish & solids after 105°C (g)	Mass of dish & solids after 550°C (g)	TSS (mg/l)	TFSS (mg/l)	TVSS (mg/l)

Part (III) Total Volatile Dissolved Solids (TDS)							
ID	Mass of Empty dish (g)	Volume of Filtrate Sample (ml)	Mass of dish & solids after 105°C (g)	Mass of dish & solids after 550°C (g)	TDS (mg/l)	TFDS (mg/l)	TVDS (mg/l)

Observations & Calculations:

(4) Sludge Volume Index

The sludge volume index (SVI) is the volume in milliliters occupied by 1 g of a suspension after 30 min settling. SVI typically is used to monitor settling characteristics of activated sludge and other biological suspensions. Although SVI is not supported theoretically, experience has shown it to be useful in routine process control.

Sludge Volume Index (SVI) is a very important indicator that determines your control or rate of desludging on how much sludge is to be returned to the basin and how much to take it out from the system. It actually serves as a very important empirical measurement that can be used as a guide to maintain sufficient concentration of activated sludge in the aeration basin whereby too much or too little can be considered detrimental to the system's overall health. Desludging or sometimes referred to as recycling sludge process, actually plays a very important role because the whole operation is needed to somehow strike a balance between removing dead or aged bacteria out of the systems or to determine how much goes back to the aeration pond.

Variations in temperature, sampling and agitation methods, dimensions of settling column, and time between sampling and start of determination can significantly affect the results.

The SVI indexes relate the weight of sludge to the volume that the sludge occupies and attempts to show how well the activated sludge separates from the mix liquor. Sludges with a low SVI have good settling and compaction characteristics.

Time required:

Three hour or less depending on the number of samples determined.

Significant Experimental Hazards

- Student should be aware of hazards associated with the use of all glassware (cuts) and during using the oven and furnace (burns).
  Student should be aware of hazards associated with the use of sludge samples.

Experiment Parts:

A. Determine the suspended solids concentration of a well-mixed sample of the suspension.

Same as mentioned in Experiment (2), for the measurement of TSS.

A well-mixed sample is filtered through a weighed standard glass-fiber filter and the residue retained on the filter is dried to a constant weight at 103 to 105°C. The increase in weight of the filter represents the total suspended solids. To obtain an estimate of total suspended solids, calculate the difference between total dissolved solids and total solids.

B. Determine the 30 min settled sludge volume.

The settled sludge volume of a biological suspension is useful in routine monitoring of biological processes. For activated sludge plant control, a 30-min settled sludge volume or the ratio of the 15-min to the 30-min settled sludge volume has been used to determine the returned-sludge flow rate and when to waste sludge.

This method is inappropriate for dilute sludges because of the small volume of settled material. In such cases, use the volumetric test for settleable solids using an Imhoff cone.

Results are not comparable with those obtained with the procedure herein.

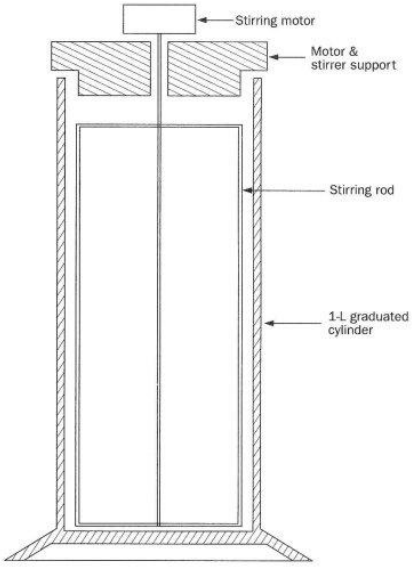


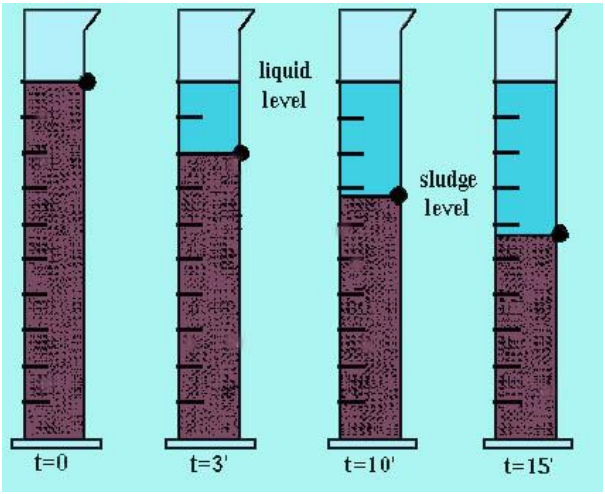
Figure 2710:1. Schematic diagram of settling vessel for settled sludge volume test.

Equipment

Graduated cylinder (equipped with a stirring mechanism)
Stopwatch.
Thermometer

Procedure

1) Place 1.0 L sample in settling column and distribute solids by covering the top and inverting cylinder three times.
2) Insert stirring rods, activate stirring mechanism, start the stop watch, and let suspension settle. Continue stirring throughout test.
3) Maintain suspension temperature during test at that in the basin from which the sample was taken.



4) Determine volume occupied by suspension at measured time intervals, e.g., 5, 10, 15, 20, 30, 45, and 60 min.
5) Report settled sludge volume of the suspension in milliliters for an indicated time interval.

C. Alternative Method (Settleable Solids)

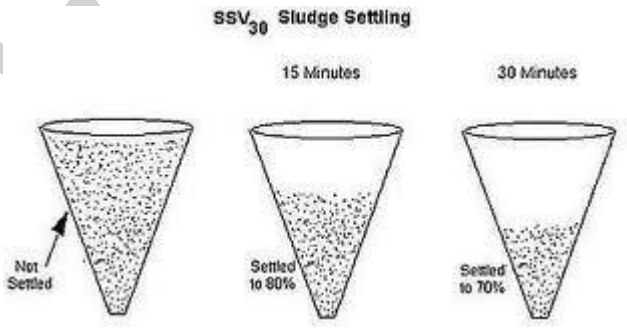
Settleable solids in surface and saline waters as well as domestic and industrial wastes may be determined and reported on either a volume (ml/l) or a weight (mg/l) basis.

Equipment:

The volumetric test requires only an Imhoff cone.

Procedure:

1) Fill an Imhoff cone to the 1-L mark with a well-mixed sample.
2) Settle for 45 min, gently agitate sample near the sides of the cone with a rod or by spinning.
3) Settle 15 min longer and record volume of settleable solids in the cone as milliliters per liter.
4) If the settled matter contains pockets of liquid between large settled particles, estimate volume of these and subtract from volume of settled solids.
5) Where a separation of settleable and floating materials occurs, do not estimate the floating material as settleable matter.
6) Replicates usually are not required.



Calculations

$$\text{SVI} = \frac{\text{Settled Sludge Volume (mL/L) X 1000}}{\text{Total Suspended Solids (mg/L)}}$$

Settleable Solids, mg/l = Total Suspended Solids, mg/l - Nonsettleable Solids, mg/l

Issues to consider for your practical report

- What are the potential sources of error in this analytical determination? How could they be overcome?
- What are alternative methods for determining the SVI , or any part of the tests? How do they compare to this method?
- What are typical SVI values of typical sludge? How do your data compare with these values?

Experiment (4): Sludge Volume Index
Experimental Results

Name **Date**

ID No. **Group**

Part (A) TSS

Part (I) Total Solids (TS)

ID	Mass of Empty dish (g)	Volume of Sample (ml)	Mass of dish & solids after 105°C (g)	Mass of solids (mg)	Total Solids concentration (mg/l)	Notes

Part (II) Total Suspended Solids (TSS)

ID	Mass of Empty dish+ filter paper (g)	Volume of Sample (ml)	Mass of dish & solids after 105°C (g)	Mass of solids (mg)	Total Suspended Solids concentration (mg/l)	Notes

Part (III) Total Dissolved Solids (TDS)

ID	Mass of Empty dish (g)	Volume of *Filtrate* Sample (ml)	Mass of dish & solids after 105°C (g)	Mass of solids (mg)	Total Dissolved Solids concentration (mg/l)	Notes

Part (B) the 30 min settled sludge volume

Time (min)	Volume occupied by suspension (ml)
0	
5	
10	
20	
30	

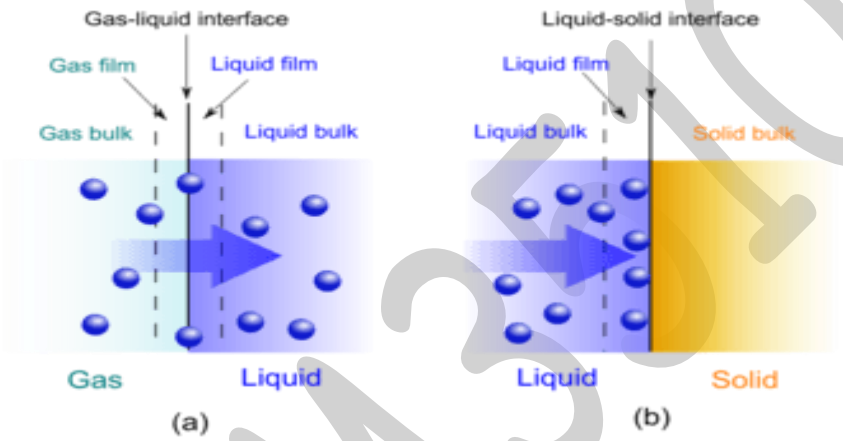
A. Part (C) Alternative Method (Settleable Solids)

Time (min)	Volume of settleable solids in the cone (ml/l)
0	
15	
30	
45	
60	

Observations & Calculations:

(5) Adsorption

Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic film (the adsorbate). It is different from absorption, in which a substance diffuses into a liquid or solid to form a solution. The term sorption encompasses both processes, while desorption is the reverse process.



Adsorption is operative in most natural physical, biological, and chemical systems, and is widely used in industrial applications such as activated charcoal, synthetic resins and water purification.

Similar to surface tension, adsorption is a consequence of surface energy. In a bulk material, all the bonding requirements (be they ionic, covalent or metallic) of the constituent atoms of the material are filled. But atoms on the (clean) surface experience a bond deficiency, because they are not wholly surrounded by other atoms. Thus it is energetically favourable for them to bond with whatever happens to be available. The exact nature of the bonding depends on the details of the species involved, but the adsorbed material is generally classified as exhibiting physisorption or chemisorption.

Physisorption or physical adsorption is a type of adsorption in which the adsorbate adheres to the surface only through Van der Waals (weak intermolecular) interactions, which are also responsible for the non-ideal behaviour of real gases.

Chemisorption is a type of adsorption whereby a molecule adheres to a surface through the formation of a chemical bond, as opposed to the Van der Waals forces which cause physisorption.

Adsorption is usually described through isotherms, that is, functions which connect the amount of adsorbate on the adsorbent, with its pressure (if gas) or concentration (if liquid).

One can find in literature several models describing process of adsorption, namely Freundlich isotherm, Langmuir isotherm, BET isotherm, etc. We will deal with Langmuir isotherm in more details:

Langmuir isotherm

In 1916, Irving Langmuir published an isotherm for gases adsorbed on solids, which retained his name. It is an empirical isotherm derived from a proposed kinetic mechanism.

It is based on four hypotheses:

1. The surface of the adsorbent is uniform, that is, all the adsorption sites are equal.

2. Adsorbed molecules do not interact.

3. All adsorption occurs through the same mechanism.

4. At the maximum adsorption, only a monolayer is formed: molecules of adsorbate do not deposit on other, already adsorbed, molecules of adsorbate, only on the free surface of the adsorbent.

For liquids (adsorbate) adsorbed on solids (adsorbent), the Langmuir isotherm (Fig. 1) can be expressed by

$$m = \frac{A_{max} k.C}{1 + kC}$$

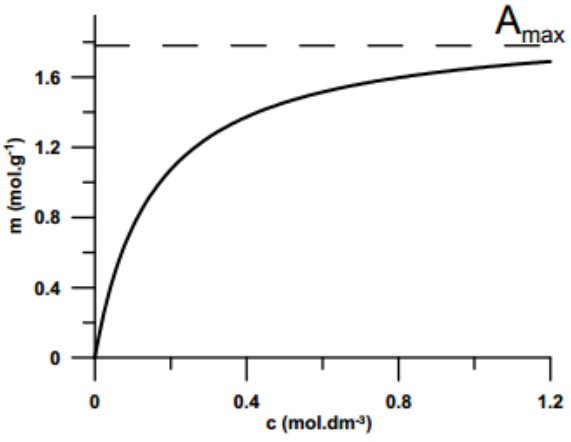
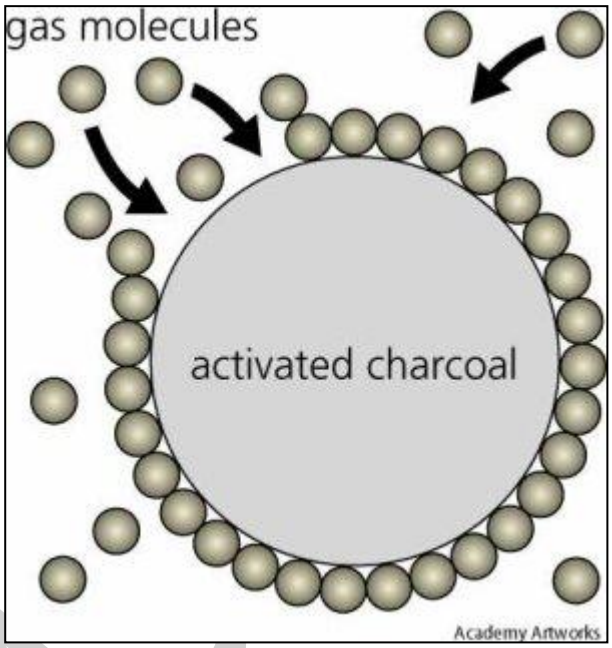


Fig. 1. Langmuir isotherm

where m is the substance amount of adsorbate adsorbed per gram (or kg) of the adsorbent, the unit of m is $mol.g^{-1}$, resp. $mol.kg^{-1}$. A_{max} is the maximal substance amount of adsorbate per gram (or kg) of the adsorbent. The unit of A_{max} is $mol.g^{-1}$, resp. $mol.kg^{-1}$. k is the adsorption constant ($mol^{-1}.dm^3$); C ($mol.dm^{-3}$) is the concentration of adsorbate in liquid.

In practice, activated carbon is used as an adsorbent for the adsorption of mainly organic compounds along with some larger molecular weight inorganic compounds such as iodine and mercury.

Activated carbon, also called activated charcoal or activated coal, is a general term that includes carbon material mostly derived from charcoal. For all three variations of the name, "activated" is sometimes substituted by "active." By any name, it is a material with an exceptionally high surface area. Just one gram of activated carbon has a surface area of approximately 500 m^2 (for comparison, a tennis court is about 260 m^2). The three main physical carbon types are granular, powder and extruded (pellet). All three types of activated carbon can have properties tailored to the application. Activated carbon is frequently used in everyday life, in: industry, food production, medicine, pharmacy, military, etc. In pharmacy, activated charcoal is considered to be the most effective single agent available as an emergency decontaminant in the gastrointestinal tract. It is used after a person swallows or absorbs almost any toxic drug or chemical.



Time required:

Two hour or less depending on the number of samples determined.

Equipment and Reagents:

1. 6 Boiling Flasks (250 ml)
2. 6 Erlenmayer's Flasks (250 ml)
3. 6 Funnels
4. 3 Burettes (50ml)
5. 10 Conical Flasks
6. 3 Pipettes
7. Holders
8. Filtering Paper
9. Rubber Stoppers
10. Acetic Acid Solution Of (1.0 M)
11. NaOH Solution Of (0.2 M)
12. Activated Charcoal
13. Phenolphthalein.

Significant Experimental Hazards

- Student should be aware of hazards associated with the use of all glassware (cuts).
- Phenolphthalein indicator maybe harmful if ingested in quantity and may irritate eyes and skin.
- Sodium hydroxide is very hazardous in case of skin contact (corrosive, irritant), of eye contact (irritant, corrosive), of ingestion, of inhalation. The amount of tissue damage depends on length of contact. Eye contact can result in corneal damage or blindness. Skin contact can produce inflammation and blistering.
- Acetic acid is very hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. Hazardous in case of skin contact (corrosive, permeator), of eye contact (corrosive). Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns.

Procedure

1. Prepare aqueous solutions of acetic acid into numbered flasks following the scheme given in the table 1. The total volume of each solution is 60 ml. Use flasks fitted with stoppers.

Flask No.	1	2	3	4	5	6
Acetic Acid, ml	**6**	**12**	**18**	**30**	**42**	**60**
Distilled water, ml	**52**	**48**	**42**	**30**	**18**	**0**
Total volume, ml	**60**	**60**	**60**	**60**	**60**	**60**

2. Transfer 10 ml of the solution from each flask into numbered conical flask, so final volume of acetic acid solution is V_A=50 ml per flask.

3. Determine the actual concentration of acetic acid in flasks by titration in this way:

4. For titration, modify the volume in each conical flask. Take away defined volume of the solution, to obtain in each flask the volume as given in the table Tab.2

Flask No.	1	2	3	4	5	6
Volume V, ml	**10**	**10**	**5**	**5**	**5**	**2**

5. Add 2-3 drops of phenolphthalein and titrate by NaOH.

6. Once the endpoint has been reached, read the burette. The volume of the base V_B (ml) that was required to reach the endpoint write down in the data sheet.

7. Calculate the actual concentration of acetic acid C_i in the flask No. 1 – 6, respectively.

8. Using practical balance and glazed paper, weigh 6 portions of activated charcoal, each portion 5 g. The accuracy of weighing must be 0.01 g.

9. Put activated charcoal into numbered flasks with stoppers (1 portion per flask).

10. Plug up the flasks, and shake them. Wait for 20 minutes, the process of adsorption is in progress. Mix the mixtures for several times by flasks shaking within this period.

11. Filter the mixtures into clean and dry flasks. To avoid disturbing effect of adsorption of acetic acid into filtering paper, remove away the first portion of filtration, app. 5 ml.

12. Determine the final concentration of acetic acid C_f in each of the flasks after adsorption: From each solution, transfer the asked volume into clean and dry conical flask, again following Tab. 2

1. (Remark: The process of adsorption is a function of time too. It is important to put charcoal into flasks at the same time, to provide adsorption for the same period in each flask).

13. Repeat points 5-7, and from the consumed base V_B (ml) ,determine the concentration of acetic acid C_f after adsorption.

Calculations

A. Determination of the concentration of acetic acid before (C_i) and after (C_f) adsorption

$$C_f = \frac{V_B C_B}{V_A}$$

Where V_B the volume of the titrant, NaOH, is C_B is the Concentration of the titrant, V_A is the volume of the analyte (Acetic Acid). Calculate the concentration of acetic acid after adsorption (C_f).

B. Determination of the substance amount of acetic acid adsorbed per gram of the charcoal m (mol.g^{-1}) in individual flask:

$$m_i = \frac{(C_i - C_f)V_A}{g}$$

where C_i , C_f are the concentrations of acetic acid before and after adsorption, respectively. V_A is the volume of the liquid phase in the mixture charcoal – acetic acid, g is the mass of the adsorbent – charcoal (in grams), i=1-6 is the number of flask. Eq. 3 supposes that V_A is the same for i=1-6, and also the mass of the charcoal (g). Write down the obtained values of m_i.

C. Determination of k and A_{max}: The Eq. 1 one can rearrange into a form:

$$\frac{1}{m} = \frac{1}{A_{max}}\frac{1}{kC} + \frac{1}{A_{max}}$$

$\frac{1}{m} = f(\frac{1}{C})$ = should be a straight line

where C is the concentration of acetic acid after adsorption. Fit the experimental points with a linear function. The slope represents the value of $1/A_{max}k$, and the intercept corresponds to max $1/A_{max}$.

D. Calculate A_{max} and k from the slope and the intercept.

Issues to consider for your practical report

- What are the potential sources of error in this experiment? How could they be overcome?
- Are there alternative methods for determining the adsorption isotherm? If so, how do they compare to this method?
- How charcoal is activated?
- What are the uses of adsorption in water and wastewater unit operation.

Experiment (5): Adsorption
Experimental Results

Name **Date**

ID No. **Group**

Flask No.	Acetic Acid, ml	Volume V, ml	Initial concentration					Final Concentration				
			Burette Reading		NaOH concentration M	Volume of NaOH (ml)	Observation (color change)	Burette Reading		NaOH concentration M	Volume of NaOH (ml)
			Initial	Final				Initial	Final		
1	6	10									
2	12	10									
3	18	5									
4	30	5									
5	42	5									
6	60	2									

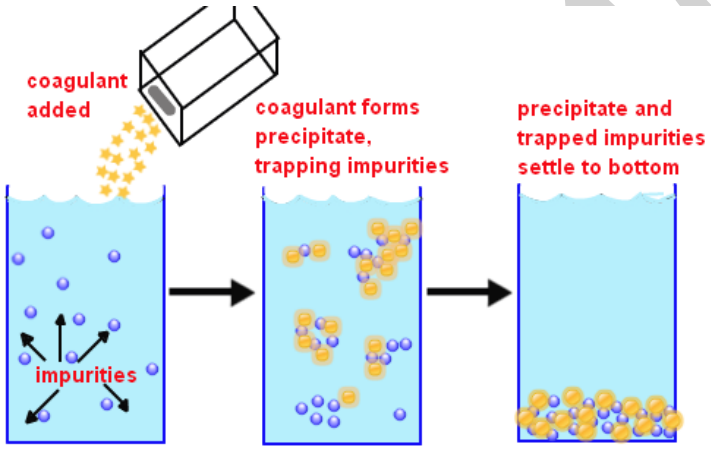
Flask No.	V_{Bi}, ml	C_i ,mol/l	V_{Bf}, ml	C_f, mol/l	m_i, mmol/g	1/ C_i, l/mol	1/ m_i, g/mmol
1							
2							
3							
4							
5							
6							

Observations & Calculations:

(6) Jar Test

The jar test is a method of measuring the effect of coagulation, flocculation, and sedimentation on turbidity. Although the procedure is not outlined in Standard Methods, it is used in most water treatment plants to find the best coagulant dosages under varying conditions.

Coagulation/flocculation is the process of binding small particles in the water together into larger, heavier clumps which settle out relatively quickly. The larger particles are known as floc. Properly formed floc will settle out of water quickly in the sedimentation basin, removing the majority of the water's turbidity.



In many plants, changing water characteristics require the operator to adjust coagulant dosages at intervals to achieve optimal coagulation. Different dosages of coagulants are tested using a jar test, which mimics the conditions found in the treatment plant. The first step of the jar test involves adding coagulant to the source water and mixing the water rapidly (as it would be mixed in the flash mix chamber) to completely dissolves the coagulant in the water. Then the water is mixed more slowly for a longer time period, mimicking the flocculation basin conditions and allowing the forming floc particles to cluster together. Finally, the mixer is stopped and the floc is allowed to settle out, as it would in the sedimentation basin.

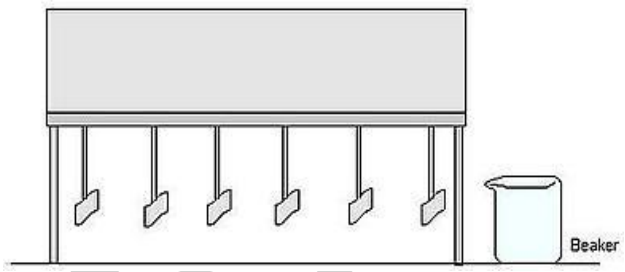
The type of source water will have a large impact on how often jar tests are performed. Plants which treat groundwater may have very little turbidity to remove are unlikely to be affected by weather-related changes in water conditions. As a result, groundwater plants may perform jar tests seldom, if at all, although they can have problems with removing the more difficult small suspended particles typically found in groundwater. Surface water plants, in contrast, tend to treat water with a high turbidity which is susceptible to sudden

changes in water quality. Operators at these plants will perform jar tests frequently, especially after rains, to adjust the coagulant dosage and deal with the changing source water turbidity.

Time required:

Two hour or less depending on the number of samples determined.

Equipment and Reagents



- Volumetric flask (1,000 ml)
- Analytical balance
- Magnetic stirrer (optional)
- Beakers (1,000 ml)
- Pipets (10 ml)
- Watch or clock

A. Turbidometer and sample tubes
B. A stirring machine with six paddles capable of variable speeds from 0 to 100 revolutions per minute (RPM)
C. Coagulants and coagulant aids

Significant Experimental Hazards

D. Student should be aware of hazards associated with the use of all glassware (cuts).

Procedure

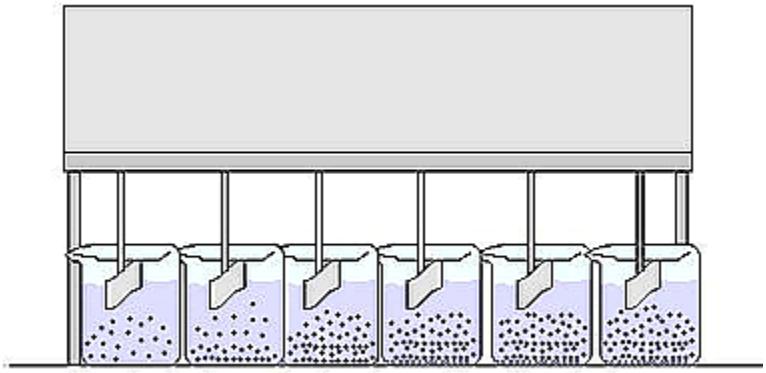
1) Decide on six dosages of the chemical(s).
2) If pre-lime has to be fed, it is usually best to hold the amount of lime constant and vary the coagulant dosage.
3) You will need to prepare a stock solution for each type of chemical used. The strength of the stock solution will depend on the chemical dosages which you decided to use in step 1. The table below shows what strength stock solution you should prepare in each circumstance.

Approximate dosage required, mg/L	Stock solution concentration, mg/L	1 ml added to 1 L sample equals
1-10 mg/L	1,000 mg/L	1 mg/L
10-50 mg/L	10,000 mg/L	10 mg/L
50-500 mg/L	100,000 mg/L	100 mg/L

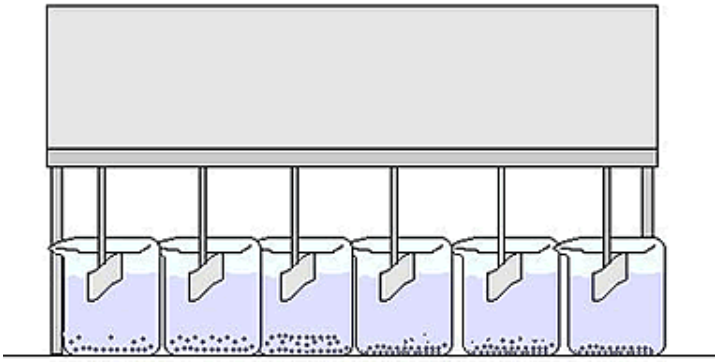
For example, if all of your dosages are between 1 and 10 mg/L, then you should prepare a stock solution with a concentration of 1,000 mg/L. This means that you could prepare the stock solution by

dissolving 1,000 mg of the chemical in 1 L of distilled water. However, this would produce a much larger quantity of stock solution than you need and would waste chemicals. You will probably choose instead to dissolve 250 mg of the chemical in 250 ml of distilled water.

4) Weigh out the proper quantity of the chemical using the analytical balance.
5) Measure out the proper quantity of distilled water in the volumetric flask.
6) Add the chemical to the distilled water. Mix well.
7) If lime is used, it is best to use a magnetic stirrer since lime is not completely soluble in water.
8) Measure 1,000 mL of raw water and place in a beaker. Repeat for the remaining beakers.
9) Place beakers in the stirring machine.
10) With a measuring pipet, add the correct dosage of lime and then of coagulant solution to each beaker as rapidly as possible.
11) The third column of the table in step 2 shows the amount of stock solution to add to your beaker.
12) With the stirring paddles lowered into the beakers, start the stirring machine and operate it for one minute at a speed of 80 RPM. While the stirrer operates, record the appearance of the water in each beaker. Note the presence or absence of floc, the cloudy or clear appearance of water, and the color of the water and floc.



13) Reduce the stirring speed to 20 RPM and continue stirring for 30 minutes. Record a description of the floc in each beaker 5, 10, 15, 20, 25, and 30 minutes after addition of the chemicals.
14) Stop the stirring apparatus and allow the samples in the beakers to settle for 30 minutes. Record a description of the floc in each beaker after 15 minutes of settling and again after 30 minutes of settling.



15) Determine which coagulant dosage has the best flocculation time and the most floc settled out. This is the optimal coagulant dosage.

    A hazy sample indicates poor coagulation. Properly coagulated water contains floc particles that are well-formed and dense, with the liquid between the particles clear.

16) Test the turbidity of the water in each beaker using a turbidometer.

    Pipet water out of the top of the first beaker and place it in a sample tube, making sure that no air bubbles are present in the sample. (Air bubbles will rinse while turbidity will sink.) Carefully wipe the outside of the sample tube clean. Place the sample tube in a calibrated turbidometer and read the turbidity. Repeat for the water from the other beakers.

    The least turbid sample should correspond to the optimal coagulant dosage chosen in step 10.

17) If lime or a coagulant aid is fed in addition to the primary coagulant, you should repeat the jar test to determine the optimum dosage of lime or coagulant aid. Use the concentration of coagulant chosen in steps 10 and 11 and alter the dosage of lime or coagulant aid.

Issues to consider for your practical report

- What are the potential sources of error in this experiment? How could they be overcome?
- Are there alternative methods for determining the optimum dosage of coagulant? If so, how do they compare to this method?
- What are typical water turbidity values in sea, rivers, streams, lakes and drinking water? How do your data compare with these values?
- What are the potential human health and environmental effects (if any) of excess coagulant dosage?
- Based on the type of coagulant used, write the chemical equations that occurred during the experiment.

Experiment (6): Jar Test
Experimental Results

Name **Date**

ID No. **Group**

Coagulant Type:
Variable dosing coagulant at constant pH
Temperature:

Beaker ID	Initial Water Quality			Final Water Quality			
	Coagulant conc. mg/l	Turbidity (NTU)	pH	Coagulant (mg/l)	Turbidity (NTU)	pH	Observation

Coagulant Type:
Constant dosing at variable pH
Temperature:

Beaker ID	Initial Water Quality			Final Water Quality			
	Coagulant conc. mg/l	Turbidity (NTU)	pH	Coagulant (mg/l)	Turbidity (NTU)	pH	Observation

Observations & Calculations: