التحاليل المعملية لمياه الصرف الصحى

أهمية التحاليل المعملية

- معرفة نوعية وطبيعة المياه الداخله
- معرفة مدى مطابقة المياه الخارجه (السيب النهائى) للمواصفات
- معرفة كفاءة المعالجة
- التحكم فى عملية التشغيل

مكونات مياه الصرف الصحى

- مياه الصرف الصحى عبارة عن ٩٩.٩% مياه و٠.١% مواد صلبه
- تنقسم المواد الصلبه الى مواد ذائبة ومواد عالقه
- كل من المواد الصلبةالذائبه و العالقة ينقسم الى عضوى وغير عضوى
- محطة معالجة الصرف الصحى دورها الاهم هو التخلص من الملوثات المواد العضوية الغير ثابته والقابلة للتحلل وتحويلها الى مواد غير عضوية ثابته غير قابلة للتحلل

العينة وأنواعها

- جزء مستقطع من المياه بحيث يمثل الحجم المأخوذ التصرفات الوارد وطبيعة المياه ويكون كافى لعمل التحاليل المطلوبة
- شروط أخذ العينات

١-ان تكون العينة ممثلة تمثيلا جيدا للمخلفات السائلة

٢- استخدام طريقة سليمه لأخذ العينات

٣- حفظ العينات حتى وقت التحاليل

أنواع العينات

- **لحظية**

تؤخذ مرة واحده من مكان اخذ العينة فى اى وقت لعمل الاختبارات اللازمة

- **مركبة**

عينات تؤخذ بانتظام على مدار اليوم من مراحل المعالجة المختلفة لتمثل نوعية المياه على مدار اليوم ويفضل هذا النوع من العينات فى تجارب التشغيل مثل COD&BOD&TSS

والتى تعطى تمثيلا حقيقيا عن الاحمال العضوية الحقيقية الداخلة والخارجه من المحطة

جدول حفظ العينات

التجربة	نوع الاناء	طريقة الحفظ	اقصى مدة حفظ
الرقم الهيدروجينى	زجاج او بلاستيك	فورا	٢ساعه
الاكسجين الذائب	زجاج	فورافى الموقع	
درجة الحرارة	زجاج	فورا فى الموقع	
القلوية الكلية	زجاج اوبلاستيك	الثلاجه	٢٤ ساعه
المواد الصلبة	زجاج او بلاستيك	الثلاجة	٧ ايام
الاكسجين الحيوى الممتص	زجاج او بلاستيك	الثلاجة	٤٨ ساعه
الاكسجين الكيميائى المستهلك	زجاج او بلاستيك	حمض الكبريتيك PH<2	٧ ايام
الكبريتيدات	زجاج او بلاستيك	الثلاجة وضافة اسيتات الزنك	٧ ايام

الزيوت والشحوم	زجاج	الثلاجهواضافة حمض PH< 2	٢٨ يوم
الكلور المتبقى	زجاج او بلاستيك	يقاس فورا بالموقع	
الامونيا	زجاج او بلاستيك	تحلل فورااو التبريد واضافة حمض الكبريتيك PH<2	٧ ايام
النترات	زجاج او بلاستيك	تحلل فورا او البتريد فى الثلاجة	٢٤ ساعه
النيروجين العضوى	زجاج او بلاستيك	الثلاجة واضافة حمض الكبريتيك PH < 2	٧ ايام
الفوسفور	زجاج	الثلاجة	٤٨ ساعه
المعادن الثقيله	زجاج او بلاستيك	الثلاجة مع اضافة حمض النيتريك PH<2	٦ اشهر

خصائص مياه الصرف الصحى

فيزيائية	كيميائية	بيولوجية
– اللون – الرائحة – درجة الحرارة – المواد الصلبة الكلية سواء كانت ذائبة اوعالقه	– مواد عضوية – غير عضوية – الرقم الهيدروجينى PH – الكلوريدات – القلوية – النيتروجين – الفوسفور – الكبريت – المعادن الثقيله – المواد السامه	– البكتيريا (هوائية – لاهوائية – اختيارية) – البروتوزوا (الأوليات)

التحاليل المعملية فى محطات المعالجة

مدخل (مياه خام)	احواض معالجة	مخرج (السيب النهائى)
– درجة الحرارة – الاكسجين الذائب DO – الرقم الهيدروجينى PH – المواد الصلبة العالقة الكليةTSS – المواد الصلبة الذائبة TDS – القلوية – الكلوريدات – الأمونيا – النتبرات – الكبريتيدات – الزيوت والشحوم – BOD – COD	– MLSS – MLVSS – SV30 – SVI – F/M – S.age – Microscopic exam.	– درجة الحرارة – الاكسجين الذائب DO – الرقم الهيدروجينى PH – المواد الصلبة العالقة الكليةTSS – المواد الصلبة الذائبة TDS – القلوية – الكلوريدات – الأمونيا – النترات – الكبريتيدات – الزيوت والشحوم – BOD – COD – الكلور المتبقى R.cl2

Terminology

- TS : Total solids
- TDS : total dissolved solids
- TSS : total suspended solids
- VSS : volatile suspended solids
- FSS : fixed suspended solids
- BOD : biological oxygen demand
- COD : chemical oxygen demand
- MLSS : mixed liquor suspended solids
- MLVSS : mixed liquor volatile suspended solids

Terminology

- SVI : sludge volume index
- SV30 : settled sludge volume via 30 min.
- DO : dissolved oxygen
- F/M : food/ microorganisms
- SA : sludge age
- AS : activated sludge
- RAS : Return activated sludge
- WAS : waste activated sludge

يمكن تقسيم التحاليل المعملية

- تحاليل فيزيائية
- تحاليل كيميائية
- تحاليل ميكروسكوبية
- تحاليل ميكروبيولوجية

١- تحاليل فيزيائية

وتشتمل على

- درجة الحرارة
- اللون
- الرائحة
- قياس الأس الهيدروجينى
- قياس TS
- قياسTSS
- قياس TDS

٢- التحاليل الكيميائية

وتشتمل على

- قياس DO
- قياس BOD
- قياس COD
- قياس الكلور المتبقى R.cl2
- الكبريتيدات
- الامونيا
- النترات
- الكلوريدات

قياس الاس الهيدروجينى

قياس درجة الحرارة

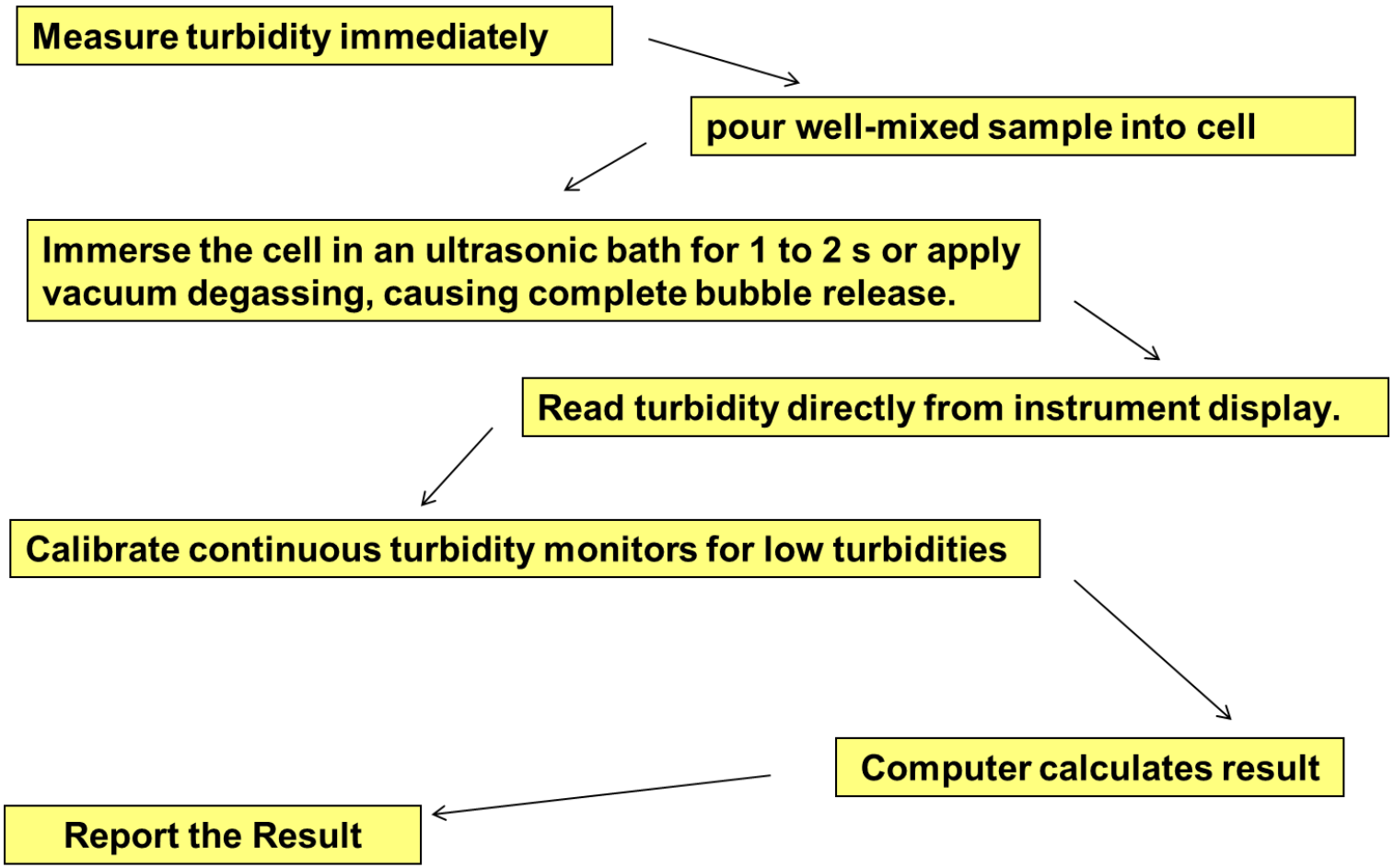
Thermometer Types

Beaker

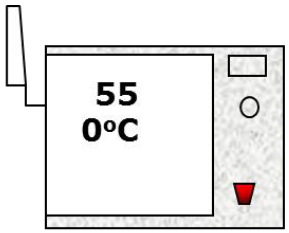
قياس العكاره

Nephelometer & Cells

Flow chart


Measure turbidity immediately
pour well-mixed sample into cell
Immerse the cell in an ultrasonic bath for 1 to 2 s or apply vacuum degassing, causing complete bubble release.
Read turbidity directly from instrument display.
Calibrate continuous turbidity monitors for low turbidities
Computer calculates result
Report the Result

قياس TS



1. Ignite empty evap. dish in muffle furnace

2. Cool in • desiccator

3. Weigh empty • dish – W1

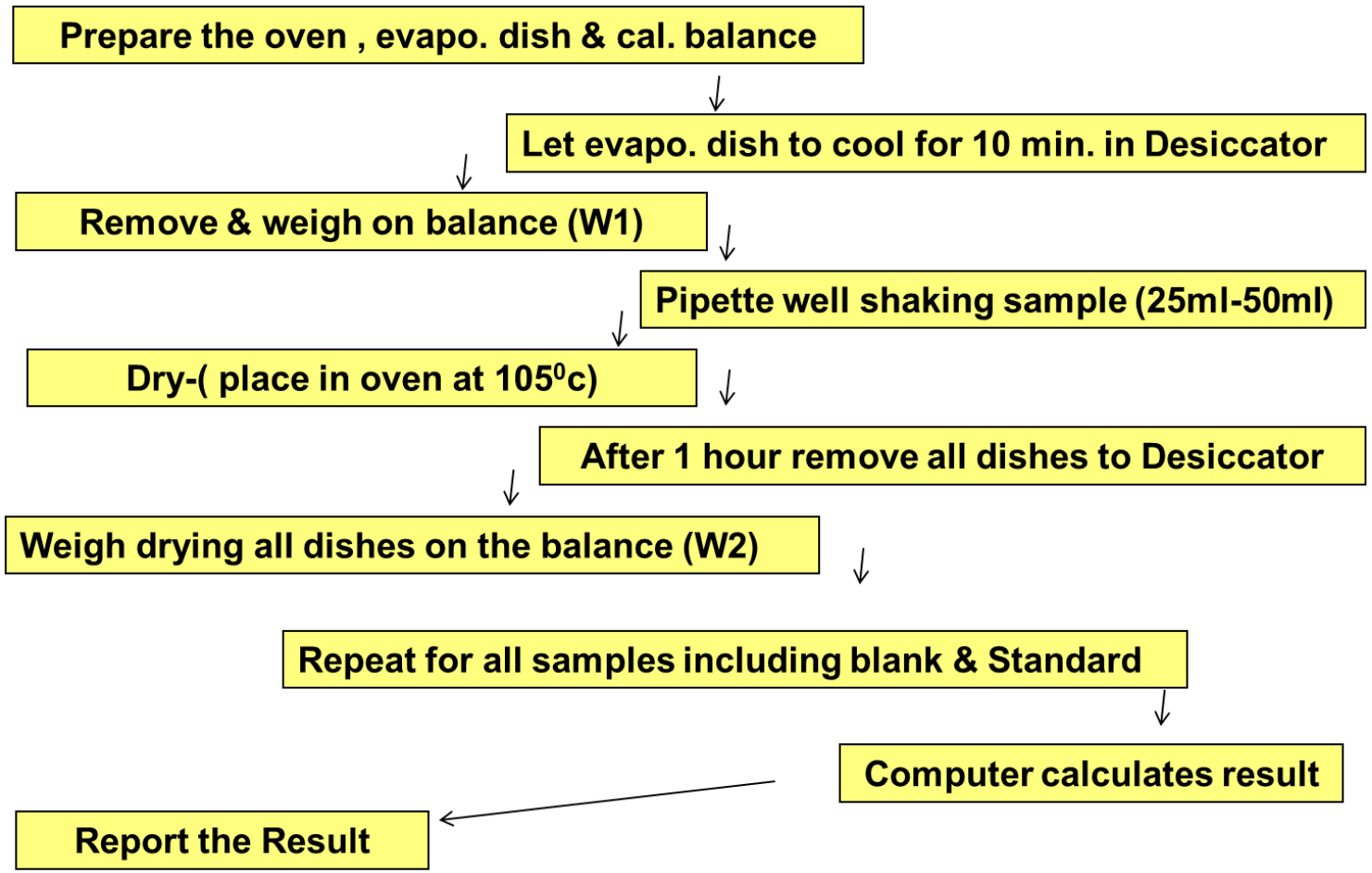
4. place a 25 to 50 ml sample aliquot on a prepared evaporating dish and evaporate on water bath.

5-Dry the samples .at 103°C to 105°C

6. Cool in desiccator

7. Weigh dish and dry residue – W2

TS flow chart

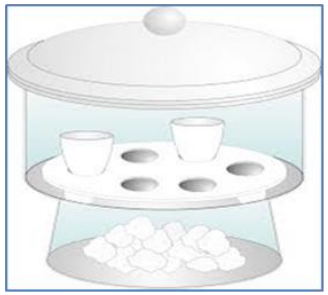


TVS &TFS

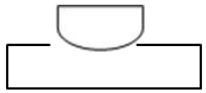
Drying oven

Electronic Balance

Muffle Furnace

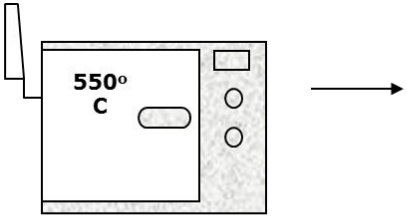


Desiccator



Evap. dish
&
water bath.

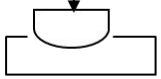
OUTLINE OF PROCEDURE FOR FIXED AND VOLATILE SOLIDS



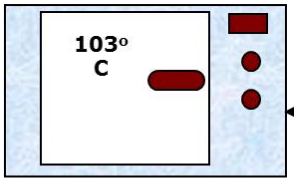
1. Ignite empty dish in muffle furnace

2. Cool in desiccator

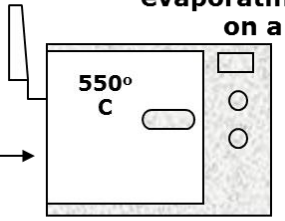
3. Weigh empty dish



Place a 25 to 50 ml sample aliquot on a prepared evaporating dish. Evaporate on a water bath.



Dry the samples at 103°C to 105°C for 1 to 2 hours.



8. Ignite dried residues in muffle furnace

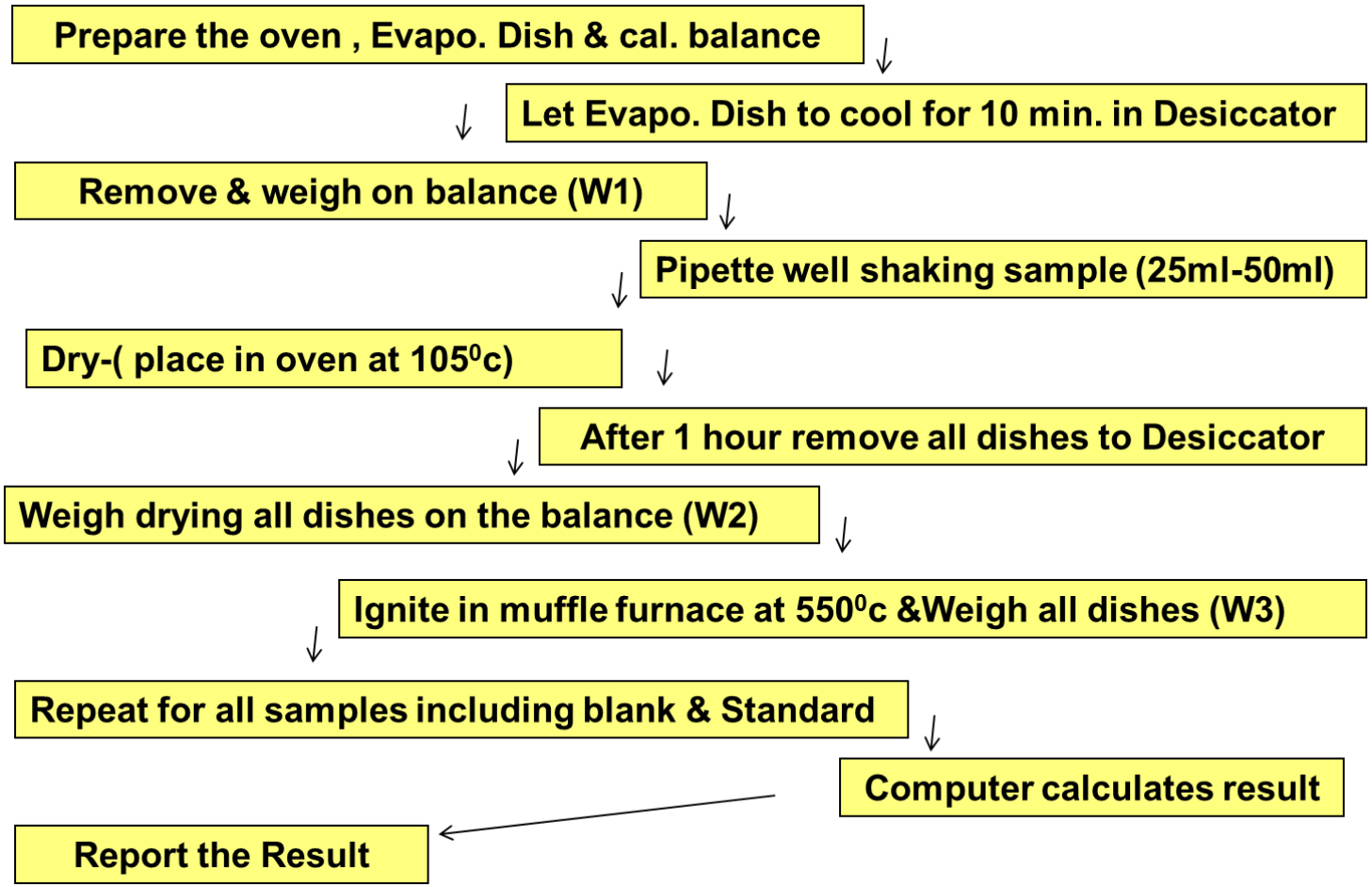
9. Cool in desiccator

10. Weigh dish and ash.

6. Cool in desiccator

7. Weigh dish and dry residue

TVS Flow Chart



Calculations

-Calculate the Volatile solids mg/l as follow:

$$\text{Total Volatile Solids mg/l} = \frac{(\text{W total} - \text{W fixed}) \times 1000{,}000}{\text{ml of sample}}$$

W total = weight of dried residue and dish (grams)

W fixed = weight of Ash and dish (grams)

Total Dissolved Solids (TDS)

Vol. Flask

Magnetic Stirrer

Magnetic Bars

Conductivity Meter

Total Dissolved Solids (TDS)

Drying oven

Electronic Balance

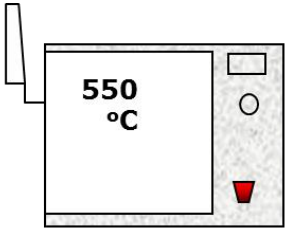
Steam both

Filtration System

Desiccator

Evaporating dish and water bath.

OUTLINE OF PROCEDURE FOR TDS



1. Ignite empty evap. dish in muffle furnace

2. Cool in • desiccator

3. Weigh empty dish – • W1

4. Filter, take • the filtration

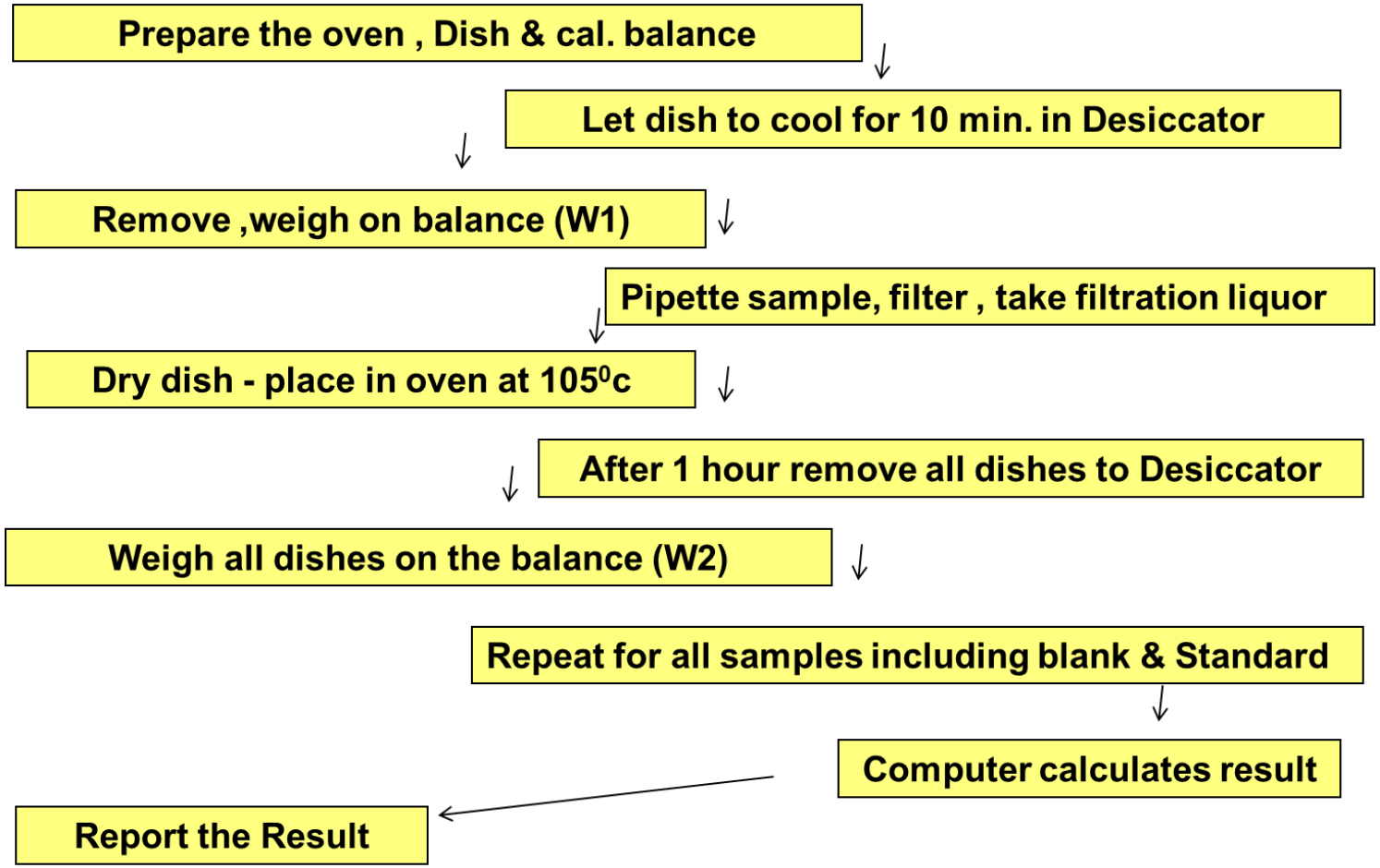
4. place a 25 to 50 ml sample aliquot on a prepared evaporating dish and evaporate on water bath.

5-Dry the samples at .103°C to 105°C

6. Cool in desiccator

7. Weigh dish and dry residue – W2

TDS Flow Chart



Total Suspended Solids (TSS)

Drying oven

Electronic Balance

Muffle Furnace

Filtration Unit

Desiccator

OUTLINE OF PROCEDURE FOR TSS

1-Dry filter paper at .103°C to 105°C

2. Cool in desiccator•

3. Weigh empty paper(w1)•

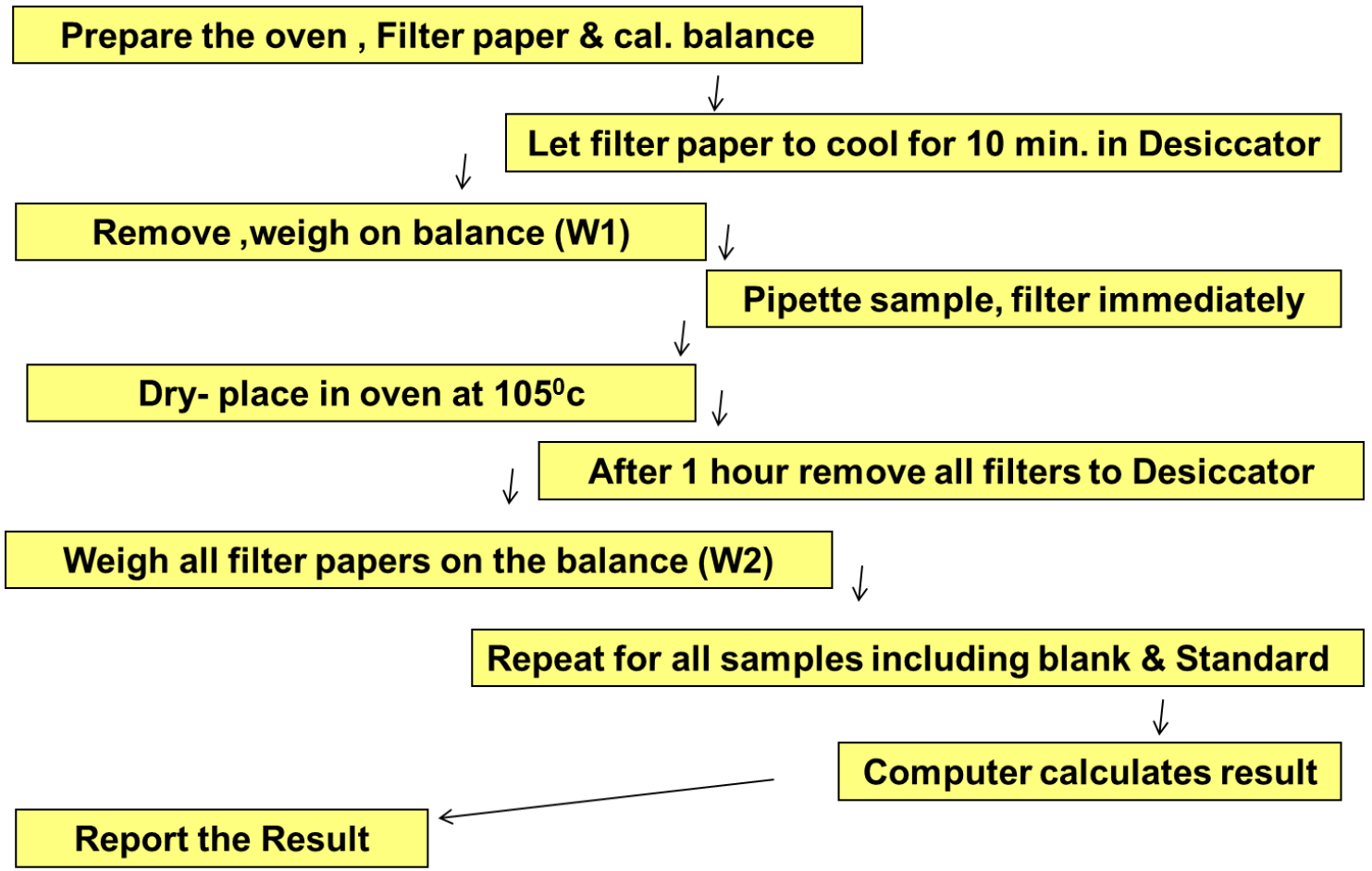
4. Filter the sample

5-Dry the samples .at 103°C to 105°C

6. Cool in d

7. Weigh after drying (w2)

TSS Flow Chart



Settleable Solids (SS)

Drying oven

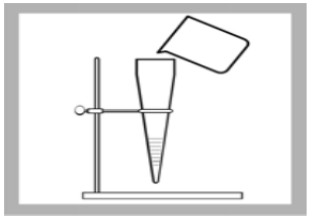
Desiccator

Elect. Balance

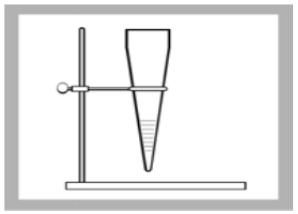
Filtration System

Imhoff cones

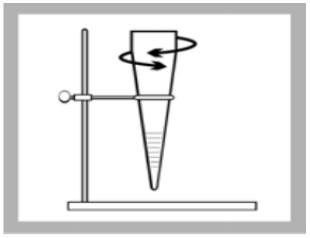
OUTLINE OF PROCEDURE FOR SS MATTER



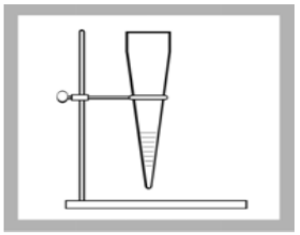
1. Fill an Imhoff cone to the 1L with a mixed sample



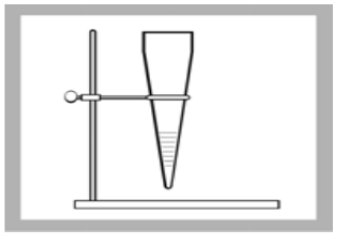
2. Wait 45 minutes for the undisturbed sample to settle.



3. Spin the cone forward and backward several times to dislodge materials on the inclined side of the cone.

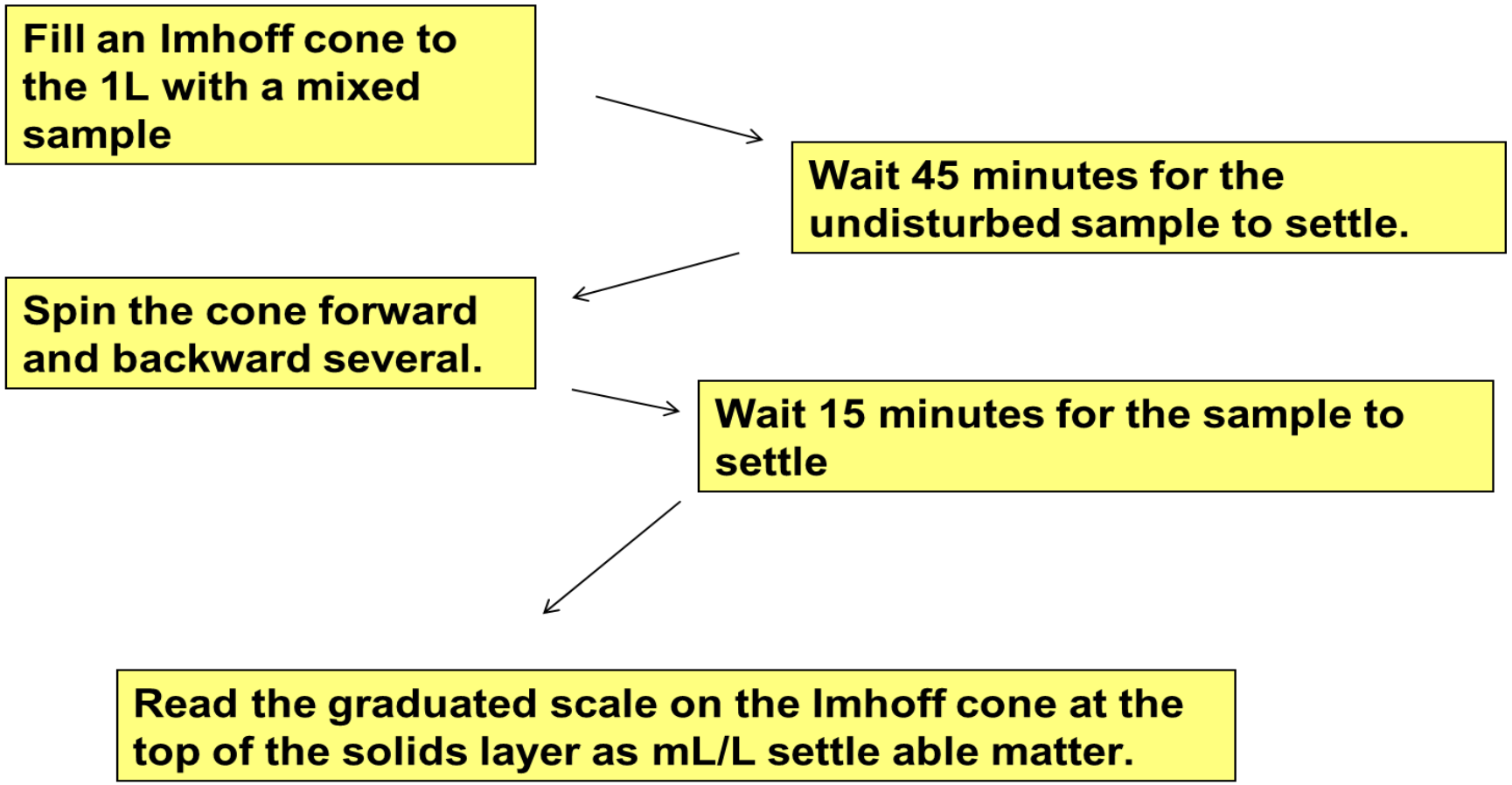


4. Wait 15 minutes for the sample to settle



5. Read the graduated scale on the Imhoff cone at the top of the solids layer as mL/L settle able matter.

Settleable Solids Flow Chart



Calculation

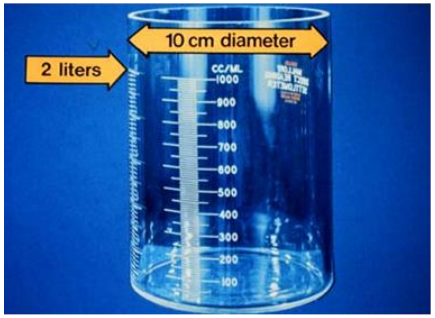
mg settle able solids/L = mg total suspended solids/L mg nonsettleable solids/L

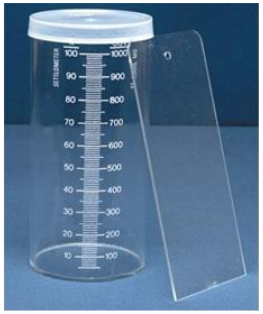
solids (mg/L) = A - B

Where: A = Total suspended solids (mg/L)

B = Non settle able solids (mg/L)

Settled Sludge Volume (SV30)





Settling Column

Timer



Thermometer Types

Beaker

SV_{30} or SSV_{30} Flow Chart

Shak sample, pour it into the settling column distribute solids inverting column 3 times.

Stir well and let suspension settle.

Determine the volume after 30 minutes (SSV or SV).

Sludge volume index (SVI) or Stirred sludge volume index (SSVI)

Shak sample, pour it into the settling column distribute solids inverting column 3 times.

Stir well and let suspension settle.

Determine the volume after 30 minutes (SSV).

Determine the MLSS in g/L.

Calculation:

-The individual SSV (ml/1000 ml) is calculated by dividing the volume of sludge after 30 min. by the volume taken for analysis then multiply by 1000.

$$\text{Inividdual SSV} = \frac{\text{volume of sludge after 30 min. (units/50 units)}}{\text{Volume of sample taken for analysis (units)}} \times 1000$$

-The individual SSVI (ml/g) is calculated by dividing SSV by MLSS.

$$\text{Individual SSVI} = \frac{\text{SSV (ml/ 1000 ml)}}{\text{MLSS (g/1000 ml)}} \text{ ml/g}$$

Percentage of Dry & volatile Solids (DS% & VS%)

Cylinders

Desiccators

Steam bath

Wash bottle

Elec. Balance

Vol. Flask

Drying Oven

Muffle furnace

Beaker

Flow chart

Weigh a clean evaporating basin (W_1)

Pour a sample into the evapo. basin (app. 100-125 ml).

Weigh the evapo. basin and sludge sample (W_2).

Repeat for all samples, QC standard and blank distilled water.

Transfer the evapo. basins to a steam bath to evaporate the water for 1 h.

After drying, Remove the evapo. Basins to the desiccator to cool, Weigh (W_3)

Dry again for 30 min. , Remove the evapo. Basins to the desiccator to cool, Weigh (W_4)

Turn Over

Place evaporating Basins into the furnace at 550 0C for 1 h.

Cool for 30 min. at 110 0C then at desiccator , Weigh (W_5)

Repeat the instructions for the QC standard.

Calculation:

Should the calculation need to be done manually then the following equations must be used:

Total solids = (W3-W1/W2-W1) X 100% w/w (as a % of the wet sludge)

Volatile solids = (W3-W4/W3-W1) X 100% w/w (as a % of the dried sludge)

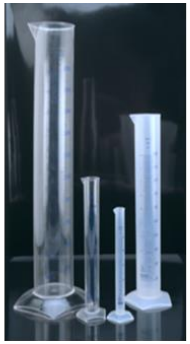
Where: W1 = Empty Dish Weight.

W2= Weight of dish + wet sludge.

W3 = Weight of dish + dry sludge.

W4 = Weight of dish + ashed sludge.

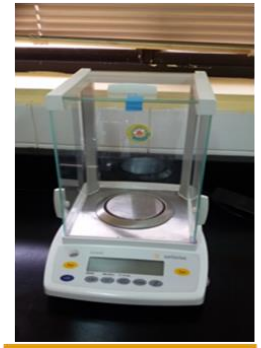
total Alkalinity- T. Alk.



Cylinders

Desiccators Cabinet

Hot Plate



Elec. Balance

Vol. Flask

Drying Oven set at 140 0c

Beaker

-T. Alkalinity (T. Alk.) Flow Chart

A blank distilled water sample must be analyzed with each batch of sample.

Use a pipette or graduated cylinder to place 50 ml of sample in 100 ml beaker.

Fill burette with 0.1 or 0.02 N H_2SO_4

Titrate to PH 4.5 with H_2SO_4

Record on the Alkalinity work sheet, the volume of titrant added to the end-point.

Calculation:

Calculation of Alkalinity for Sewage Sludge.

$$\text{Alkalinity} = \frac{A \times N \times 50, \text{ are}}{\text{Mg CaCo3} \quad \text{ml sample}}$$

A= ml of standard sulfuric acid

N= Normality of standard acid

$$\text{Normality, } N = \frac{A \times B}{53.0 \times C}$$

Where,

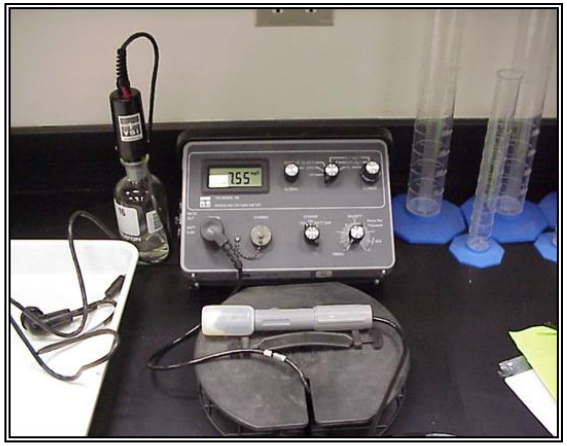
A = g Na2Co3 weighed into 1- L flask

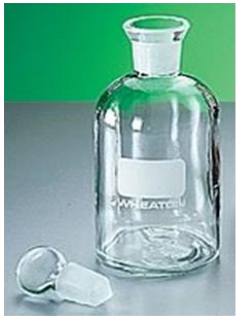
B = ml Na2Co3 solution taken for titration

C = ml acid used

Dissolved Oxygen (DO)

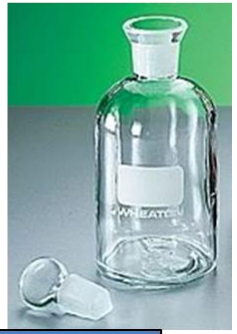
D.O. Meter →





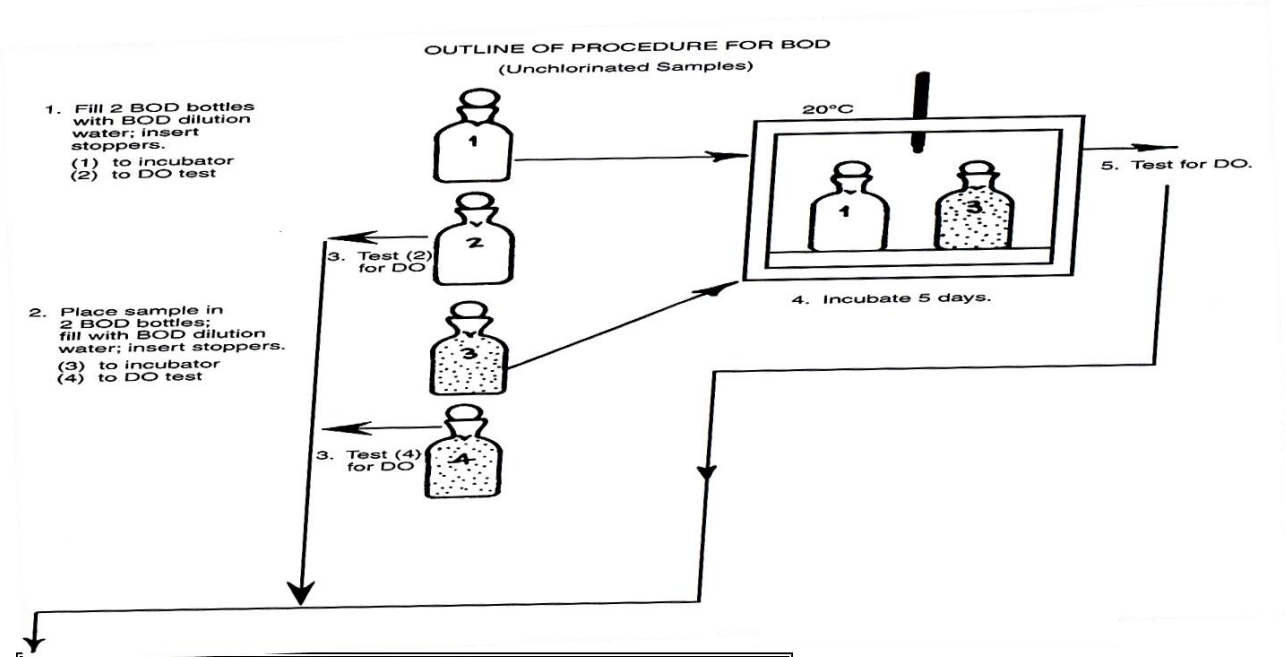
BOD Bottles (300 ml)

Biochemical Oxygen Demand(BOD)

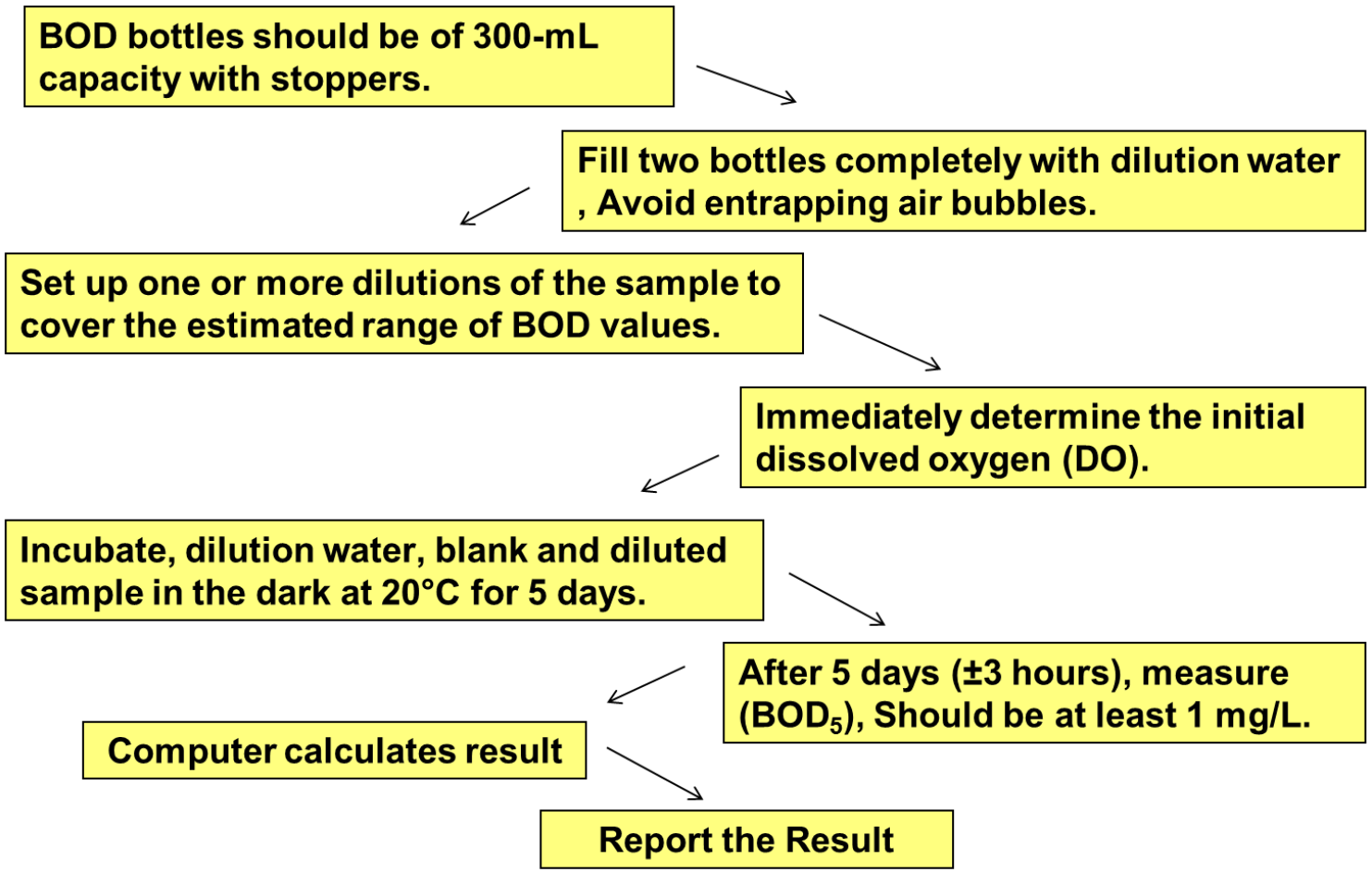


BOD Bottles (300 ml)

BOD Incubator (20 - ,+ 1OC)


OUTLINE OF PROCEDURE FOR BOD
(Unchlorinated Samples)
1. Fill 2 BOD bottles with BOD dilution water; insert stoppers.
(1) to incubator
(2) to DO test
2. Place sample in 2 BOD bottles; fill with BOD dilution water; insert stoppers.
(3) to incubator
(4) to DO test
3. Test (2) for DO
3. Test (4) for DO
20°C
4. Incubate 5 days.
5. Test for DO.

BOD flow chart



$$\text{BOD as mg } O_2/L = \frac{(\text{DO of blank} - \text{DO of sample})*300}{\text{ml of sample}}$$

Or

$$\text{BOD as mg } O_2/L = \frac{[(S_{\text{initial}} - S_{\text{final}}) - (B_{\text{initial}} - B_{\text{final}})]*300}{\text{ml of sample}}$$

Where:

S_{initial} is the concentration of DO in the sample bottle before incubation

S_{final} is the concentration of DO in the sample bottle after incubation

B_{initial} is the concentration of DO in the blank bottle before incubation

B_{final} is the concentration of DO in the blank bottle after incubation

Chemical Oxygen Demand (COD)

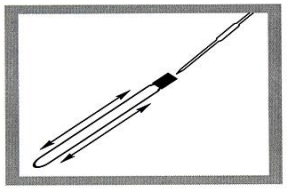
Apparatus

a) COD HACH digestion vessels

b) Heating block digester with 16 or 25 holes and temperature up to 200 ^{o}C to operate at 150 ^{o}C

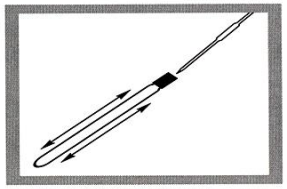
c) Spectrophotometer (620 nm)

OUTLINE OF PROCEDURE FOR COD •



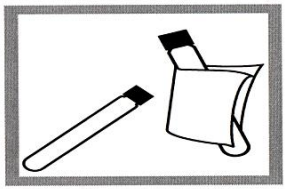
5. Hold one vial at a 45-degree angle. Use a clean volumetric pipet to add 2.00 mL of sample to the vial. This is the prepared sample.

Note: *Use a TenSette pipet to add 0.20 mL for the 200–15,000 mg/L range.*

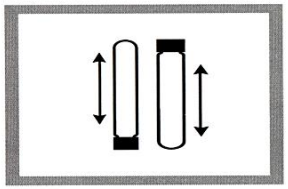


6. Hold a second vial at a 45-degree angle. Use a clean volumetric pipet to add 2.00 mL of deionized water to the vial. This is the blank.

Note: *Use a TenSette pipet to add 0.20 mL for the 200–15,000 mg/L range.*

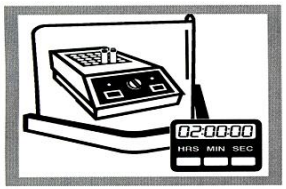


7. Cap the vials tightly. Rinse them with deionized water and wipe with a clean paper towel.

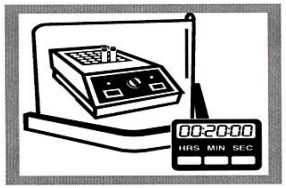


8. Hold the vials by the cap over a sink. Invert gently several times to mix. Place the vials in the preheated COD Reactor.

The sample vials will become very hot during mixing.

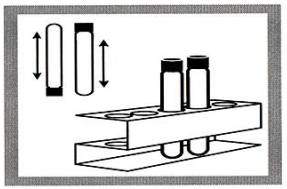


9. Heat the vials for two hours.



10. Turn the reactor off.

Wait about 20 minutes for the vials to cool to 120 °C or less.



11. Invert each vial several times while still warm. Place the vials into a rack and cool to room temperature.

12. Proceed to the *Colorimetric Determination Method 8000* on page 3.

-Take 1.5 ml dichromate solution + 3.5 ml sulfuric acid solution and + up to 2.5 ml sample.

-Prepare a blank by suing 2.5 ml distilled water.

-Shake well and put on the heating block and leave for two hours.

-Turn of and collect the vessels in metal rack and leave to cool to roo temperature

-Measure at 620 nm using HACH spectrophotometer as concentration.

-Use standard to calculate the factor of the apparatus.

Computer calculates result

Report the Result

$$\text{COD as mg O2/L} = \frac{\text{Reading} * 2.5 * \text{factor}}{\text{ml of sample}}$$

How to calculate the factor

-Prepare standard COD solution using potassium hydrogen phthalate (PHP).

-Crush and then dry PHP to constant weight at 120 oC.

-Take 425 mg PHP and dissolve in 1000 ml (this solution has theoretical value of 500 mg COD/l)

-The solution is stable for 3 months in the refrigerator in the absence of biological growth.

-Take 2.5 ml and treat as sample and get the factor of the apparatus.

You can make dilution to get 400, 300, 200 and 100 mg COD/L to have more points for the factor

425 mg potassium hydrogen phthalate to 500 ml distilled water.

This solution 1000 mg COD/L

Take 50 ml and diluted to 500 ml with distilled water to give 100 mg COD/L

From the last solution we take the following

1- 10 ml in 100 ml to give solution with 10 mg/l of COD

2- 25 ml in 100 ml to give solution with 25 mg/l of COD

3- 50 ml in 100 ml to give solution with 50 mg/l of COD

4- 75 ml in 100 ml to give solution with 75 mg/l of COD

5- and the original solution with 100 mg/l of COD

Do as in the previous method and measure at 436 nm to get the value for the factor

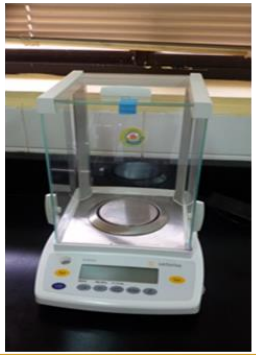
$$\text{Factor} = \frac{\text{Theoretical value of COD}}{\text{measured value (reading)}}$$

$$\text{COD as mg O2/L} = \frac{\text{Reading} * 2.5 * \text{factor}}{\text{ml of sample}}$$

Chlorides

Drying Oven set at 140 0c

Hot Plate



Elec. Balance

Vol. Flask

Cylinders

Reagent bottle

Beaker

Chlorides Flow Chart

-Take ,25 mL of sample and transfer to a 250 mL flask.

-Dilute the sample to app. 100 mL with distilled water.

Add 1 mL of potassium chromate solution.

Add Ag_2NO_3 titrant, stirring the sample, to a pink/yellow end-point.

Record the titer and standard NaCl solution volume taken, on the chloride work sheet.

Report the Result

Calculation:

Use the following equation

$$\text{mg } CL^{`} / L = \frac{(A-B) \times N \times 35.450}{\text{ml Sample}}$$

Where:

A = ml titration for sample

B = ml titration for blank, and

N = Normality of Ag NO_3

mg NaCl / L = (mg CL^{-} / L) × 1.65

Ammonia - nitrogen

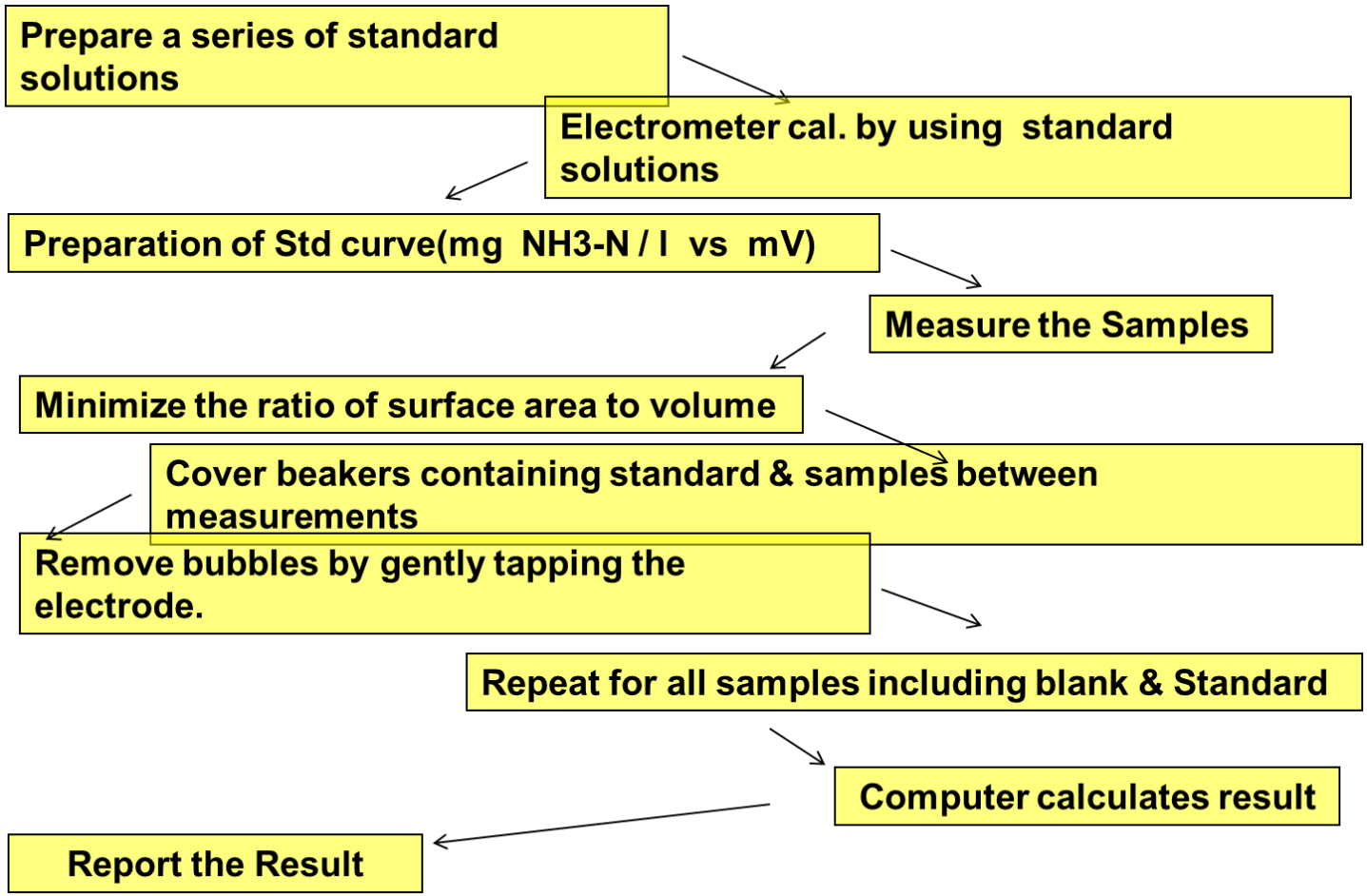
PH Meter

ISE ammonia Meter

Dispenser

Volumetric pipettes

Ammonia – N Flow Chart



16-Total Nitrogen - TN

Digestion Unit

Titration & Distillation Unit

Scrubber Unit

Total Nitrogen – TN Flow Chart

#The assay can be divided in three separate steps:

a-Digestion with sulfuric acid and a catalyst

b-Distillation with water steam

c-Titration and calculation of results

a. Digestion

The oxidative disintegration of the N-containing bonds with conc. H_2SO_4 to N as ammonium-ion.

b.Distillation

Control rate of steam generation to boil contents in distillation unit occurs.

c.Titration

The volumetric determination of the nitrogen content is now done using the end point titration to a set pH Value.

Calculation of the Result

- The following formula is used to calculate the nitrogen content in percentage:
- % N = (c x (V – VBL) x M x100 %) / E
- c = Concentration of the titration solution in mol/ml
- V = Consumption of titration solution in 1
- VBL = Consumption titration solution blank value in 1
- M = Molar mass nitrogen in g/mol
- E = Initial sample weight

OIL AND GREASE(O&G)

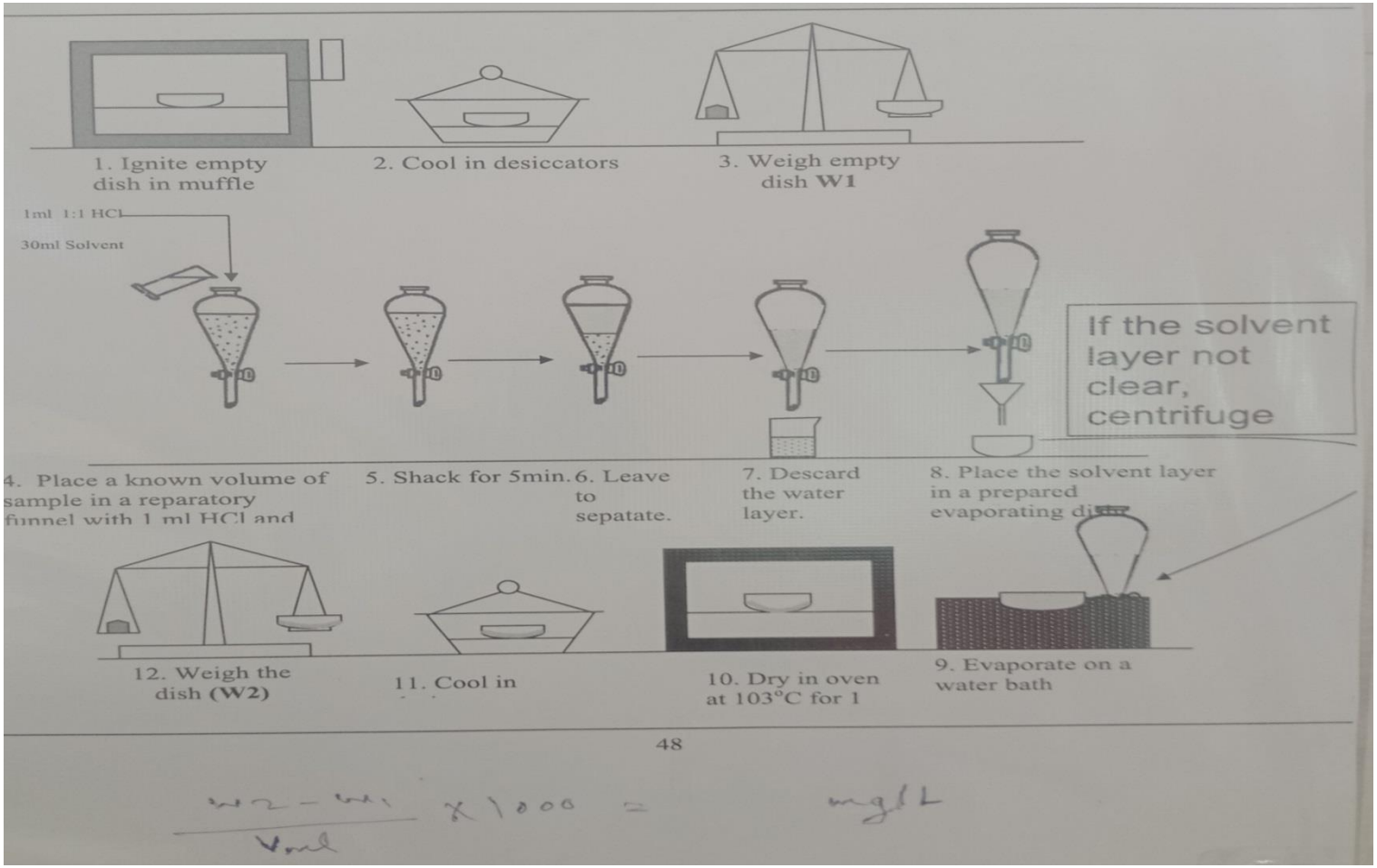
Separating funnel

Elec. Balance

Steam both

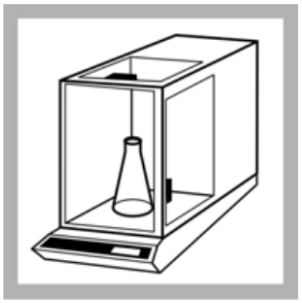
Condense

Desiccator


1. Ignite empty dish in muffle
2. Cool in desiccators
3. Weigh empty dish W1
1ml 1:1 HCl
30ml Solvent
If the solvent layer not clear, centrifuge
4. Place a known volume of sample in a reparatory funnel with 1 ml HCl and
5. Shack for 5min.
6. Leave to sepatate.
7. Descard the water layer.
8. Place the solvent layer in a prepared evaporating di
12. Weigh the dish (W2)
11. Cool in
10. Dry in oven at 103°C for 1
9. Evaporate on a water bath
48
W2 − W1 / Vml x 1000 = mg/L

1. Collect 500 mL of sample in a clean 1000-mLseparatory funnel

2. Add 4 mL of 1:1Hydrochloric, Mix well, pH must be ≤ 2 Check sample.



3. weigh dried and Record the weight of the flask.

4. Add 20 mL of n-hexane to the sample

Sulphides - H2S

Beaker

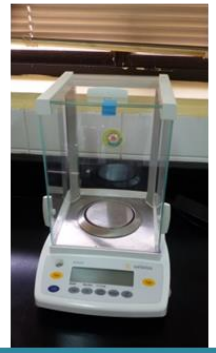
Flask

Cylinders

Magnetic Bars

Magnetic Stirrer

Vol. pipettes

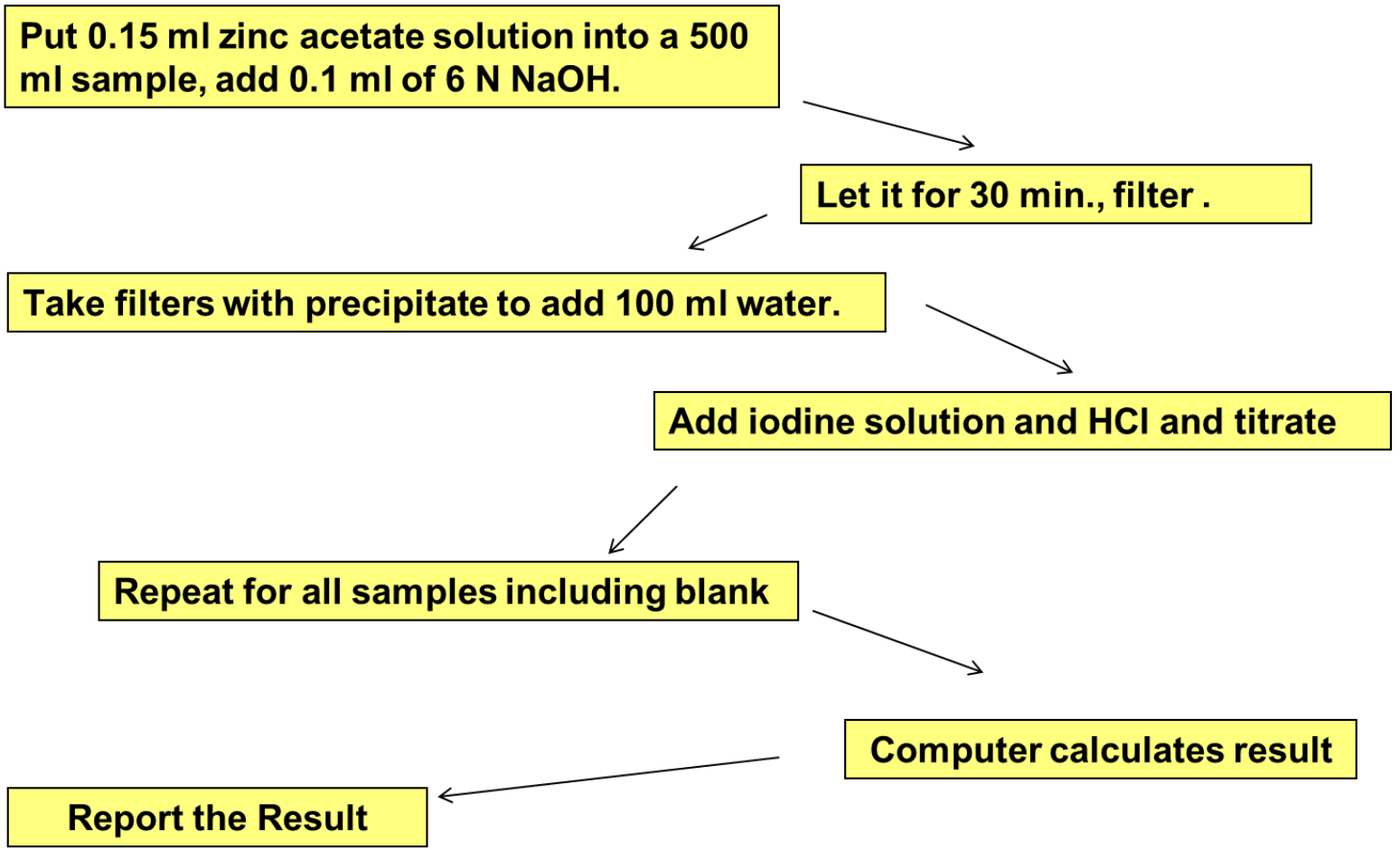


Elec. Balance

Vol. Burette

Vol. Flask

Sulphides Flow Chart



Calculation:

$$\text{mg S-2/L} = \frac{[\,(A \times B) - (C \times D)\,] \times 16000}{\text{ml of sample}}$$

Where:

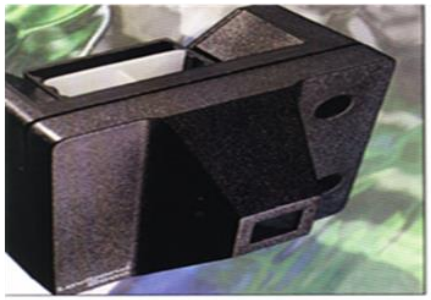
A = ml of iodine solution added

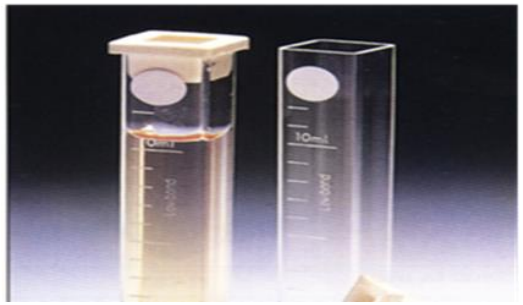
B = normality of iodine solution

C = ml of sodium thiosulfate solution added

D = normality of sodium thiosulfate solution

قياس الكلور





- ما هى اهم التجارب فى المعمل ؟

عوامل التحكم فى عملية التشغيل

- عمر الحمأة SA

$$\frac{\text{تركيز الكائنات الحية فى حوض التهوية}}{\text{تركيز الكائنات الحية الخارجه من حوض التهوية}}$$

$$\frac{MLVSS * V}{WASvss * Qwas}$$

- نسبة الغذاء الى الكائنات الحية F/M

$$\frac{\text{وزن المواد العضوية}}{\text{وزن الكائنات الحية}}$$

$$\frac{BOD * Q}{VSS * V}$$

دليل حجم الحمأة SVI

$$\frac{\text{الحمأه حجم المترسبة فى 30 دقيقة}}{\text{تركيز المواد الصلبة العالقة فى حوض التهوية}}$$

$$\frac{SV30 \ * 1000}{MLSS}$$

- كمية الحمأه المعادة QRAS
- $$\frac{MLSS * Q}{RASss - MLSS}$$
- كمية الحمأة المنصرفة (الزايدة) Q WAS
- $$\frac{MLVss * V}{SA * WASvss}$$