STANDARD OPERATING PROCEDURES OF THE EQUIPMENT AND SERVICES AT CENTRAL INSTRUMENTATION FACILITY

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HOUSE KEEPING GENERAL

1. All equipment test apparatus for testing such as flask, beaker, pipettes, burette, sieves etc. are cleaned in appropriate cleaning solutions, rinsed with water, dried and stores.
2. All used material, containers and polythene bags and other debris must remove from the area.
3. Area is swept and also cleaned (floors, walls, ceilings and ventilators) to remove the dust. The floor is mopped with detergent and water.
4. When the cleaning has been done, the technical manager supervises the cleaning work of area/equipment.

GENERAL HOUSEKEEPING PROCEDURE:-

1. General housekeeping is carried out by IISS-Central Lab employee. Appropriate cleaning material is used in the different area.

2.The frequency for cleaning also varies from area to area. The testing areas are cleaned daily or more if required. Other general areas are normally cleaned once a day. The staff from the concerned areas supervises these operations.

LABORATORY DISCIPLINE

1. Analysis is being carried out according to the Standard procedure or specification.

2. All observations and calculations are to be recorded on analytical data calculation sheet/register.

3. Wrong entries are scored with a single line and the new entry made along side with an initial of the person correcting the mistake. Do not overwrite.

4. Analytical work carries the date on which it was carried out. Test Report are attached to the analytical report.

5. All reagent bottles/chemicals/volumetric solutions are labelled/marked and stored in the identified Area or shelves. These are to be checked /standardized on regular basis.

6.All working standards reference standards are stored in appropriate place immediately after use.

7. Labels indicate the date on which the reagents were prepared and standardized.

8. Labels, which are not correct, are removed or crossed with marker. Whenever a Solution changed or freshly standardized, solvent bottles are kept in respective shelves after use.

9. Laboratory is maintained in a clean and orderly manner all the times.

10. Care is taken that instruments are switched "OFF". Instruments are calibrated as per frequency given in the individual work instruction.

11. Keep your workbench clean and uncluttered. Remove unnecessary apparatus promptly.

12.At the end of the working hours, analysts ensure that all things maintained properly live used Glassware, reagent bottles etc.

LABORATORY SAFETY

CORROSIVE CHEMICALS:

1. Avoid direct contact of the chemicals with body surface including elementary and respiratory system. Handling should be careful and use rubber gloves, lab coats and their necessary safety apparels.

2. Do not store large bottle of acid and other corrosive materials above waist level.

3. Do not leave apparatus containing corrosive material at the sink, as it is always empty the apparatus before leaving for washing.

4. Bottles containing strong acids should be placed at ground level. Acid splashed should be washed with water and then neutralized with Sodium Bicarbonate solution.

GLASS WARE:

1. Examine defects of damaged glassware before any experiment. Do not used cracked, chipped or otherwise damaged glassware.

2. Reagent bottles and other glassware should be labelled correctly and clearly.

3. Pour liquids in a fire section away from label to avoid damaging of labels. If any liquid spills on outside of bottles, wash the outside of bottle with water before returning to shelves.

4. To remove tight stoppers, tap alternatively on each side of the stopper. If this does not work and the contents of the bottle are not flammable toxic, gently warm the neck of the bottle.

5.When glass tube or rod is to broken in smaller parts, use cloth gloves for protection.

ELECTRICAL HAZARDS:

1. Ensure that there are no open connection and bare wire in the department.

FIRE HAZARDS:

1. Fire extinguisher is fastened so that they cannot roll or fall.

2. All laboratory staff must be familiar with the position and operation of all firefighting in their vicinity.

3. Vacuum pumps must be checked for oil level at regular intervals.

GLOVES:

These should be used for handling concentration alkali and other corrosive material, where finger skill is essential. Surgeon's gloves can be used.

LABORATORY COATS:

These must be worn / used during analysis work for protection of body and clothes. Apron can be used while handling corrosive materials.

FUMING CHAMBER:

Any work involving fumes and harmful gases should be carried out in a fuming chamber. Make sure the ventilation is in order during the operation.

FIRST AID BOX:

This is equipped with medicine like antiseptic lotions, ointments for burns etc.

CLEANING OF GLASSWARE

GLASSWARE'S ARE CLEANED IN THE FOLLOWING MANNER: -

1. Tap water / Fresh water
2. Chromic acid
3. Tap water
4. Rinsing with distilled water

PROCEDURE:

1. All the glassware is dipped in plastic tubes with toluene and water for 30 minutes.

2.Individual glassware's are washed under tap water and held vertically downward on stand to render them free from water and dried in an oven after rinsing with distilled water.

3. If no stain is found, they are put in the oven for drying.

4. In case of any stain is there, the affected glassware's are washed with chromic acid followed by washing with freshly collected water and then kept under the tap for rinsing and washing. If no stain is found they are rinsed with distilled water and put in the oven for drying.

5. In case if further stain exists; all that glassware are dipped overnight in chromic Acid solution and the next day. These glassware's are taken out of the chromic acid solution and washed with tap water. They are further rinsed with distilled water.

NOTE: As and when required, brushing can be done, but normally brushing is avoided to prevent scratching on the walls of the glassware.

PREPERATION OF CHROMIC ACID

Dissolve 200 gm of potassium dichromate in about 100 ml of distilled water, cooled in an ice bath to which 1500 ml of sulphuric acid has been added slowly with stirring. This mixture should be prepared in a hard, borosilicate glass beaker.

PRECAUTIONS WHILE USING CHEMICALS

1. Keep the chemical in appropriate environment condition, as per provided by the manufacture.

2. Keep the container tightly closed.

3. Keep container in a well-ventilated place.

4. Keep away from food and drink.

5. Keep away from heat ignition.

6. Handle and open container with care.

7. While using chemicals, do not eat or drink.

8. While using chemicals, do not smoke.

9. Avoid contact with the skin.

10. Avoid contact with eyes.

11. Take off immediately all contaminated clothing.

12. Use gloves, while using hazardous chemicals.

13. Proper ventilation is required in storage area of chemicals.

14. In case of fire and/or explosion do not breathe fumes.

15. Keep always fire extinguisher near the approach, if happened any accident.

16. In case of accident by inhalation, take away casualty to fresh air and keep at rest.

17.If swallowed, rinse mouth with water (only if the person is conscious).

OPERATING PROCEDURE OF FLAME PHOTOMETER

1. Power the flame photometer and compressor pump on.

2. On the knob of the LPG gas cylinder and ignite the flame of the photometer.

3. Set the flame cone to desired sharpness.

4. Go to the calibration menu, select the element Na or K or both.

5. Enter different standard value one by one.

6. Now calibrate the instrument with standards of 0, 10, 25, 50 and 100 ppm by aspirating the distilled water and each standard one by one, till the "calibration over" is displayed on the LCD screen.

7. Rinse the sample tubing with distilled water.

8. Go to the sample menu, press sample 1 and record the reading in ppm of the sample and save.

9. Rinse the sample tubing with the distilled water before putting next sample.

10. Similarly continue the analysis of the rest of the samples and save the data.

11. After analysis is over, off the flame and close the LPG cylinder

12. Now off the power of the compressor pump and instrument.

13. Clean the surrounding area after analysis is over.

OPERATING PROCEDURE OF AAS

1. Power the Atomic Absorption Spectrophotometer, Compressor pump and Computer system on.

2. On the knob of the acetylene gas or nitrous oxide cylinder as per requirement of the element.

3. Double click the win_AAS / SPWin_AAS software on the monitor of the computer.

4. Set the desired parameters as per cookbook menu of the AAS.

5. Load the desired element from the loaded HCL lamp table in the software by clicking the element.

6. Set the lamp current and slit value to get maximum output.

7. Select the autos sampler from the menu and put the number and concentration of standards and samples at the desired places.

8. Set the number of standard in the menu and enter the value of the each standard.

9. Set the units of the measurement of the standard.

10. Set the number of standard run to get the linear curve.

11. Set the number of measurement to be recorded as blind run and number of measurement as actual for the standards.

12. Set the units of the measurement of the samples.

13. Set the number of measurement to be recorded as blind run and number of measurement as actual for the samples also.

14. Set the number of samples to be analysed.

15. Set the statistical parameter applied to the results.

16. Ignite the flame and check and select the burner width, length and height.

17. Power on the exhaust fan system to clear out the burnt gases.

18. Now calibrate the AAS with standards of 0, 0.5, 1 and 2 ppm by aspirating the distilled water and each standard one by one, till the linear "calibration curve" is displayed on the monitor screen in the software.

19. Append the linear calibration curve to the entire samples.ac.

20. Rinse the sample tubing with distilled water and click the sample menu and run the samples and append them to the linear calibration curve drawn.

21. Close the software and shut down the computer system.

22. Now power off the AAS, compressor pump and exhaust system.

23. Clean the surrounding area after analysis is over.

OPERATING PROCEDURE OF ICP-OES

1. Power the Inductively Plasma Emission Spectrophotometer (ICP), and Computer system on.

2. Power on the both water circulating chillers pump and wait for half an hour to reach the desired temperature level of 10 and 20°C each chiller pump.

3. On the knob of the argon gas cylinder.

4. Double click the "ICPE solution" software on the monitor of the computer, click new and power on the vacuum pump in the menu.

5. Set the desired pressure of the argon gas.

6. Load the desired number of elements from the menu in the software by clicking the elements one by one, selecting its wavelength and adding in the analysis table.

7. Select standard torch and set standard, sample and rinsing time for all the samples and standards.

8. Put the different concentration of standards and samples at the desired places in the autos sampler.

9. Set the number of standard in the menu and enter the value of the each standard.

10. Set the units of the measurement of the standard.

11. Set the number of standard run to get the linear curve.

12. Set the number of measurement to be recorded as actual for the each standard.

13. Set the units of the measurement of the samples.

14. Set the number of measurement to be recorded as actual for the each sample also.

15. Set the number of samples to be analysed.

16. Set the statistical parameter applied to the results.

17. Power on the exhaust fan system to clear out the burnt gases.

18. Ignite the plasma and wait for the ready signal to appear at the top-right side of parameters window on the monitor.

19. When all the parameters on the right side of the monitor become green or white i.e., no parameter should be in red color then ready sign will appear.
20. Now start the ICP and calibrate with 0, 125,250 and 500 ppb standards by aspirating the distilled water and each standard one, till the linear “calibration curve” is displayed on the monitor screen in the software of each element separately.
21. Click the continuous mode of measurement.
22. After the completion of the analysis, go the calibration and profile menu to reprocess all the samples by peak searching and wave length integration.
23. Take the printout of the result after reprocessing the data.

24. Close the software and shut down the computer system.
25. Now power off the ICP, vacuum pump and exhaust system.
26. Clean the surrounding area after analysis is over.

OPERATING PROCEDURE OF IC

1. Power the lon chromatograph and computer system on.

2. Prepare fresh eluent solution, dilute sulphuric acid and distilled water and refill all chemicals in the separate bottles on top of the instrument.

3. Double click the MagicNet 2.0 software on the monitor of the computer and also power on the autosampler

4. Set the desired method in the analysis table on which anion analysis is to be done.

5. Reset the analysis table and enter the standard as well as sample method, position, volume, dilution etc in the analysis table.

6. Load the number of standards and samples starting from position number 2

7. Generally, position 2, 4 and 6 are kept for standard during standard curve drawn and also while verifying the known value.

8. Position greater than 6 i.e., 8 to 34 are used for keeping samples.

9. As per the method developed, even number are used for keeping standards and samples, while odd positions are used for keeping distilled water to rinse the previous sample. i.e., after every standard or sample, there is a distilled water tube in the auto sampler.

10. Select the method, keep the standards and samples at the desired position and run the ion chromatograph.

11. Check out the flow of the all three reagent in the drain and the sample drain also. All three solutions should come out slowly in the drain behind the instrument.

12. As the equipment starts, peak will start coming on the monitor screen at regular interval in the order i.e., F, CI, NO_3, PO_4 and SO_4.

13. Analysis of one sample for five parameters to take about 30 minutes.

14. Keep checking the flow of the solution and the sample during the whole day.

15. After the analysis is over, reprocess the sample with the table of reprocessing to get the correct data.

16. Close the software and shut down the computer system.

17. Now power off the ion chromatograph and auto sampler

18. Clean the surrounding area after analysis is over.

STANDARD OPERATING PROCEDURE OF pH METER

1. Powers the pH meter on. After power on, the meter automatically begins in the mode that was last used. Calibration and memory values are retained even if meter is unplugged.

2. Press MODE to select pH.

3. Dip the pH and ATC electrodes into pH buffer and press **CAL/MEAS.**

4. To eliminate temperature errors associated with the pH electrode, attach the automatic temperature compensation (ATC) probe for best accuracy. Without temperature compensation, pH accuracy will worsen as samples deviate from 25°C.

5. The secondary display will lock on the appropriate buffer value. Provide stirring for best results. When the **READY** indicator appears, press **ENTER** to accept. The primary reading will flash briefly before the secondary display begins scrolling the remaining available buffers.

6. Rinse the pH and ATC electrodes then dip into the next pH buffer. The secondary display will lock on the appropriate buffer value. When the **READY** indicator appears, press **ENTER** will flash briefly then display the percent efficiency (slope) before the secondary display begins scrolling the remaining available buffers.

7. To calibrate another buffer repeat step 5 or press **CAL/MEAS** to return to the measurement mode. The meter will automatically return to measurement mode upon successful completion of the number of specified calibration points.

Note: A single point (offset) calibration is only allowed with pH 7.00 or pH 6.86 buffers. When the first calibration value is accepted during a new calibration, all prior calibration values are erased. Press CAL/MEAS at any time to abort calibration and return to measurement mode.

STANDARD OPERATING PROCEDURE OF HOT AIR OVEN

1. Connect the power supply.

2. Switch "ON" the main power supply and instrument mains.

3. Press SET POINT (x/w) key to set the required temperature. Press ↑ to increase the temperature and↓ to reduce the temperature

4. The temp. Sensor will maintain the set temp which is indicated by the blinking of set temp on the display screen.

5. The duration of time can also be adjusted using the time adjustment knob.

6. After use, SWITCH OFF the power supply.

Cleaning: Wipe the surface, walls, top, bottom and trays of the oven with dry lint free cloth on daily basis so that there will be no dust particles in the oven. Wipe all the parts and outer surface of the Oven with wet lint free cloth) soaked in purified water, on weekly basis and weekly cleaning.

STANDARD OPERATING PROCEDURE FOR DIGITAL WEIGHING BALANCE

1.1. Procedure

1.1.1. Operate the instrument as per respective standard operating procedure

1.1.2. Switch on the balance

1.1.3. The display will blink 8.8.8.8.

1.1.4. After few second display will show 0.0000g

1.1.5 If display is not stable, close all the shutters, running nearby fans, press tare key and wait until display shows 0.0000g.

1.1.6. Perform internal calibration as in Annexure -1

1.2 For Accuracy

1.1.7. Place 0.5 gm. on the weighing pan.

1.1.8. Note the weight.

1.1.9. Calculate the difference between the weight in certificate and observed weight.

1.1.10. Repeat the above steps using 1 gm & 5 gm. weights.

1.1.11. Record the reading in Annexure-II.

1.2. For precision

1.2.1. Place 1 gm. in the weighting pan.

1.2.2. Note the weight.

1.2.3. Repeat the above two steps five times.

1.2.4. Record the weight in Annexure-II.

1.2.5. Calculate the standard deviation.

1.2.6. Calculate the measurement uncertainty using following equation

1.2.7.

$$\text{Measurementuncertainty} = \frac{3 \times SD}{\text{Actual weight from certificate}}$$

1.2.8. Frequency

Internal calibration and accuracy = once in month

Precision: Once in a three month

2. Cleaning procedure for balance

2.1. Switch OFF the instrument and mains.

2.2. Open the shutter of the instrument and remove the pan which is inside the balance.

2.3. Remove the dust and powder with the help of tissue paper.

2.4. Soak the tissue paper in isopropyl alcohol and clean the balance with this tissue paper.

2.5. Finally clean with tissue paper and care to be taken for proper drying during use of isopropyl alcohol.

2.6. Clean outside with tissue paper wetted with isopropyl alcohol and then with dry tissue paper.

2.7. After cleaning close the shutter and maintain record of cleaning.

2.8. Frequency of cleaning is wherever necessary.

3. Abbreviations

3.1. SD Standard deviation

3.2. gm.= Gram

Annexure-I

Calibration Date	Date of Last Calibration	Next due date for Calibration

Instrument name	Instrument make	Instrument Identification no.

For Precision

Observations weight Box I.D. No: Instrument Identification no.			
Theoretical weight	Actual weight from certificates	observation weight	Tolerance
1 g			Not more than 0.001
	SD		

Annexure II

DATE

EQUIPMENT ID

SECTION--

CALIBRATION

Nominal wt(g)	Actual Value	Observed reading							(3*SD)/ Nominal	0.1% Nominal weight
0.5000		1	2	3	4	5	Average	Std. Dev		
1.0000										
2.0000										
5.0000										
20.0000										
50.0000										

RESULTS

CALIBRATION STATUS

CORRECTIVE STEPS (If ANY)

CALIBRATED BY

STANDARD OPERATING PROCEDURE OF SHAKER

A shaker is a piece of laboratory equipment used to mix, blend, or agitate substances in a tube or flask by shaking them. It is mainly used in the fields of chemistry and biology.

While working on a Shaker, one must read the safety guidelines provided by the manufacturer.

Procedure:

1. Check for all electrical connections and plug points. Ensure all the connections are in good condition.
2. Connect the shaker with the electrical plug point.
3. Press the main switch to ON position. (On/OFF)
4. Mount your samples evenly to ensure balanced shaking.
5. Ensure that the platform and clamps are securely attached and tightened to prevent sample spillage.
6. Start the shaker by turning on the shaker switch.
7. Adjust the speed and time settings according to your application requirements. Start with lower speeds and gradually increase if necessary.
8. After stipulated time, turn off the shaker and allow it to slow down before cleaning.
9. Clean the shaker thoroughly to prevent contamination of future experiments.

STANDARD OPERATING PROCEDURE OF BOD INCUBATOR

An incubator is a heated, insulated container used in laboratories to cultivate and preserve cell or microbiological cultures at specific temperature.

Procedure:

1. Make sure that the incubator is securely linked to the power source.
2. Turn on the main power.
3. Next step is to set the temperature of the incubator.
4. To set the incubator, press the knob to RESET and by pressing the temperature button (UP/DOWN), increase/decrease the temperature to arrive at a fixed value.
5. For changing the temperature, use the knob to reset and modify the temperature as described earlier.
6. Keep a daily and nightly temperature log, specifically, in the morning and at night. A 2°C difference from the stated temperature is acceptable.

STANDARD OPERATING PROCEDURE OF LAMINAR FLOW

Laminar Air Flow is intended to carryout work under aseptic condition to avoid contamination of the microbiological material and also to prevent escape of microbes from designated lab.

Procedure:

1. Ensure that all the electrical plug points are working properly.
2. Switch On the main plug of LAF chamber from main board.
3. Prior to starting work under LAF chamber, switch on the UV lamp inside the chamber for 15min
4. After 15 min. switch off the UV lamp and switch on the Bright light.
5. Lift the front door of LAF Chamber and switch on the air blower.
6. Clean the bench thoroughly with 70% ethanol and ignite the burner is needed.
7. Perform all the work under flame zone to avoid any contamination
8. After finishing the work, put off the burner, wipe the LAF bench with 70% ethanol
9. Switch off the light and flow of blower and close the front door of LAF chamber

Precaution:

1. Always perform work inside the chamber to avoid contamination
2. do not take any material out of LAF chamber
3. LAF should not be crowded with unnecessary materials
4. Person working should wear safety goggles, gloves and mask while working
5. keep alcohol away from flame during disinfection
6. do not talk or eat near LAF chamber

STANDARD OPERATING PROCEDURE OF AUTOCLAVE

Procedure:

1. Clean the outer body of the autoclave
2. Check the water level in autoclave and make up to the mark with distilled water
3. Put the materials to be autoclaved in the inner bucket and tight the clamps
4. Switch on the plug from main board and then from equipment panel
5. Set the temperature to 121ºC from the set button and then press enter
6. Set time and then press enter then long press Start
7. Observe for increase in temperature, once the autoclave reach to the desired temperature the timer stars and beeps at the end of cycle.
8. Switch off the autoclave from equipment panel and then from main board.
9. Let the pressure released slowly by turning the steam release lever and then take out the materials from the autoclave once the pressure is released totally.

STANDARD OPERATING PROCEDURE OF EC METER

1. Switch on the instrument by pressing the power button.
2. Rinse the probe with distilled water and shake off the excess water or tap it dry using a tissue paper.
3. To eliminate temperature errors associated with the EC electrode, attach the automatic temperature compensation (ATC) probe for best accuracy.
4. Check the calibration condition of the instrument using 0.01 *N* KCl or 0.1 *N*KCl standard solution. The value of EC should be 1.411 dS m^{-1} and 12.89 dS m^{-1} for 0.01 *N* KCl and 0.1 *N*KCl at 25°C, respectively.
5. If any discrepancy is noted, calibrate the instrument using the 0.01 *N*KCl/ 0.1 *N*KCl standard solution.

   For Calibration follow the steps given below:

   - The display should read as 0.00 µS/cm with probe in air. This should be done first and then immerse the probe into the standard solution. Raise and lower the probe to refill the center cavity and tap the probe repeatedly to remove any air bubbles that may be trapped inside the sleeve
   - Press [**Cal**/Modify] to enter calibration. The "CAL" tag and the recognized standard value will appear on the LCD. If necessary, press the **ARROW** keys to select a different standard value. Wait for some time and then press [GLP/**CFM**] to confirm the standard reading and save the setting.
6. Use the instrument only after proper calibration.
7. For analysis of the test solution, rinse the probe with distilled water. Submerse the probe in the test solution. The sleeve holes must be completely submersed.
8. EC reading will be displayed on the LCD. Wait until the reading gets stabilized and stops blinking
9. Record the stabilized reading in dS m^{-1}/ µS cm^{-1}.
10. Similarly, continue the analysis of the rest of the samples and record the data. Rinse the probe with distilled water after every sample.
11. After analysis is over, rinse the probe with distilled water and pat dry lightly using tissue paper or shake off the excess water.
12. Clean the surrounding area after analysis is over.

Note

- Calibrate the instrument frequently, especially if high accuracy is required. The instrument should be recalibrated:
  - ✓ Whenever the EC probe is replaced.
  - ✓ At least once a week.
  - ✓ After testing aggressive chemicals.

- Pour small quantities of the standard solutions into a beaker. If possible, use a plastic beaker to minimize any EMC interferences. For accurate calibration and to minimize cross-contamination, use two beakers for the standard solution. One for rinsing the probe and one for calibration.
- If possible, center the probe in the beaker away from the bottom or beaker walls, immerse the probe in the test solution up to the set mark in the electrode.

STANDARD OPERATING PROCEDURE OF SPECTROPHOTOMETER (SYSTRONICS DOUBLE BEAM UV-VIS)

1. Switch on the instrument by pressing the power button.
2. After the instrument initializes automatically, allow the instrument to warm up for 20 minutes.
3. In order to prepare a standard curve, follow the given steps:
    - On the screen, select [**Quantitation**] followed by [**create curve**] and [**ok**].
    - Select the *'fit mode'* as '*first order'* and *'Calib methods'* as *'std sample'*.
    - Enter the *sample number*, *Concentration unit* and set the *wavelength* in nm and select [**Ok**]
    - Fill the cuvette with the standard solution and place it inside the instrument. Fill another cuvette with blank solution and place it in reference holder. Care should be taken to hold the translucent side of the cuvette and place its transparent side in line with the light source.
    - Input concentration and record the observation for each standard solution.
    - At the end press [**Ok**], a graph will be generated. Check for the fit of the curve using 'R^2' value and proceed if found satisfactory. Save the file with a file name.
4. For analysis of the samples, press [**Esc**], reselect [**Quantitation**] followed by [**Open curve**] and select the desired file and press [**Ok**].
5. Fill the cuvette with the test solution and place it inside the instrument. Wipe any excess water on the cuvette with a tissue paper and tap if any bubbles are observed.
6. Record the observation (absorption/concentration) for each sample.
7. After analysis is over, rinse the cuvette with distilled water and wipe it dry using tissue paper and place it back in its case.
8. Clean the surrounding area after analysis is over.

STANDARD OPERATING PROCEDURE OF KJELDAHL APPARATUS-N DISTILLATION UNIT (KELPLUS UNIT)

1. Switch on Kelplus distillation unit.
2. Switch on Kelplus freezer unit.
3. Ensure that water level in the freezer unit and the dilution bottle is filled up to the marked level.
4. Fill in the required reagents ($KMnO_4$/NaOH) into the specified bottles.
5. Ensure the inlet tubes are immersed in the reagents.
6. Weigh the required quantity of soil and put it in the distillation flask.
7. Place the distillation flask properly at the distillation end. Ensure there is no leakage.
8. Place an Erlenmeyer flask (250 mL) containing boric acid (H_3BO_3) mixed with indicator solution under the tip of the condenser, with the tip touching the surface of the solution.
9. Tap on [**Power**] icon followed by [**Auto run/Manual**] on the LCD screen of the instrument.
10. For Auto run option, program needs to be set and saved as per the quantity of required reagents and can be used for analysis directly.
11. For manual option, tap on [**Alkali**] followed by [**Run**]. Run for the required seconds (for 50 mL run for 13 seconds).
12. Select [**$KMnO_4$**] and run for the required seconds (for 50 mL run for 13 seconds).
13. Tap on [**Process**] to start the distillation process
14. Continue with distillation until 150 mL of distillate is collected.
15. Use the collected distillate for further titration with sulfuric acid.
16. Repeat the process for all the samples.
17. Clean the surrounding area after analysis is over.

Note:

- Run the distillation process once with distilled water before the start and end of the analysis to rinse the instrument.
- Remove the distillation flask with the help of gloves
- Keep the outlet tube immersed in the boric acid solution
- Maintain the water levels in the freezer and dilution bottles up to the set mark.

STANDARD OPERATING PROCEDURE OF HYDROMETER METHOD FOR SOIL TEXTURE

- Weigh 50 g of air-dried soil passed through 2 mm sieve into a 500 mL beaker.
- Add 100 mL of 5% Sodium hexametaphosphate (Calgon) solution,
- 250 mL of distilled water, and allow the sample to soak overnight.
- Weigh another sample of the same soil (20g) for determination of oven-dried weight. Dry overnight at 105°C, cool, and weigh.
- Transfer the Sodium hexametaphosphate -treated sample to a dispersion cup and stir for 5 minutes with a mechanical stirrer.
- Transfer the suspension to a 1000 ml cylinder and add distilled water to bring the volume to 1000 ml.
- Allow time for the suspensions to equilibrate to room temperature (between 20°C and 25°C).
- Insert the plunger and move it up and down to mix contents thoroughly. Finish stirring with two or three slow and smooth mixing. As an alternative mixing procedure, stopper the cylinder and use end-to-end shaking for one minute. Record the time of completion of stirring. Add a drop of amyl alcohol if the surface of the suspension is covered with foam.
- Lower the hydrometer carefully into the suspension, note the reading at 40 seconds and record the temperature of the suspension.
- Remove the hydrometer carefully after taking the reading at 40 seconds. Rinse it with water, and wipe it dry.
- Reinsert the hydrometer carefully, take another reading at 2 hours and also record the temperature of the suspension.
- Make up a blank cylinder with water and sodium hexametaphosphate. Record the blank hydrometer reading. If the reading is above 0 (zero) on the hydrometer scale, record the blank correction as a negative number. Read at the top of the meniscus as before.

Calibration of hydrometer

- Add 100 ml of the HMP solution (calgon solution) to a sedimentation cylinder and make up to 1 litre with distilled water at room temperature.
- Mix thoroughly the suspension with the plunger and record temperature after equilibrium.
- Lower the hydrometer into the solution carefully and take the hydrometerreading of the solution as Blank (RL). Read the upper edge of the meniscus surrounding the stem.
- Periodically recheck RL during the course of the hydrometer test. The calibration value RLis used in the analysis to correct for solution viscosity and to correct the soil solution concentration, C.

STANDARD OPERATING PROCEDURE OF MICROWAVE DIGESTION

Procedure

- ✓ Power on the microwave digestion system.
- ✓ Choose or program the appropriate digestion method based on the sample type and acid used.
- ✓ Choose programs include ramping temperature and pressure settings.
- ✓ Place the sealed digestion vessels into the microwave cavity.
- ✓ Start the microwave digestion process according to the selected program.
- ✓ Observe the digestion process, ensuring no leaks or unusual occurrences.
- ✓ Allow the vessels to cool down before handling.
- ✓ Carefully open the digestion vessels in a fume hood.
- ✓ Transfer the digested samples to appropriate containers for further analysis.
- ✓ Now power off the MDS system.

Programs settings for ramping temperature and pressure

1st cycle with 0.25 gm sample + 8 ml HNO_3					
	Temp(°C)	Pressure (bar)	Ramp time (min.)	Hold time (min.)	Power %)
1	195	30	15	45	90
2	50	30	5	10	0
2nd cycle with 2 ml HF					
4	185	30	15	45	90
5	50	0	0	0	0

Safety Precautions

- ✓ Always wear appropriate PPE.
- ✓ Handle acids with care, using appropriate fume hoods.
- ✓ Ensure proper ventilation in the working area.
- ✓ Be aware of the chemical properties and potential hazards of the materials being digested.

STANDARD OPERATING PROCEDURE OF CHNS/O ANALYZER

- ON the Helium gas from the Cylinder, after that switched ON the Instrument.
- Switched ON the computer, and open the Software Callidus 5.1.
- Go to Operator, then login (User Name and Password) click Ok.
- After that click on Instrument > Setting > Standby Auto ready > click on Out of Standby.
- Go to back Instrument Set > Load the Method > Click Open > Select CHNS > again Open > Send to Instrument> Click Ok.
- Now the Instrument comes under conditioning it takes 1:30hr, for achieving the temperature 980˚c.
- After the temperature achieved, Instrument status is changed conditioning to Special.
- Click on Instrument > Setting > Click ON to TCD.

For Calibration:

Before the sample analysis calibration is must require.

- For calibration you must take 2 bypass sample, and 6 standard samples, weights between (0**.5 - 1.5mg**)
- Go to Analysis > New Auto Run > Auto fill > select sample type, total no. of samples etc. > click Ok. Firstly, you will take 2 bypass samples.
- Again click on > Auto Fill > select sample type (STD) > give the name of standard sample find from the standard sample table > total no. of samples > Click Ok.
- After that select the calibration type always select linear calibration.
- Now click on save, give sample source name and sample type name. When you click the both names it shows below box & note down this auto run name. Click on Ok then Save.
- Load the samples one by one firstly load two bypass and after that 6 standard samples.
- If TCD signal is under minus or very higher you must auto zero the TCD signal, by the help of Auto Zero option shows on top of the software.
- Again check the Oxygen in ON, load samples, the click on Start.
- 1 single sample take more than 5min (320sec) for all four-element analysis.
- After the calibration finished it automatically saved on Sample manager.
- Go to sample manager & double click on auto run.

For Sample Analysis

After calibration you can analysis your samples.

- Go to Sampler manager > select calibration auto run file > right click on this file then click on Export Calibration.

- Now a table show on the display it shows firstly your calibration samples under that you can fill your samples their weights and click on save.
- Give the sample source name & sample type name. Click on Save.
- After that click on Start.
- When auto run finished it's saved on Sampler Manager you can find from that.
- If you want to analysis the another sample in the same auto run file you can go to Sampler manager click on the same auto run file, right click on this >Open for appending > add the samples > click on save & Start.

For Shut Down

- Firstly turned OFF the TCD.
- Then goes to Instrument > Setting > Click on Open > Select on Zero or Shut down method >send to instrument > Click Ok.
- After that goes to Standby auto ready > click on gas save.
- Now the instrument status changed conditioning to standby.
- OFF the Oxygen gas from the cylinder, Helium remains ON.
- It takes around of 3-4hr for cooling or comes on room temperature.
- Now you can shut down the Computer.