

GTN/CHE/DBT/SOP

1. OPERATION OF THE CONDUCTIVITY METER

1.1 Switch on the main supply and press the ON/OFF button of the conductivity meter to switch on the meter and switch off the meter.

1.2 For automatic temperature compensation (ATC). Simply attach the conductivity \TDS electrode to the meter ensure that the ATC mode has been selected in the setup menu The ATC indicator will light on the LCD.

1.3 Note: If the conductivity/TDS electrode is not properly attached to the meter or it has been damaged, the ATC indicator will blink and the temperature display will show "Ur".

1.4 Rinse the electrode with de-ionized or distilled water before use to remove any impurities adhering to the electrode body. Shake or air dry. To avoid contamination or dilution of your sample rinse the electrode with the small volume of your sample.

1.5 Press ON to switch on the meter.

1.6 Dip the electrode into the sample.

1.7 When dipping the electrode into the sample, take care to ensure that the liquid level is above its upper steel band. Stir the electrode gently in the sample to create a homogeneous sample.

1.8 Allow time for the reading to stabilize. Note the reading on the display

2. CALIBRATION OF THE CONDUCTIVITY METER

2.1 Before starting calibration makes sure that the instrument is in the correct measurement mode.

2.2 Wash the electrode with deionized water after and before use and Store it dry.

2.3 Change the buffer after one week or when required, and record the details.

2.4 Perform the calibration using a standard buffer of 1413 μ S and record the details.

2.5 Press the mode key to select conductivity mode the conductivity indicator appears in the upper right hand corner of the display.

2.6 Rinse the electrode thoroughly with deionized water or a rinsing solution do not wipe the probe this causes the electrostatic charge on the glass surface.

5.2.7 Dip the electrode into the calibration buffer, the end of the probe must be completely immersed into the sample. Stir the probe gently to homogenize the sample.

2.8 Press CAL/MEAS key to enter conductivity calibration mode. The CAL indicator will be shown. The primary display will show the measured reading while the smaller secondary display will indicate the conductivity standard buffer solution.

2.9 Press HOLD/ENTER key to confirm calibration. The meter is now calibrated to the current buffer.

2.10 Rinse the electrode with de-ionized water or a rinse solution and store it dry.

3 ELECTRODE CARE AND MAINTENANCE

3.1 Keep the conductivity electrode clean, the electrode twice, and gently swirl it while you take readings. For best accuracy soak a dry electrode for at least for 5 to 10 minutes.

3.2 Never scratch the bands with a hard substance.

3.3 Do not strike the electrode against any hard surface.

3.4 Do not immerse the electrode in an oily substance.

3.5 Clean the electrode thoroughly by stirring it in a mild detergent or isopropyl alcohol.

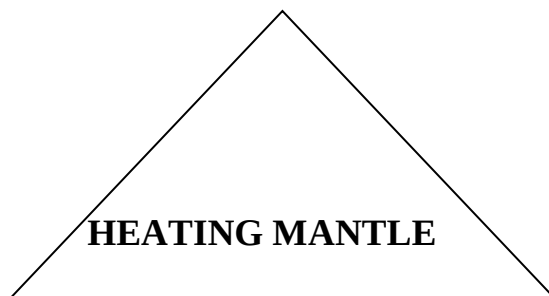
3.6 Wipe the electrode with soft tissue paper clean the electrode with tap water and recalibrate before use.



- 1. Fill the instrument with water and switch on**
- 2. Set the temperature of water as per requirement.**
- 3. Place the sample in water bath.**

HOT PLATE

1. Switch on the hot plate.
2. Set the temperature as per requirement.
3. Place the sample for heating.

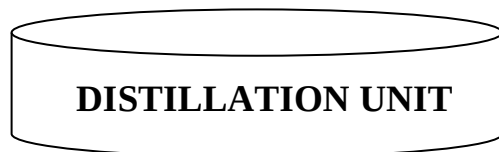


1. Switch on the heating mantle.
2. Set the temperature.
3. Place the sample in the round bottom flask.



SOXHLET APPRATUS

1. Assemble the apparatus.
2. Fill the round bottom flask with solvent (n-Hexane).
3. Put the thimble containing sample into extraction tube.
4. Attach the extraction tube with flask containing solvent.
5. Attach a condenser unit with the extraction tube and run the water.
6. Fix the soxhlet apparatus on hot plate and heat the flask containing solvent.
7. The solvent starts to evaporate and falls in the extraction tube after condensing.
8. Continue this process till all the fat is extracted.
9. Discontinue the process and take out the thimble.
10. Again attach the extraction tube with flask containing solvent along with fat and condenser unit.
11. Again heat the flask to recover the solvent.
12. Discontinue the process and clean the extraction tube and thimble.



1. Fill the round bottom flask of distillation unit with water
2. Attach it with condenser and start the water supply
3. Switch on the unit.
4. Collect the distilled water in container.

DIGITAL WEIGHING BALANCE

1. Switch on the balance
2. Wait till reading on scale is shown as zero
3. Place weighing materials on plate of balance
4. Either note the weighted materials or tare it off.



CLEVANGER APPARATUS

Clevenger apparatus is used for the determination of percentage of volatile oils present in the oil-bearing material. Take weighed quantity of material in a sample flask and add water. Generally the ratio of the material to water is 1:8. Connect the flask with Clevenger apparatus and open the tap of water for running the flow of water in a condenser. Start heating on a heating mantle and control the heat so that oil with water vapors comes into the graduated distillate receiving tube and excess water goes back into the flask. Continue heating for 8 hours, cool the assembly and remove the water from the distillate receiving tube, and take the oil into the rotary evaporator for removing the traces of water and cool the flask in a desiccators. After cooling, weight it accurately and calculate as follows:

Oil % =

$$\frac{\text{Weight of the oil} \times 100}{\text{Weight of the material taken}}$$

pH Meter

1. Pre-Operational Preparations:

- 1.1. Ensure that the pH meter is clean and free from any residue or contaminants from previous measurements.
- 1.2. Familiarize yourself with the specific operating instructions and technical specifications provided by the manufacturer for the particular model of the pH meter being used.
- 1.3. Gather all necessary equipment and materials.

2. Calibration:

- 2.1. Before starting any measurements, ensure that the pH meter is properly calibrated according to the manufacturer's instructions.
- 2.2. Prepare fresh buffer solutions of known pH values (pH 4, 7, and 10) using the appropriate stock solutions and following the recommended preparation procedures.
- 2.3. Immerse the pH electrode(s) in each buffer solution, allowing sufficient time for stabilization.
- 2.4. Adjust the pH meter reading to match the corresponding buffer pH value by following the calibration procedures provided by the manufacturer.
- 2.5. Document the calibration process and results as per the laboratory's standard operating procedures.

3. Measurement Procedure:

- 3.1. Rinse the pH electrode(s) with distilled or deionized water to remove any residual substances.
- 3.2. Gently blot the electrode(s) with a lint-free cloth to remove excess water without damaging the sensitive glass membrane.
- 3.3. Immerse the electrode(s) into the sample solution, ensuring proper immersion depth and avoiding contact with the container walls or other solid particles.
- 3.4. Allow the pH reading to stabilize on the meter display, typically indicated by a stable value or stable reading indicator on the meter.
- 3.5. Record the pH reading obtained from the sample solution.

4. Maintenance and Storage:

- 4.1. After each measurement, rinse the pH electrode(s) with distilled or deionized water to remove any residual sample.
- 4.2. If necessary, clean the electrode(s) using an appropriate electrode cleaning solution or procedure recommended by the manufacturer.
- 4.3. Store the pH electrode(s) in a suitable storage solution or container to maintain their performance and integrity.

COLUMN CHROMATOGRAPHY

Preparing for the SOP:

- **DON'T** use silica gel outside the fume cupboard. **DON'T** breathe in silica dust.
- **DON'T** use cracked and chipped glassware.

Before the procedure:

1. Choose eluent, columns size and volumes of fractions to be collected. Use a column with a sintered filter embedded at the bottom of it.
2. Close the tap at the bottom of the column and place the flash column in a fume cupboard where there is access to N₂ gas.
3. Make slurry of the silica and eluent in a large beaker, stirring well with a glass rod. Slowly pour the slurry into the column allowing it to run gently down the walls until a few cm below the lower end of the ground glass joint.
4. Apply air pressure using the fitted pump to fully compress the gel - you will notice when the upper edge of the dispersed silica stops moving downwards. It may be necessary to run more eluent through the column while applying a steady air pressure until all the gel is evenly wet with the eluent and no part of the column is hot. It is recommended to use a t-adaptor with a stopper on one end, which can be shot out to release excess gas if present, preventing the breakage of the column.
5. Excess eluent will continue to drip out of the column until the upper level of the silica gel is reached.

During the process:

- Application of the sample absorbed to stationary phase:
 - 1.1. Dissolve the sample in a suitable solvent in a suitably sized round bottom flask
 - 1.2. Add a small amount of dry silica (usually around the same mass as the crude mixture being purified).
 - 1.3. Evaporate the solvent. This process requires special attention and should be supervised by an experienced user.
 - 1.4. Scrape the silica mixture away from the sides of the flask with a bent spatula and apply the dry residue on top of the packed column.
- Eluting the column:

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1. Ensuring the tap is closed, fill the column with the eluent once more, allowing the solvent to run gently down the walls of the column so as not to disturb the top of the silica bed.
 2. Put a suitably sized beaker under the column, open the tap and apply N_2 pressure so that the liquid volume decreases with a rate of roughly 1 mm/s. It is recommended to use a T-adaptor with stopper on one end, which can be shot out to release excess gas if present, preventing the breakage of the column.
 3. Collect fractions with the recommended volume.
 4. Elute the column until the liquid is a few cm above the gel surface. Release the pressure and close the stopcock. Check the fractions. If all the wanted products have departed the column, the elution can be stopped. If not, the column can be refilled with more eluent, and the elution continued.
- Examining fractions and evaporation of pure fractions:
 1. Check the collected fractions using TLC
 2. Fractions pure on TLC with regard to the wanted product, are combined.
 3. If necessary, filter the solution to remove silica particles etc.
 4. Evaporate the solution in a weighed round bottom flask.

After the procedure:

1. Disassemble the column and dispose silica and TLC as silica waste.
2. Make sure you **leave the fume cupboard and working area clean.**
3. Wash glassware following

Disposal:

If any chemical waste is produced, ensure it is disposed of via the appropriate chemical waste route. All glassware should be cleaned, or disposed.

THIN LAYER CHROMATOGRAPHY

1. Using scissors or paper cutter, cut TLC plate to appropriate size and place on foil.
2. Using a ruler and pencil, draw a line 1 cm above and parallel to TLC plate bottom.
3. Using a glass Pasteur pipet and pipet bulb, add compound(s) to TLC plate.
4. Let all compounds applied to TLC plate dry completely.
5. Thoroughly wash TLC tank.
6. Make fresh TLC solvent and add to tank.
7. Cover tank opening with plastic wrap, followed by tank lid, and place lead weigh on top. Allow the tank to equilibrate for 5-10 minutes.
8. Place plate(s) in equilibrated TLC tank.
9. Let plate(s) sit in tank until solvent front reaches top of plate.
10. Remove plate and let dry completely on foil in chemical fume hood.
11. Place plate in TLC spraying area.
12. Thoroughly spray TLC plate with desired developer in TLC sprayer attached to compressed air line in chemical fume hood.
13. If necessary, heat TLC plate with heat gun to complete plate development.
14. Immediately scan developed TLC plate to preserve results.
15. Using a metal spatula, scrape desired silica from TLC plate and transfer to a clean 16 x 100 mm glass tube.
16. Add 8 to 10 ml of CHCl_3 - CH_3OH (2:1) to each tube and vortex vigorously.
17. Centrifuge at 3000 RPM, room temperature for 15 minutes.
18. Using a glass Pasteur pipet, transfer organic supernatant to a clean 16 x 100 mm glass tube and dry under a stream of N_2 .
19. Repeat steps 16 to 18 twice more.
20. Re-suspend extracted compound in a minimal amount of CHCl_3 - CH_3OH (2:1) and analyze by TLC.

POTENTIOMETER

1. Before connecting the mains keep the controls as under (a) SELECTOR switch to „O“. (b) STIRRER switch to „MIN“. (to its built-in „OFF“ position) (c) SET POINTER to about the middle of its full range.
2. Connect the mains supply (230 Volts 50 HZ) and switch “ON” the MAINS switch. The red pilot lamp will then light up and the display will show some reading near about to 0 mv.
3. Rotate the STIRRER control knob and adjust the stirrer speed.
4. Connect the pins of the sensors to the sockets provided on the machine on both sides of the plated rod. (Calomel to black socket) and lower the sensors with the spring clamp. So that the tips of the sensors are well immersed in the solution in the beaker. Care should be taken to see that the tips may not get smashed by the rotating magnetic rotor. Turn SELECTOR to „POT-G“ or „POT-M“ as the case may be. To „POT-G“ if Class/Calomel pair is used and to „POT-M“ for Metal/Calomel pair.
5. The display will now show some positive or negative reading depending on the nature and strength of the solution. Adjust it near to „O“ by SET POINTER knob. Note: The instrument is provided with „COARSE“ and „FINE“ SET POINTER controls. Fine control can be used to adjust the milli-volts reading precisely to 0 on the digital display. While coarse control is used for major change in the milli-volt reading. When “SELECTOR” is in „O“ or „O-R“ position, both FINE and COARSE “SET POINTER” will have no control on Digital display.
6. Add some drops of solution from the burette and note the reading on the digital display. Add fixed volume of solution from the burette (say 1ml, 0.5 ml OR 0.1 ml) every time and note the reading of “CHANGE IN MILLIVOLTS” for each addition.

It will be found that for the same addition every time the display will show larger and larger change in millivolts and at the END POINT, the change will be suddenly large. With further additions, the change will be progressively smaller and smaller. Thus the change in millivolts will be maximum at the END POINT.

The reading corresponds to the maximum change in millivolts should be considered for calculations.

A special feature of this instrument is that, one can bring the display reading back to 0 mv and add the further solution from the burette. Since only change in millivolts is to be noted after each addition the shifting of reading to 0 mv makes no difference.

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Thus by adjusting display reading to 0 mv with each addition highest accuracy can be obtained by skillful handling.

If the changes in millivolts are very small a graph of the change in millivolts “v/s” volume added may be drawn for accurate location of “END POINT”.

COLORIMETER

1. Insert plug in the socket.
2. Switch on the instrument at least 15 minutes before use.
3. Ensure to keep water blank before switching on the instrument.
4. Set the required wavelength.
5. Select %T using the knob and adjust to 100%.
6. Switch the knob to O.D which should be zero.
7. Clean the cuvettes after use. 8. Make an entry in the log book.

CENTRIFUGE

Procedure:

1. Turn ON the electrical power to the centrifuge.
2. Load the centrifuge tubes into the rotor.
3. Close the lid of the centrifuge.
4. Select the required time by adjusting the timer knob.
5. Select appropriate speed with the speed controller.
6. Wait next to the centrifuge until it has reached the desired speed and has not stopped due to it being imbalanced.
7. Wait until the centrifuge run has being completed.
8. Unscrew the rotor lid and remove the samples.
9. Switch off the instrument after use.

Spill Response Procedure:

1. In case of spillage (water/ solution/ buffers), mop with absorbent.
2. Then clean up with soap solution.
3. Report spillage in the log book.

Precautions: 1. Tubes are to be filed to the sample level with the same density fluid. 2. Ensure the tubes to be centrifuged are matched in pairs or evenly distributed around the rotor. 3. If centrifuge is vibrating a little too much, stop the rotor and wait until it has stopped spinning and redistribute the tubes to make sure the rotor is completely balanced. 4. Entry in log book is mandatory

UV CABINET

1. Ensure the area is clean.
2. Switch on the main power supply.
3. Switch on “VISIBLE LIGHT” marked switch.
4. Open the door.
5. Keep the TLC plate in the chamber.
6. Switch off “VISIBLE LIGHT”.
7. Close the door.
8. Switch on the required UV Light (i.e. Shortwave (254 nm), long wave (360 nm)).
9. Visualize the sample from outside the chamber through glass provided to visualize the sample inside the chamber.
10. After visualizing the sample note down the observation, switch off the UV light and remove the plate from the chamber.
11. Take care that any part of the body should not come in direct contact with UV light.

SPECTROPHOTOMETER

Photometric mode:

1. To connect spectrophotometer with software click on connects after connecting a green color indicator will be highlighted on the computer.
2. Select [Window] →[Photometric], it will be display UV Probe (system Administrator)- [Photometric].
3. Create the method enter the wavelength & add the parameter, select calibration and enter raw data and save the method.
4. For blank correction, take two clean cuvettes, after proper rinsing keep the blank solution prior to the measurement in the both cuvette and wipe them clean from the outside using tissue paper.
5. Place one cuvette in the reference cell holder and the other in the sample cell holder. Close the sample compartment.
6. Press „AUTO ZERO“ key. In case of multi wavelength press „Baseline key.
7. After Blank correction, keep the sample in sample compartment. Click on „Read Unk.“ to measure the absorbance.
8. After completion of measurement remove the cuvettes and wash them with purified water followed by acetone, so that the cuvettes get dried easily.
9. Click Report button, it will displayed report template of respective test.

Spectrum mode:

1. Select [Window] → [Spectrum], it will be display UV Probe (system Administrator) – [Spectrum].
2. To create the measurement file select method enter the wavelength range from start to end select scanning rate at medium speed then save the method file at desired path (If method is already exist then open the method and do the baseline correction and scan the sample).
3. For baseline correction take two clean cuvettes, After proper rinsing, keep the blank solution prior to the measurement in the both cuvettes and wipe them clean from the outside using tissue paper.
4. Place the cuvette in the sample cell holder. Close the sample compartment.
5. Click on baseline button, the “Baseline Parameters” file gets opened, select the wavelength range (nm) start to end. And click on „OK“ to measure the baseline.
6. Keep the standard or sample in sample compartment. Click on Start button for scanning, it will display the [New Data Set] , Save the data with respective name and path.
7. After scanning, Select [Operation] → [Peak Pick] to the data at each wavelength of the spectrum can be read.
8. Select Report button, it will displayed report template of respective test.