Laboratory manual

Analytical Instrumentation Laboratory

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**EXPERIMENT NO. 1.** To analyze the chemical structure of substance using a UV-VIS spectrophotometer.

**Aim:** To measure light transmitted through a sample solution.

**Theory**

Absorption spectroscopy relates the amount and type of radiant energy absorbed by a material to its structure, concentration, and identity. Electromagnetic radiation is usually viewed as a stream of discrete packets of energy called photons. According to quantum mechanics, atoms and molecules can occupy a limited series of energy states that correspond to distinct energy levels. To be absorbed, the energy of the incoming radiation must exactly match the difference between two of the substance energy levels. Since atoms and molecules have mostly unique energy configurations (electron configurations, vibrational/rotational modes, etc.), detection by absorption spectroscopy can be made specific.



**Beer's Law**

UV spectroscopy can be used to quantify the amount of an absorbing material present. The absorbance A of a solution is defined as:

A = -log10T = log10(Po/P) (1)

Where T, the transmittance, is the fraction of the incident radiation that is transmitted by the solution, P0 is the power of the incoming radiation, and P is the power of the attenuated radiation that has passed through the sample. Absorption is proportional to the path length b through a sample solution and the concentration c of an absorbing species according to Beer's Law.

A = ebc (2)

The constant of proportionality in Beer's Law, e, is the molar absorptivity and is expressed in units L mol-1 cm-1 when c is expressed in molarity and b in cm. To avoid reflection and scattering error by the sample holding cell and solvent, the power of the radiation beam transmitted by the solution is compared to the power of a beam transmitted by an identical cell containing only the solvent. Thus, Beer's Law is usually written as:

A = log10(Psolvent / Psolution) (3)

Beer's Law can be derived by considering that the fraction of the incoming beam absorbed is proportional to the probability of capture by the analyte in solution. The probability of capture is proportional to the surface area of absorbing particles which can then be related to the number, and concentration of particles. Using Beer's Law, the absorbance of a sample can be related to its concentration. Absorbances are additive, and Beer's Law can be used to determine the concentrations of a mixture of species, as long as they are not interacting. Beer's Law is limited in that it applies only to dilute solutions. In concentrated solutions (>0.01 M) particles interact altering the analyst's ability to absorb a certain wavelength of radiation which causes nonlinear deviations from Beer's Law. Changes in the refractive index of the solution and chemical reaction will also cause an error in applying Beer's Law to a sample. Finally, Beer's Law is only valid for monochromatic radiation. Thus, the most accurate result will be obtained by using a source able to produce intense radiation at a single wavelength.

**UV-Visible Spectrophotometers:** Spectrophotometers are made up of the stable source of radiant energy, a transparent sample container, a device for isolating specific wavelength, a radiation detector that converts transmitted radiation to a usable signal, and a signal processor and readout.

**Sources**
A radiation source for spectroscopy must generate a beam with sufficient power, wavelength range and stability for detectable and reproducible results. Many UV-Vis spectrophotometers such as the Cary 1-E, use a deuterium lamp for the UV range and switch to a tungsten filament lamp at 350 nm for the visible range. The electrical excitation of deuterium at low pressure results in a continuous spectrum of emitted radiation from 160 nm to the beginning of the visible (375 nm). An arc is formed between a heated, oxide-coated filament and a metal electrode. When about 40 Volts is applied to the heated filament, a direct current is produced resulting in an intense ball of radiation.

**Sample containers**

Sample containers (cells or cuvettes) must be constructed of a material that is transparent to radiation in the wavelength range of interest. Containers of quartz or fused silica are necessary for the UV range, and can be used into 700-3000 nm infra-red region. Glass and plastic can be used in the visible region as well. To minimize reflection cuvettes should have windows that are perfectly normal to the direction of the beam.

**Wavelength Selector, Radiation Detector**



|  |  |  |
| --- | --- | --- |
| **No.** | **Label** | **Description** |
| 1 | D2/QI lamp | Radiation Source |
| 2 | Grating 1 | A colored glass filter wheel that isolates a rough region of wavelengths around the desired wavelength. |
| 3 | Entrance slit | Isolates a thin beam of radiation coming from the filter wheel, limits the range of incidence angles. |
| 4 | Grating 2 | A holographic grating that disperses the radiation allowing a very precise selection of wavelengths. The grating is 30 x 35 mm in size and can move between wavelengths at 3000 nm/minute. |
| 5 | Exit slit | Radiation from the holographic grating is reflected by a mirror to the exit slit. The desired wavelength is selected by rotating the grating relative to the exit slit. Radiation of undesired wavelengths is absorbed by black matte walls surrounding the grating. |
| 6 | Chopper 1 | Radiation from the exit slit is directed by mirrors at the first chopper. Light hitting the black section is absorbed. Thus, the beam hits the sample and reference almost instantaneously. |
| 7 | Sample position | The beam is focused on the center of the sample compartment to allow maximum light throughput and reduce noise. |
| 8 | Chopper 2 | The second chopper ensures that the sample beam and reference beam hit the phototube detector at the same position and angle since the detector is sensitive to these quantities |
| 9 | Phototube | The phototube detector first corrects for any residual signal in the detector by zeroing itself during the moment when the black section of the choppers block the light beam.  |

**Interferences:**
The most obvious source of error in correlating UV-Vis absorption with DOC is from nonabsorbing organic material present in the sample. Simple sugars, aliphatic acids, alcohols, and amino acids, such as glycine, do not absorb in the UV range. However, these compounds are those most likely to be consumed by microbes as food and are not expected to be present in natural water samples from rivers and lakes at high concentrations.

**Calibration Procedure:**
A standard curve relating A254 to the concentration of KHP in ppm C was plotted as shown in Figure. A typical regression analysis was performed on the data resulting in the linear curve fit and equations as shown. The relevant parameters are displayed in the Results section. All errors are reported at the 95% confidence level.



[**Results**](http://www.civil.northwestern.edu/EHE/COURSES/eac/exp2/res2.htm)**:**

The light incident : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

The light transmitted : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Filter Selected : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Concentration in %age : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

**Experiment No. 2.** To analyze the concentration of elements in a liquid sample using an atomic absorption spectrometer.

**Aim of the experiment- To** quantitatively estimate the various elements in a sample solution.

## Therory

The Atomic absorption is an absorption process in which the amount of absorption of a reference emission beam by a ground state atomic vapor is measured and related to concentration. The emission beam is attenuated by atomic vapor absorption according to Beer's Law. The attenuated emission is focused directly on to a monochromator and the selected emission detected by a [photomultiplier tube](https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Supplemental_Modules_%28Analytical_Chemistry%29/Instrumental_Analysis/Spectrometer/Detectors/Photomultiplier_Tube%3A_Shockwave) (PMT). The phenomenon responsible for atomic absorption was first observed in 1802 with the discovery of the Fraunhofer lines in the sun's spectrum. Since that time, it has been recognized that the phenomenon of atomic absorption is responsible for self-absorption and self-reversal in emission methods of analysis. However, it was not until 1955 that Walsh proposed that atomic absorption could be used for quantitative analysis.

The design of the instrument is geared to produce the maximum number of neutral atoms of the element of interest in their ground electronic state. The traditional method of producing these atoms is a flame, a technique which is easily adapted to the analysis of liquid solutions. When a sample is introduced into the flame, the sample solution is dispersed into a fine spray, the spray is desolvated into salt particles, and these particles are then vaporized into neutral atoms, ionic species, and molecular species. These conversion processes occur in geometrically definable regions of the flame, and therefore it is important that the light path from the source lamp is directed through the region of the flame containing the maximum number of neutral atoms. The emission beam is produced by a high intensity light source, a hollow cathode discharge lamp in which the cathode is made of the same element which is to be determined. Therefore, this radiant energy has precisely the wavelength that is absorbable by the atoms in the flame, the absorption process being

Mg + hν → Mg∗

Mg is the atom of interest in the gas phase, Mg∗Mg∗, its excited state, and hνhν, the quantum of absorbable radiation. This method provides not only sensitivity but also selectivity since other elements in the sample generally do not absorb close enough to the chosen wavelength to interfere with the measurement. In order to reduce background interference, the wavelength of interest is isolated by a monochromator placed between the sample and the detector. The efficiency of production of free atoms depends on a great number of variables, some of which can easily be varied to optimize the sensitivity of the technique. Some of these variables are the oxidant-fuel composition used to produce the flame (e.g. air-acetylene, nitrous oxide-acetylene), the flame temperature, the sample feed rate, the design of the burner, and the solvent and other components in the sample matrix. In the following experiment, the role of these variables will be examined in sections E(a), E(b), and E(c), and atomic absorption spectroscopy used to determine Fe and Mn in plant tissue.

### Sample preparation

#### **Standard Solutions**

The method of serial dilutions is employed to eliminate unnecessary error when preparing standards from stock solutions. Prepare one solution containing both 100 ppm Fe and 100 ppm Mn from the 1000 ppm laboratory stock solutions provided in the lab. This is a 10x dilution. Using your freshly made 100 ppm stock, prepare a 10 ppm standard and finally from this 10 ppm standard prepare the following standards: 5 ppm, 2.5 ppm, 2 ppm, 1 ppm, and 0.5 ppm. Reserve the 100-ppm solution for the standard addition technique. You need 10ml of the 100, 5.0, 2.5, 2.0, and 0.5 ppm solutions, 50 ml of the 1 ppm solution, and 50 ml of the 10 ppm solution. Make your dilutions accordingly.

#### **B. Digestion of Unknowns**

Carefully weigh out three samples (~2 g each) of the plant tissue and note the exact weight. The analysis will be performed on each sample.

Place the weighed portion of the plant material in a 250 mL clean Erlenmeyer flask. Now add approximately 30 mL of conc. HNO3 acid and gently heat to just below boiling by adjusting the temperature on the hotplate. Typically a setting of 3 or 4 is sufficient. When the volume is diminished, place a stemless glass funnel upside down over the mouth of the Erlenmeyer flask to act as a condenser. This will prevent over-drying and possible charring and degradation of your sample. After ~30 minutes most oxidation will have ceased. Cool to room temperature and add ~10 ml of 30% H2O2. Again, gently heat to just below boiling. When most oxidation has stopped, boil the mixture down to near dryness using a hot plate. Watch the mixture carefully as it approaches dryness to prevent burning. Let it cool before transferring it to a 100-ml volumetric flask. Dilute the solution to the mark with DI/Degassed water. After the sample is made, filter it into a clean flask or bottle. The filtered solution is the one to be analyzed. Digestion of the sample with HNO3 and H2O2 breaks down the organic material in the plant tissue which complexes the iron. This must be done to get free Fe3+ in solution.

### Procedure

* Make sure the AA power switch has been turned on before starting the software on the computer.
* Doubleclick the SpectrAA icon on the desktop.
* Click on the Worksheet button.
* Click on the "New From…." button. Select the worksheet "Chem 105 AA.AAWS," give it a unique name, and fill in the Analyst and Comment entries.

The worksheet opens to the Analysis page, since this method has been previously developed for this application. Note the different windows - the calibration entries are in the lower right hand corner, and the sample entries are in the upper middle. You can click on the Fe or Mn boxes in the sample entries to see the analysis parameters for each of these elements. Note that the analysis wavelength has been chosen based on the element, but it is possible to change to a different emission line if necessary. After having the lab instructor check the system, light the flame by pressing the ignition button and holding it until the flame ignites. The ignition button is the one with the drawing of a flame next to it. Remove the aspiration tube from the distilled water and note the change in the flame sound. If there is no discernible change in the sound, the tube may be blocked, and must be cleared. Place the tube back in the distilled water. Click the Start button on the worksheet. A dialogue box will appear telling you to prepare for instrument zero. Make sure the aspiration tube is in deionized water, and press OK or Enter. In a few moments, a dialog box will appear for Calibration Zero. Continue aspirating the deionized water and press OK or Enter.The next dialog box to appear will ask you to Present Standard Solution 1, which will be your 0.5 ppm standard. Place the aspiration tube into the standard, and click OK. Do not remove the aspiration tube from the standard until the measurement is complete and the next dialog box appears.

Continue through the standards, until a complete calibration curve has been generated for Mn. At this time, the software will ask you to Present Solution Sample 1. Do the three samples you prepared as the dialog boxes prompt you. After these are complete, the same sequence will be repeated, but this time the wavelength will be changed to 248.3 to measure the Fe.

After all standards and samples are completed for the Fe analyses, the flame will be automatically extinguished and the computer will begin playing a happy little tune. When you get sick of the happy little tune, click OK in the dialog box. Save all of your results with the menu item File…Save. Go to the Reports page using the menu bar item Window…Reports.

Click on your file name, even if it's already been highlighted. Then click on the Report tab. Click on the "Print Report" button, and your data will be sent to the network printer.

## Sample analysis

### *A. Determination of the linear working ranges for Fe and Mn*

Using the procedure above, sequentially measure all six standard solutions. Record all concentrations and their absorbance values. From this data determine the linear working range of each of the two elements. These will be your standard calibration curves for Fe and Mn.

### *B. Determination of Fe and Mn levels in dried plant tissue*

Atomic absorption analyses of trace elements in plant tissue are now routinely carried out by soil and plant tissue laboratories. The analysis of plant tissue usually leaves, provides a better guide to plant fertilizer requirements then that provided by soil analysis. In this experiment, we shall analyze sugar beet leaves for their Fe and Mn content.

Obtain the absorbance for each of the three digested leaf samples previously prepared and report the final corrected results in ppm Fe and Mn in the original dried leaf sample. Finally, use the method of standard additions to obtain a more accurate result. Read the absorbance resulting after each addition to check that the increase in absorbance is that expected. Read section 1E2 of your textbook (pp 15-17) for a discussion on the Method of Standard Additions (remember to spike your unknown with a concentration that lies within the linear working range). Use only three additions for each of your three samples and test only for iron.

The following should also be included in the lab report.

1. clean and ordered tabulated absorption data for the standards, unknowns and spiked unknowns,
2. Standard Calibration Curves for both Mn and Fe with all three unknown absorbances marked and
3. Standard Addition curves for all three samples. Additionally, denote the working range of your standard calibration curve and include the least-squares fit for your working calibration curve.

**Experiment No. 3.** To test the purity of a substance, and separating the different components of a mixture using a gas chromatograph.

**Aim of the experiment –** The estimate the constituents of a sample solution.

**Theory**

A schematic outline of a typical instrument is shown below. When a sample is injected into the correct column, a carrier gas sweeps the sample through the column. If necessary, an oven heats the system to vaporize the sample and speed its passage through the column. The different components of the sample will be separated by the column because of each of the components “sticks” to the liquid coating that on the column packing differently. The greater the “stickiness,” the longer it takes for a substance to pass through the column. When a substance leaves the column, it is sensed by a detector. The detector generates a voltage that is proportional to the amount of the substance. The signal from the detector is then displayed by a chart recorder and/or fed into a computer. Modern gas chromatographs are connected to a computer which displays the peaks of all the substances in the sample. This is called the chromatogram. Software can perform all the calculations you will do in this experiment. An example of an analysis for cholesterol esters is shown above. If it could be done at all, this separation would take weeks by traditional wet chemistry. Here it took 21 min for the analysis and a few hours to prepare the sample for injection into the chromatograph. However, so that you can understand how the computer does its analysis, we will supply you with peaks drawn by a chart recorder and you will perform all the measurements and calculations. The kind of signal displayed by a chart recorder is more-or-less a triangular-shaped peak. This is because the detector signal causes a vertical deflection of the recorder pen at the same time that the chart paper is moving under the pen. The time that it takes a substance to pass through the instrument from injection to detection is called the **retention time, tr**. The retention time, tr, is measured from the **injection point** to the **peak height**. The peak height is the highest point of the peak and is the only reproducible point on the peak. Since the chart paper moves at a constant speed, the box divisions are proportional to tr and you can measure tr in box divisions for this experiment.

The amount of substance in a sample is proportional to the **area under the peak** of that substance. However, the proportionality constant is different for each substance and detector. Therefore, to do quantitative analysis by gas chromatography, you must first determine the proportionality constant for each substance in the sample. You will do this by constructing calibration lines as described below. There is another piece of information that you need in order to do quantitative GC analysis. This is the **attenuation (attn)**. Since most signals from the detector are too large for the recorder mechanism to handle, there is a switch on the chromatograph that attenuates or reduces the size of the signal. For example, if the attenuation of a peak is 512, the signal has been reduced 512 times. So you must multiply the area under the peak (the area that you measure on the chart) by 512 to get the true area that is proportional to the original signal. Thus, it is **area x attn** that is proportional to the amount of substance injected.



There are two kinds of information available on the recordings in the packet.

1. The retention times of each substance as determined from the distance in box units between the injection point and peak of each curve. As stated above, we will use box units for retention times because they are proportional to the time and serve us just as well.
2. The peak areas corresponding to the injection of different amounts of each pure substance. You are given several peaks (usually 7 or 8) for each pure compound made by varying the quantity (in microliters, µL) injected, and the attenuation setting for each peak.

You are to plot a calibration line for each substance using Vernier’s Graphical Analysis program. You will then perform a linear regression on each line to determine the slope and y-intercept of the line. After the calibration lines are plotted and approved by the instructor, you will be given the chromatogram of an unknown mixture to identify. By determining the retention time of each peak of the unknown, you can identify the substance that the peak corresponds to. By measuring the area x attn of each peak, you can use the formula of a straight line (y = mx + b) from the calibration line to determine how much of each substance was injected. Determining Retention Times and Preparing Calibration Graphs.

On the chromatogram of all the pure substances (the last page of the packet you will be given in lab), measure the retention time of each peak in box units, to the nearest 0.1 box units, and record it directly on the graph near the peak. For each peak, the retention time is measured from the injection point to the top of the peak as shown below. Transfer the values to Data Table – Retention Times.

Open Graphical Analysis. Using Graphical Analysis, you will plot the area x attn on the vertical axis (the yaxis) and the **microliters (** µ**L)** injected on the horizontal axis (the x-axis). **A separate graph will be prepared for each substance.**  You will end up with four separate graphs containing four straight lines, one for each substance. These will be your calibration lines.

t

r

start

# Using Graphical Analysis to Plot Your Data

1. Enter the data into the Data Table Window. You can move from one cell to another by hitting the Enter button on your keyboard, or by using the mouse to click on the cell. Microliter (µL) values are entered in the column labeled **x**. Area x attn values are entered in the column labeled **y**. Since the area x attn numbers are very large, they should all be converted to scientific notation and then changed so that all the numbers have the same exponent. Choose this one exponent so that all the number parts are larger than 1 but otherwise as small as possible. Then you can just plot the number part (the abcissa) and note the exponent (the ordinate) on the y-axis label. Suppose two of your numbers are 35,600 = 3.56 x 104 and 274,000 = 2.74 x 105. For plotting purposes, you would write them as 3.56 x 104 and 27.4 x 104 and only plot 3.56 and 27.4.
2. Click on the graph’s title and change the title to the name of the substance plotted. For example, the first graph will probably be called “Benzene” or even better, “Calibration Line for Benzene.” At this point, you should sort the data so it is listed from the smallest volume to the largest volume. This is so the entries in the Data Table Window are easy to read. Do this as follows: Make the Data Table Window active by clicking on it. Then Data  Sort Data  Volume  OK.
3. Double click on the **x** in the Data Table Window and change the axis label. Label the x-axis **volume injected** with units of **µL**. Now label the y-axis by double clicking on the **y** in the Data Table Window. For the numbers shown in #1 above, you would label the y-axis **area x attn x E–4** in units of **boxes.**  (You can’t enter exponents in a graph title or an axis label in this program, so use E–4 to mean 10–4. The E stands for exponent, in this case 10.) The reason the exponent is 10–4 and not 10+4 is that you multiplied the original numbers by 10–4 to get the numbers written on the y-axis. This is the convention that is used in scientific journals.
4. Make the graph the active window by clicking any place in the graph. Remove the connecting line on the graph by un-checking Connecting Lines in the Graph menu. Perform a linear regression on each graph in the following way: In the Analyze menu, select Curve Fit. Choose Linear in the resulting window, and then click try fit and then OK. Move the regression box on the graph away from the line so the line and the points are not covered. Do this by placing the cursor anyplace in the regression box, and while holding down the left mouse button, move the mouse to reposition the box. A typical calibration graph for benzene is shown above.
5. Print each data table and calibration graph in the following way: First fill in the Text Box on the screen with your name, course, section, name of lab experiment, and date. Generally, the landscape orientation is preferable to portrait. Landscape can be selected in page setup menu under file. Choose Printer Setup in the File menu. Choose the correct printer for your lab as directed by your instructor. Click OK. Then choose Print in the File menu.
6. Saving your data: Insert a formatted 3 ½ in. floppy disk into the a: drive. In the File menu, click on Save As. In the Save Data As window, and then in the Filename: box, erase any existing file name and type in your file name, such as **GC benzene**. Then click OK. You have now saved the graph for benzene. Save the 3 other graphs in a similar way.

Show your calibration graphs to your instructor who will then give you a GC recording of an unknown.

# Analyzing Your Unknown

Determine the **retention time** in box units of each peak in the unknown. Identify each substance present by comparison with the retention times of the substances contained in the chromatogram of all the pure substances. Enter the retention times in the Data Table—Retention Times opposite the substances you have found in your unknown.

To find out how many **microliters** of each substance was injected, let’s first look at the calibration graph of benzene on page A-5. Assume that an unknown contains benzene for this example. From the calibration graph for benzene, the equation for the calibration line (from the regression box) is

# y = Mx + B y = 11.5 x + 3.45

where **x** represents the number of microliters of benzene and **y** represents the area x attn for the benzene curve. Since all the y-axis values on the graph had been multiplied by 10–4, the slope and intercept now need to be multiplied by 10+4 to get back to the original numbers. Thus the equation you will use to find the number of microliters of benzene is

# y = 11.5 x 104 x + 3.45 x 10+4

Suppose that the area x attn that you measured for benzene on the unknown is 197,000 boxes. This value is **y** in the above equation. To find the number of microliters of benzene injected, solve for **x**. Now, **for your unknown**, measure the area under each curve as was done for the pure substances. Multiply **area x attn** for each peak and use the appropriate calibration curve to determine the number of microliters of each pure substance that is present in the unknown sample. The sum of the microliters of all substances in your unknown is the number of microliters of the unknown sample that was injected into the gas chromatograph. Find the percent of ***each*** substance in your unknown by using the following formula:

µL of one unknown x 100 = % of the unknown µL total injected

Enter your percents in the Results Table.

**Experiment No. 4.** To detect cracks in pipelines using an Ultrasonic Flaw Detector

**Aim of the experiment –** To estimate the cracks in pipelines.

**Theory –** In the broadest sense, a transducer is a device that converts energy from one form to another. Ultrasonic transducers convert electrical energy into high-frequency sound energy and vice versa.



Typical transducers for ultrasonic flaw detection utilize an active element made of a piezoelectric ceramic, composite, or polymer. When this element is excited by a high voltage electrical pulse, it vibrates across a specific spectrum of frequencies and generates a burst of sound waves. When it is vibrated by an incoming sound wave, it generates an electrical pulse. The front surface of the element is usually covered by a wear plate that protects it from damage, and the back surface is bonded to backing material that mechanically dampens vibrations once the sound generation process is complete. Because sound energy at ultrasonic frequencies does not travel efficiently through gasses, a thin layer of coupling liquid or gel is normally used between the transducer and the test piece. There are five types of **ultrasonic transducers** commonly used in flaw detection applications.

- **Contact Transducers** -- As the name implies, contact transducers are used in direct contact with the test piece. They introduce sound energy perpendicular to the surface, and are typically used for locating voids, porosity, and cracks or delaminations parallel to the outside surface of a part, as well as for measuring thickness.
- **Angle Beam Transducers** -- Angle beam transducers are used in conjunction with plastic or epoxy wedges (angle beams) to introduce shear waves or longitudinal waves into a test piece at a designated angle with respect to the surface. They are commonly used in weld inspection.
- **Delay Line Transducers** - Delay line transducers incorporate a short plastic waveguide or delay line between the active element and the test piece. They are used to improve near surface resolution and also in high temperature testing, where the delay line protects the active element from thermal damage.
- **Immersion Transducers** - Immersion transducers are designed to couple sound energy into the test piece through a water column or water bath. They are used in automated scanning applications and also in situations where a sharply focused beam is needed to improve flaw resolution.
- **Dual Element Transducers** - Dual element transducers utilize separate transmitter and receiver elements in a single assembly. They are often used in applications involving rough surfaces, coarse grained materials, detection of pitting or porosity, and they offer good high temperature tolerance as well.
Modern **ultrasonic flaw detectors** such as the **EPOCH series** are small, portable, microprocessor-based instruments suitable for both shop and field use. They generate and display an ultrasonic waveform that is interpreted by a trained operator, often with the aid of analysis software, to locate and categorize flaws in test pieces. They will typically include an ultrasonic pulser/receiver, hardware and software for signal capture and analysis, a waveform display, and a data logging module. While some analog-based flaw detectors are still manufactured, most contemporary instruments use digital signal processing for improved stability and precision.

The pulser/receiver section is the ultrasonic front end of the flaw detector. It provides an excitation pulse to drive the transducer, and amplification and filtering for the returning echoes. Pulse amplitude, shape, and damping can be controlled to optimize transducer performance, and receiver gain and bandwidth can be adjusted to optimize signal-to-noise ratios. Modern flaw detectors typically capture a waveform digitally and then perform various measurement and analysis function on it. A clock or timer will be used to synchronize transducer pulses and provide distance calibration. Signal processing may be as simple as generation of a waveform display that shows signal amplitude versus time on a calibrated scale, or as complex as sophisticated digital processing algorithms that incorporate distance/amplitude correction and trigonometric calculations for angled sound paths. Alarm gates are often employed to monitor signal levels at selected points in the wave train to flag echoes from flaws.
The display may be a CRT, a liquid crystal, or an electroluminescent display. The screen will typically be calibrated in units of depth or distance. Multicolor displays can be used to provide interpretive assistance.
Internal data loggers can be used to record full waveform and setup information associated with each test, if required for documentation purposes, or selected information like echo amplitude, depth or distance readings, or presence or absence of alarm conditions.

**Procedure**
Ultrasonic flaw detection is basically a comparative technique. Using appropriate reference standards along with a knowledge of sound wave propagation and generally accepted test procedures, a trained operator identifies specific echo patterns corresponding to the echo response from good parts and from representative flaws. The echo pattern from an test piece may then be compared to the patterns from these calibration standards to determine its condition.

- Straight Beam Testing -- Straight beam testing utilizing contact, delay line, dual element, or immersion transducers is generally employed to find cracks or delaminations parallel to the surface of the test piece, as well as voids and porosity. It utilizes the basic principle that sound energy traveling through a medium will continue to propagate until it either disperses or reflects off a boundary with another material, such as the air surrounding a far wall or found inside a crack. In this type of test, the operator couples the transducer to the test piece and locates the echo returning from the far wall of the test piece, and then looks for any echoes that arrive ahead of that backwall echo, discounting grain scatter noise if present. An acoustically significant echo that precedes the backwall echo implies the presence of a laminar crack or void. Through further analysis, the depth, size, and shape of the structure producing the reflection can be determined.



Sound energy will travel to the far side of a part, but reflect earlier if a laminar crack or similar discontinuity is presented. In some specialized cases, testing is performed in a through transmission mode, where sound energy travels between two transducers placed on opposite sides of the test piece. If a large flaw is present in the sound path, the beam will be obstructed and the sound pulse will not reach the receiver.
- Angle Beam Testing - Cracks or other discontinuities perpendicular to the surface of a test piece, or tilted with respect to that surface, are usually invisible with straight beam test techniques because of their orientation with respect to the sound beam. Such defects can occur in welds, in structural metal parts, and many other critical components. To find them, angle beam techniques are used, employing either common angle beam (wedge) transducer assemblies or immersion transducers aligned so as to direct sound energy into the test piece at a selected angle. The use of angle beam testing is especially common in weld inspection.
Typical angle beam assemblies make use of mode conversion and Snell's Law to generate a shear wave at a selected angle (most commonly 30, 45, 60, or 70 degrees) in the test piece. As the angle of an incident longitudinal wave with respect to a surface increases, an increasing portion of the sound energy is converted to a shear wave in the second material, and if the angle is high enough, all of the energy in the second material will be in the form of shear waves. There are two advantages to designing common angle beams to take advantage of this mode conversion phenomenon. First, energy transfer is more efficient at the incident angles that generate shear waves in steel and similar materials. Second, minimum flaw size resolution is improved through the use of shear waves, since at a given frequency, the wavelength of a shear wave is approximately 60% the wavelength of a comparable longitudinal wave.



**

The angled sound beam is highly sensitive to cracks perpendicular to the far surface of the test piece (first leg test) or, after bouncing off the far side, to cracks perpendicular to the coupling surface (second leg test).

**Experiment No. 5.** To measure the concentration of Na+, Li+, and K+ ions in a given sample solution using a flame photometer.

**Aim of the experiment** – To measure the concentration of alkali metal ions in a given sample solution.

**Objectives**

After studying and performing this experiment you should be able to:

• explain the principle of flame photometric method,

• explain the principle of internal method,

• state the significance of internal standard method,

• compare the internal standard method with the standard solution method used in some of the earlier experiments,

• perform a flame photometric determination, and

• determine the concentration of sodium and potassium ions in a given sample.

**Theory:**

The compounds of the alkali and alkaline earth metals (Group II) dissociate into atoms when introduced into the flame. Some of these atoms further get excited to even higher levels. But these atoms are not stable at higher levels. Hence, these atoms emit radiations when returning back to the ground state. These radiations generally lie in the visible region of the spectrum. Each of the alkali and alkaline earth metals has a specific wavelength. This experiment is based on flame photometric method‒an atomic emission spectrometric method. It is a simple, rapid and inexpensive method for routine analysis of alkali and alkaline earth metals like, sodium, potassium, lithium, calcium and barium in environmental, clinical and biological samples especially in biological fluids and tissues. The convenience, speed, and relative freedom from interferences have made flame photometry a method of choice for the determination of these elements, which are otherwise difficult to determine. In the process of determination of sodium and potassium ions in a solution, you would be learning about a very important determination of biological samples. You may know that sodium ion is the major cation of the extracellular fluid whereas potassium is the major ion found inside the cells. The body maintains a delicate balance of these ions across the cellular membrane and any alteration in their normal values has significant physiological consequences. For example, an abnormal increase of potassium (hyperkalemia) or decrease of potassium (hypokalemia) can significantly affect the nervous system and heart, and if the levels become extreme, it can be fatal. In other words, an accurate determination of these ions in the body fluids can serve as an important diagnostic tool.

## ****Parts of flame photometer****

A simple flame photometer consists of the following basic components:

**Source of flame:** A Burner in the flame photometer is the source of flame. It can be maintained in at a constant temperature. The temperature of the flame is one of the critical factors in flame photometry.

| **Fuel-Oxidant mixture** | **Temperature (°C)** |
| --- | --- |
| Natural gas-Air | 1700 |
| Propane-Air | 1800 |
| Hydrogen-Air | 2000 |
| Hydrogen-Oxygen | 2650 |
| Acetylene-Air | 2300 |
| Acetylene-Oxyen | 3200 |
| Acetylene-Nitrous oxide | 2700 |
| Cyanogen-Oxygen | 4800 |

**Nebuliser:** Nebuliser is used to send homogeneous solution into the flame at a balanced rate.

**Optical system:**The optical system consists of convex mirror and convex lens. The convex mirror transmits the light emitted from the atoms. Convex mirror also helps to focus the emissions to the lens. The lens helps to focus the light on a point or slit.

**Procedure**

* Both the standard stock solution and sample solution are prepared in fresh distilled water.
* The flame of the photometer is calibrated by adjusting the air and gas. Then the flame is allowed to stabilize for about 5 min.
* Now the instrument is switched on and the lids of the filter chamber are opened to insert appropriate colour filters.
* The readings of the galvanometer are adjusted to zero by spraying distilled water into the flame.
* The sensitivity is adjusted by spraying the most concentrated standard working solution into the flame. Now the full scale deflection of the galvanometer is recorded.
* Again distilled water is sprayed into the flame to attain constant readings of galvanometer. Then the galvanometer is readjusted to zero.
* Now each of the standard working solutions is sprayed into the flame for three times and the readings of galvanometer are recorded. After each spray, the apparatus must be thoroughly washed.
* Finally sample solution is sprayed into the flame for three times and the readings of galvanometer are recorded. After each spray, the apparatus must be thoroughly washed.
* Calculate the mean of the galvanometer reading.
* Plot the graph of concentration against the galvanometer reading to find out the concentration of the element in the sample.

| **Element** | **Emitted wavelength** | **Flame color** |
| --- | --- | --- |
| Sodium | 589 nm | Yellow |
| Potassium | 766 nm | Violet |
| Barium | 554 nm | Lime green |
| Calcium | 622 nm | Orange |
| Lithium | 670 nm | Red |

 **Results:**

The concentration of Na+ : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

The concentration of K+ : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

The concentration of Ba2+ : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

The concentration of Ca2+ : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

The concentration of Li+ : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

**Experiment No. 6.** To measure the pH value of a given sample solution using a pH meter.

**Aim of the Experiment –**

•To measure the pH of various solutions using pH indicators and meter.

•To determine the value of KaKa for an unknown acid.

•To perform a pH titration (OPTIONAL, if time permits)

•To create and study the properties of buffer solutions.

**Theory**

*Part A: Using Indicators to Measure pH*

In this part of the experiment, you will use five indicators to determine the pH of four solutions to within one pH unit. An **acid-base indicator** is a chemical species that changes color at a specific pH as the pH (acidity) of the solution is varied. Acid-base indicators are themselves weak acids where the color of the aqueous acid is different than the color of the corresponding conjugate base. In this hypothetical example InIn stands for the indicator. As you can see from Equation [5.1](https://chem.libretexts.org/Bookshelves/Ancillary_Materials/Laboratory_Experiments/Wet_Lab_Experiments/General_Chemistry_Labs/Online_Chemistry_Lab_Manual/Chem_12_Experiments/05%3A_pH_Measurement_and_Its_Applications_%28Experiment%29#mjx-eqn-1)5.1, the protonated form of the acid-base indicator, HInHIn (*aq*), will be one color (yellow in this example) and the deprotonated form, In−In− (*aq*), will be another color (blue in this example). The equilibrium-constant expression is:

Kai = [H3O+] [In−] [HIn]

The five indicators you will use in this experiment, their color transitions, and their respective values of pKaipKai are given in Table.



We can use the values in Table to determine the approximate pH of a solution. For example, suppose we have a solution in which methyl violet is violet. This tells us that the pH of our unknown solution is greater than or equal to 2 because methyl violet turns violet at pH values of 2 or greater. Now suppose we add some congo red to a fresh sample of our solution and find that the color is violet. This tells us that the pH of our solution is less than or equal to 3 because congo red turns violet at pH values of 3 or less. From these two tests we know that the pH range our solution is between 2 and 3. Thus, we have determined the pH of our solution to within one pH unit. Proceeding in a similar manner, you will use the acid-base indicators in Table 1 to determine the pH range of four solutions to within one pH unit.

*Part B: Using pH Meters*

In this part of the experiment you will learn to use a pH meter to measure pH. Your instructor will demonstrate how to use the pH meter appropriately at the beginning of your laboratory session.

**Requirements**

Apparatus pH meter 1 No.

Glass electrode - reference electrode assembly 1 No.

Basic Experiments Beakers 100 cm3 capacity 3 No.

Buffer solution of pH 4: It is prepared by dissolving buffer tablet of pH 4 in a 100 cm3 volumetric flask and diluting it up to the mar "I with distilled water. Alternatively, it may be prepared by dissolving 1.021 g of potassium hydrogen phthalate (C8H5O4K) in distilled water in a 100 cm3 volumetric flask and making the solution up to the mark.

Buffer solution of pH 7: Dissoh buffer tablet of pH 7 in distilled water in a 100 cm3 volumetric flask and make up the, solution up to the mark. Alternatively, dissolve 0.340 g of potassium dihydrogen phosphate (KH2P04) and 0.3550 g of disodium hydrogen phosphate (Na2HP04) in distilled water in a 100 cm3 volumetric flask and make up the solution to the mark.

Buffer solution of pH 9.18: Dissolve a buffer tablet of pH 9.18 in distilled water in a 100 cm3 volumetric flash and make up the solution up to the mark. Alternatively, dissolve 1.906 g of borax in distilled water in a 100 cm3 volumetric flask and make up the solution up to the mark.

**Principle**

Hydrogen ions in solution, like other ionic species, conduct an electric current. When a glass electrode is dipped in a solution containing hydrogen ions, a potential difference develops across a very thin glass membrane separating two solutions of different hydrogen ion concentrations - one within and the other outside the glass electrode. A pH meter is an electronic voltmeter that measures this difference of potential and through its internal calibration, converts it to a pH reading which is displayed on a scale. The scale is normally taken extending from 0 to 14 pH units with a least count of 0.05 pH units or better. A pointing needle moves across the graduated scale and the pH of the solution can be read directly on the scale. These days digital pH meters are becoming more popular as compared to scale - needle instruments.

**Procedure**

1. Set the selector switch to 'zero' position and adjust the zero position by a, screw driver if the pointer does not indicate zero.
2. Mount the electrode assembly - a glass electrode and a saturated calomel electrode or a combination electrode in the clip on the stand. Wash the electrodes well with distilled water.
3. Connect the power cable to a 220 V AC supply. Switch on the instrument and wait for a few minutes till the instrument warms up.
4. Take about 20 cm3 of the standard buffer solution of pH 7.0 in a beaker and lower the electrode assembly into it. It should be ascertained that the glass electrode membrane is completely immersed in the solution. The electrodes should not touch each other or the sides or the bottom of the beaker. Swirl solution in the region of the glass electrode surface gently so as to bring it into pH equilibrium.
5. Measure the temperature of the solution and adjust the 'temperature I compensation knob' to this temperature.
6. Put the selector switch to a suitable pH range (0-7 for acidic or 7-14 for basic solutions) and adjust 'set buffer knob' in a manner that the pointer reads the pH of the standard buffer solution placed in the beaker, i.e., pH 7.0.
7. Put the selector switch back to the 'zero' position. Remove the electrodes from the buffer solution. Wash the electrodes with distilled water and wipe them dry with a tissue paper. Transfer the standard buffer to the storage bottle.
8. You will need to make an additional adjustment when precise pH values are rcquired in the acid or basic range. To do this, select the pH 4.0 buffer for an acid reading or the pH 9.18 buffer for a basic reading.
9. Take about 20 cm3 of the second buffer solution (of pH 4.0 or 9.18) in another beaker and place the electrode assembly into it. Set the selector switch in the suitable pH range position (0-7 or 7- 14) and read the pH of the solution on the scale. If the meter reading does not agree exactly . with the known pH of the buffer, set the 'slope' control until the required reading is obtained. The instrument is now ready for measuring the pH of the unknown solution.
10. Put the selector switch back to zero position and remove the electrodes from the buffer solution. Wash the electrodes with distilled water and wipe them gently with tissue paper. Transfer the buffer solution to the storage bottle.
11. Take about 20 cm3 of unknown solution in another beaker. Introduce the electrodes into the solution and swirl the solution gently. Put the selector switchback in the suitable range position and read the pH of the solution on the scale.
12. Put the selector switch back to zero position. Remove the electrodes from the solution and keep the electrodes in distilled water when not in use.

**Precautions**

(i) Never touch the membrane of the glass electrode with anything else except soft tissue paper since it is fragile and is easily ruined if scratched

or bumped.

(ii) The electrodes must not be removed from the solution unless the selector switch is at zero.

(iii) Never dip the glass electrode in a solution with a dehydrating action.

(iv) If used for measuring pH of albuminous substances, the glass electrode must be cleaned with suitable solvents and then the electrode be placed

in distilled water for a few hours before it is used to measure the pH of the other solution.

(v) For basic solutions with pH more than 11, glass electrodes of special composition are required to avoid interference due to sodium ion.

(vi) The glass electrode may be covered with a sleeve to save it from jerks.

**Experiment No. 7.** To measure the conductivity of a sample solution using a conductivity meter.

**Aim of the experiment-** To measure conductivity od a sample solution.

**Requirements –**

Conductivity meter 1

Conductivity cell 1

Wash bottle 1

Thermometer 1

**Theory –**Measurement of conductance is potentially a very sensitive method for measuring ionic concentrations. It has many applications in chemical analysis. Measurement of conductance is one of the best ways for checking and monitoring the purity of water. A continuous recording of conductance is helpful in monitoring the purity of water purified by ion-exchange or obtained from large distillation units. Moisture content of soils can also be determined in the fields using portable instruments. Conductometric measurements are also used to ascertain the end-point in many titrations, e.g., acid- base, precipitation and complexometric titrations. These are also used in . determining equilibrium constants, e.g., ionisation constants of weak electrolytes, solubility products of sparingly soluble salts, hydrolysis constants, etc.

**Principle –**

Solutions containing ions conduct electric current due to the movement of ions towards oppositely charged electrodes. The ability of a solution to conduct current is given by its conductivity. The conductivity of a solution is proportional to the number of ions, the charge on each ion, and the speed with which the ions move. The speed of an ion, in turn, depends on the nature of the ion, nature of the solvent, presence of other ions, temperature, and the applied field. The conductance G is defined as the reciprocal of resistance R, i.e.,

$$G=1/R$$

Resistance is measured in ohm (a) and conductance in siemens (S), i.e.,

$$1 S=1 Ω^{-1} $$

 The resistance of a homogeneous body of uniform cross-section is proportional to its length, 1 and inversely proportional to the area of its cross-section, A. The proportionality constant, $ρ$, is called the resistivity (specific resistance). The units of $ρ$ are $Ωm$. the inverse of resistivity, i.e., , The units of $ρ$ are $Ωm$

$$R=ρ(l/A)$$

The conductivity k (Kappa) is the inverse of resistivity, i.e.,

$$k=1/ρ$$

Conductivity of a solution can be defined as the conductance of a solution contained between two parallel electrodes which have cross-sectional area 1 meter square and which are kept 1 meter apart.

$$\frac{1}{R}=\frac{1}{ρ}.1/ (l/A)$$

$$G=k/(l/A)$$

or $k=G(l/A)$

The ratio $l/A$ is called cell constant and it depends on the geometry of the cell - the distance between the electrodes $(l)$ and the area of the electrodes $(A)$. The equation can be written as Conductivity = conductance x cell constant

The cell constant values are usually obtained by an indirect method and not by measuring $l$ and $A$ directly. The indirect method involves measuring the conductance G of a standard solution of KC1 of known conductivity, k.

**Procedure –**

1. Plug in the instrument to the power supply. ,

2. Put the frequency selector switch to the required frequency (say 1000 Hz).

3. Keep the sensitivity knob roughly midway between the lowest and highest sensitivity.

4. Clean the conductivity cell with distilled water (conductivity water). Connect the conductivity cell electrodes to the appropriate terminals of the instrument.

 5. Take the standard KC1 solution (say 0.1 M) in a clean beaker. Introduce a stirring rod (to be used for magnetic stirring) in the solution and put the solution beaker on a magnetic stirrer plate.

 6. Insert the conductivity cell in the solution. Ensure that the platinum plate electrodes are completely immersed in the solution and they do not touch the stirring rod. I

7. Switch on the instrument and allow it to warm up for 2-5 minutes.

8. Measure the conductance, Gs, of the standard KC1 solution. First adjust the coarse balance by rotating the range selection knob, and then increase the sensitivity to obtain the fine balance. Convert the reading for the proper value (Gs) by multiplying the factor used on the range selection knob.

9. Remove the KC1 solution from the beaker wash the conductivity cell properly with distilled water. Take the unknown solution in the beaker and measure its conductance, Gu, in the manner as for standard KC1 solution.

10. Calculate the cell constant from the conductance and conductivity values of the standard, KC1.

1 1. Calculate the conductivity (specific conductance) of the unknown solution.

 12. For titration work, the value of cell constant is not required to be calculated, since the cell constant will remain unchanged during any given titration.

**Observations**

1. Mass of KC1 taken for preparing 100 crn3 0.1 M solution = 0.7446 g
2. Temperature of standard KC1 solution = .. . . . . . . . . . .. tOC
3. Molarity of standard KC1 solution = . . . . .... M
4. Conductance of standard KC1 solution = . . . .x S
5. Conductance of unknown sample solution = . . . .y S

**Precautions**

1. The conductivity cell, when not in use, should be kept in distilled water to prevent drying of the platinum electrodes.
2. In case of fouling of the conductivity cell electrode plates, clean them b). keeping in dilute K2Cr207 containing HzS04 solution (i.e. dilute chromic acid) for 24 hours and

**Experiment No. 8.** To determine the viscosity of a unknown sample solution using Ostwald viscometer.

**Aim of the experiment** - Determine the absolute viscosity of organic liquids.

**Requirements:**

***Chemical***

Distilled water

ethanol

Acetone

***Apparatus***

Ostwald viscometer

Electronic balance

Stopwatch

**Theory-**

The internal property of a fluid for its resistance to flow is known as viscosity. In 1844 Hagen–Poiseuille did their work concerning the interpretation that liquid flow through tubes and he proposed an equation for viscosity of liquids. This equation is called Poiseuille’s equation.



Where *η* is called the viscosity coefficient, *t* is the time of flow of liquid, *V* is the volume of the liquid, *P* is the hydrostatic pressure, and *L* is the distance travelled by the liquid during time *t*. In the honour of Hagen–Poiseuille the unit of viscosity is called the Poise (P). The official SI unit for absolute viscosity is kg/m s (or Pascal-seconds, Pa s).



Figure. The Ostwald Viscometer

 Viscosity can be measured using a viscometer. The different types of viscometer are as follows:

* Ostwald viscometer
* Falling sphere viscometer
* Falling piston viscometer
* Oscillating piston viscometer
* Vibrational viscometers
* Rotational viscometers
* Bubble viscometer

 Ostwald viscometer is a commonly used viscometer, which consists of a U-shaped glass tube held vertically. For more accurate measurements it is held in a controlled temperature bath. It is also known as a glass capillary viscometer. A liquid is allowed to flow through its capillary tube between two etched marks and the time of flow of the liquid is measured using a stopwatch.

In an Ostwald viscometer the measured distance the liquid travels, *L*, will be always a constant; the radius, *r* will always be a constant; and by procedure the volume of liquid, *V* will also be constant. Equation (1) can then be simplified to:





 The hydrostatic pressure is *P* proportional to the density of the fluid being measured. In our experiment we will be measuring the mass of equal volumes of liquid so that the viscosity will be proportional to the masses measured. Therefore we have the relation:



Where K and t are defined above and m is the mass of the liquid.

For finding the viscosity of liquids it is important to calibrate the viscometer using a reference liquid. Water is a commonly used reference liquid. The viscosity of water at 30.0 °C is 0.8007 centipoise (cP). Knowing the values for the reference liquid and relation (3), we get:



Where: *ηr* is viscosity coefficient of the reference sample (water), *mr* is the mass of the reference sample, and *tr* is the time flow of the reference sample. Note that *K* cancels out. The other variables are the viscosity coefficient, mass, and time flow of the sample respectively.

With an Ostwald viscometer we can measure the time flow of a liquid (mass can be measured using standard laboratory procedures, e.g. a relative density bottle and a scale) and determine its viscosity by solving equation (4) for *η*.



### Temperature Dependence of Viscosity

In 1889 Arrhenius expressed an equation for temperature dependent chemical reaction rates. Since then, many temperature dependent chemical and physical processes have been found to behave in accordance with Arrhenius-like equations. For the viscosity of many liquids, the viscosity decreases as the temperature increases in accordance with the following equation:



Where *A* is a constant known as the Arrhenius constant, *Eη* is the activation energy for flow, *R* is the ideal gas constant, and *T* is the temperature of the liquid using an absolute scale (almost always K — the units of *Eη* and *R* and *T* should be chosen so that the ratio is dimensionless, *A* will have the same dimensions as η, in our case cP). A plot of *η* vs. 1/*T* should be linear and have a slope equal to *Eη* /*R* if the liquid’s viscosity exhibits Arrhenius-like behaviour.

 **Procedure**

* The viscometer mounted is vertical position on a suitable stand.
* Wart is filled in to the viscometer up to mark A.
* The time is counted for water to flow from mark A to mark B.
* The same procedure is repeated for the test liquid by using the above formula viscosity of the test liquid can be determined.

**Observation Table**

|  |  |  |  |
| --- | --- | --- | --- |
| **Liquid** | **Time of flow** | **Density** | **Viscosity** |
|  | **I** | **II** | **III** | **Mean** |  |  |
| **Water** |  |  |  |  |  |  |
| **Ethanol** |  |  |  |  |  |  |
| **Acetone** |  |  |  |  |  |  |
| **Toluene** |  |  |  |  |  |  |

**Plot**

 Using an Ostwald viscometer, we can also calculate the unknown composition of a mixture. The viscosities of mixtures of different known compositions are measured and a graph is plotted with viscosity against the compositions of the different mixtures. From the graph, the composition of the unknown mixture corresponding to the viscosity can be determined.



**Conclusion**- The viscosity oof the uunknown liquid is \_\_\_\_\_\_\_\_\_\_\_ is found to be \_\_\_\_\_\_\_\_ is determined by using Ostwald viscometer.

**Experiment No. 9.** To determine the moisture content of a sample with the loss on drying principle using a moisture analyzer.

**Aim of the experiment –** To determine moisture content of a sample.

**Theory-** A moisture analyzer, which is also referred to as a moisture balance or moisture meter, is an instrument used for the determination of the moisture content of a sample. It consists of an infrared weighing and heating unit. A moisture analyzer is extremely important because moisture analysis is necessary across a number of industries. For example, the measurement of moisture content is highly necessary in the food, pharmaceutical, textile and agricultural industries. This is because it affects quality control criteria such as weight, shelf life, pricing and quality.For example, high levels of moisture will decrease the value of products and reduce shelf life due to a greater potential for microbial activity. Most products will have an optimum moisture content for the highest quality and there are many laws and regulations set in place to determine the correct moisture content for certain regulated products. For example, there are national food regulations defining the maximum moisture content permissible for certain food produce. Therefore, moisture analysis is essential for compliance to such regulations and the production of a quality product. The best method for moisture analysis is via the use of a Moisture analyzer.

Scope. Water is present in most naturally occurring soils and has a profound effect in soil behaviour. A knowledge of the moisture content is used as a guide to the classification. It is also used as a subsidiary to almost all other field and laboratory tests of soil. The oven-drying method is the definitive method of measuring the moisture contents of soils. The sand-bath method is used, where oven drying is not possible, mainly on site.

Definition. The moisture content of a soil sample is defined as the mass of water in the sample expressed as a percentage of the dry mass, usually heating at 1050C, i.e. moisture content,

$$w= \frac{M\_{W}}{M\_{D}}×100 \%$$

where, MW = mass of water MD = dry mass of sample

**Sample requirements**

Sample mass. The mass required for the test depends on the grading of the soil, as follows;

a) Fine-grained soils\*, not less than 30 grams

b) Medium-grained soils\*, not less than 300 grams

c) Coarse-grained soils\*, not less than 3 kg

\*Soils group

 i) Fine-grained soils: Soils containing not more than 10% retained on a 2 mm test sieve.

 ii) Medium-grained soils: Soils containing more than 10% retained on a 2 mm test sieve but not more than 10% retained on a 20 mm test sieve.

 iii) Coarse-grained soils: Soils containing more than 10% retained on a 20 mm test sieve but not more than 10% retained on a 37.5 mm test sieve.

**Apparatus**

1) Thermostatically controlled drying oven capable of operating to 105±5 0C.

2) Glass weighing bottles or suitable metal containers (corrosion-resistant tins or trays).

3) Balance (to the required sensitivity).

4) Dessicator containing anhydrous silica gel.

5) Scoop, other small tools as appropriate. Optional: Test sieves - 2 mm, 20 mm, 37.5

**Procedure**-

1. Start the application and tare the empty weighing pan.
2. Add the sample.
3. Start the measurement and see your results in just a few minutes.
4. One clean container with the lid (if fitted) is taken and the mass in grams is recorded (m1) together with container number. Note: The container plus lid or bottle plus stopper should have the same number and be used together.
5. The sample of wet soil is crumbled and placed in the container. The container with the lid on is weighed in grams (m2).
6. The lid is removed and both lid and container are placed in the oven. The sample is then dried in a thermostatically controlled drying oven which is maintained at a temperature of 105±50C. A period of 16 to 24 hours is usually sufficient, but this varies with soil type. It will also vary if the oven contains a large number of samples or very wet samples. The soil is considered dry when the differences in successive weighing of the cooled soil at 4 hour intervals do not exceed 0.1% of the original mass.
7. The container is removed from the oven. For medium and coarse-grained soils, the lid should be replaced (if fitted) and the sample allowed to cool. For fine-grained soils, the container and lid, or bottle and stopper if used, should preferably be placed in a dessicator and allowed to cool. After cooling, the lids or stoppers should be replaced and the container plus dry soil weighed in grams (m3).

**Observation Table**-

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Dish No.** | **Weight of empty dish** | **Weight of initial sample** | **Weight of final sample** | **weight loss** |
| **1.** | 45.48g | 5.03g | 0.494g | 4.536g |
| **2.** | 12.74g | 5.04g | 0.548g | 4.492g |
| **3.** | 22.05g | 5.09g | 0.534g | 4.556g |
| **4.** | 36.55g | 4.03g | 0.349g | 3.527g |

Expression of results and report Moisture content should be expressed to two significant figures, e.g. 1.9%, 4.3%. Wet weight / dry weight conversion Chart (when steel ball pulverizes are not used) is presented in Table 3.1.1 and the conversion chart (when using steel ball pulverizes) is shown in Figure 3.1.2. The test report shall contain the following information.

a) Method of test used,

b) The moisture content

c) Full details of the sample origin.

**Conclusion**- The moisture content of a sample is \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_.

**Precautions**-

* The weighing of the sample should be done carefully.
* The temperature should be performed at controlled temperature conditions.

**Experiment No. 10.** To estimate the TDS of the water sample.

**Aim of the experiment-** To estimate the total dissolved solids in water.

**Requirements-**

1) Membrane filter with funnel.

2) Gooch crucible, 25-mL to 40-mL capacity, with Gooch crucible adapter.

3) Filtration apparatus with reservoir and coarse (40- to 60- µm) fritted disk as filter support.

4) Suction flask of sufficient capacity for sample size selected.

5) Drying oven, for operation at 180 ± 2°C. 6) Common chemical balance.

**Theory** – Potable water should be of such quality that it produces no sensation of taste and odor. An estimation of total dissolved solids gives information on the real quality of water used in fish processing industry. The minerals dissolved in water tend to concentrate in the central part of ice blocks and gives a cloudy appearance in the centre which is undesirable. The two principal methods of measuring total dissolved solids are [gravimetric analysis](https://en.wikipedia.org/wiki/Gravimetric_analysis) and [conductivity](https://en.wikipedia.org/wiki/Conductivity_%28electrolytic%29). Gravimetric methods are the most accurate and involve evaporating the liquid [solvent](https://en.wikipedia.org/wiki/Solvent) and measuring the mass of residues left. This method is generally the best, although it is time-consuming. If inorganic salts comprise the great majority of TDS, gravimetric methods are appropriate.

Primary sources for TDS in receiving waters are [agricultural runoff](https://en.wikipedia.org/wiki/Surface_runoff) and [residential (urban) runoff](https://en.wikipedia.org/wiki/Urban_runoff), clay-rich mountain waters, leaching of [soil contamination](https://en.wikipedia.org/wiki/Soil_contamination), and [point source](https://en.wikipedia.org/wiki/Point_source_%28pollution%29) [water pollution](https://en.wikipedia.org/wiki/Water_pollution) discharge from industrial or [sewage treatment](https://en.wikipedia.org/wiki/Sewage_treatment) plants. The most common chemical constituents are [calcium](https://en.wikipedia.org/wiki/Calcium), [phosphates](https://en.wikipedia.org/wiki/Phosphate), [nitrates](https://en.wikipedia.org/wiki/Nitrate), [sodium](https://en.wikipedia.org/wiki/Sodium), [potassium](https://en.wikipedia.org/wiki/Potassium), and [chloride](https://en.wikipedia.org/wiki/Chloride), which are found in [nutrient](https://en.wikipedia.org/wiki/Nutrient) runoff, general [stormwater](https://en.wikipedia.org/wiki/Stormwater) runoff and runoff from snowy climates where road [de-icing](https://en.wikipedia.org/wiki/De-icing) salts are applied. The chemicals may be [cations](https://en.wikipedia.org/wiki/Cation), [anions](https://en.wikipedia.org/wiki/Anion), [molecules](https://en.wikipedia.org/wiki/Molecule) or agglomerations on the order of one thousand or fewer molecules, so long as a soluble micro-[granule](https://en.wiktionary.org/wiki/granule) is formed. More exotic and harmful elements of TDS are [pesticides](https://en.wikipedia.org/wiki/Pesticide) arising from [surface runoff](https://en.wikipedia.org/wiki/Surface_runoff). Certain naturally occurring total dissolved solids arise from the weathering and dissolution of rocks and soils. The United States has established a secondary water quality standard of 500 mg/l to provide for palatability of drinking water. Total dissolved solids are differentiated from [total suspended solids](https://en.wikipedia.org/wiki/Total_suspended_solids) (TSS), in that the latter cannot pass through a sieve of 2 micrometers and yet are indefinitely suspended in solution. The term settleable solids refers to material of any size that will not remain suspended or dissolved in a holding tank not subject to motion, and excludes both TDS and TSS.[[3]](https://en.wikipedia.org/wiki/Total_dissolved_solids#cite_note-3) Settleable solids may include larger particulate matter or insoluble molecules.Electrical conductivity of water is directly related to the concentration of dissolved ionized solids in the water. Ions from the dissolved solids in water create the ability for that water to conduct an [electric current](https://en.wikipedia.org/wiki/Electric_current), which can be measured using a conventional [conductivity meter](https://en.wikipedia.org/wiki/EC_meter) or [TDS meter](https://en.wikipedia.org/wiki/TDS_meter). When correlated with laboratory TDS measurements, [conductivity](https://en.wikipedia.org/wiki/Conductivity_%28electrolytic%29) provides an approximate value for the TDS [concentration](https://en.wikipedia.org/wiki/Concentration), usually to within ten-percent accuracy.

The relationship of TDS and specific conductance of groundwater can be approximated by the following equation:

*TDS = ke E C*

where TDS is expressed in mg/L and EC is the electrical conductivity in microsiemens per centimeter at 25 °C. The correlation factor *ke* varies between 0.55 and 0.8.

**Objective**

After performing this experiment, you will be able to:

• estimate the total dissolved solids in water.

**Procedure**-

1) Stir the sample with a magnetic stirrer.

2) Pipet a measured volume onto a whatman filter paper with applied vacuum.

3) Wash with three successive 10-mL volumes of reagent-grade water and continue suction for about 3 min after filtration is complete.

4) Transfer total filtrate (with washings) to a weighed evaporating dish.

5) Evaporate to dryness on a steam bath or in a drying oven.

6) Dry evaporated sample for at least 1 hr in an oven at 180 ± 2°C.

7) Cool in a desiccator to balance temperature and weigh.

**Observations** –The experimental procedure will give you the content of total dissolved solids.

**Observation Table**

|  |  |  |
| --- | --- | --- |
| **Description** |  | **Weight (g)** |
| Weight of the clean porcelain evaporating dish (g) | W1 |  |
| Weight of the dish and the residue (g) | W2 |  |
| Weight of residue(g) | W |  |
| Volume of the Sample (mL) | V |  |
| **Total Dissolved Solids (mg/L)** | **TDS** |  |

**Tabulation for TDS**

Weight of the clean porcelain evaporating dish (g) W1 =35.4329

Weight of the dish and the residue (g) W2 =35.4498

Weight of residue (g) W =0.0169

The volume of the sample (mL) V = 50.0

**Calculations-**

Total dissolved solids = in given water sample Sample volume, mL

$$Total dissolved Solids= \frac{\left(A-B\right)×10000}{sample volume in mL}$$

Where, A = weight of dried residue + dish in mg,

and B = weight of dish in mg.

**Result**

The total dissolved solids in the given water sample = ................... mg/L

**Precautions**-

The following precautions should be observed while performing the experiment:

• Water or Wastewater samples which contain high concentrations of calcium, chloride, magnesium or sulfate can rapidly absorb moisture from the air.

• Such samples may need to be dried for a longer period of time, cooled under proper desiccation and weighed rapidly in order to achieve a reasonable constant weight.

• We should be aware prolonged drying may result in loss of constituents, particularly nitrates and chlorides.

• Volume of sample should be adjusted to have residue left after drying as100 to 200mg. It is mainly to prevent large amount of residue in entrapping water during evaporation.

• Samples with high concentrations or bicarbonate require additional drying at 180ºC to ensure that all of the bicarbonate is converted to carbonate

• Take normal precautions while experimentation