CERTIFICATE OF PARTICIPATION

Date: 17-03-2018

This is to certify that 20 numbers of B.Sc. Chemistry I Year Students of Nirmala College Muvattupuzha, attended L2L (Lecture to Lab Series) training programme at STIC during the period 16-17, March 2017. The programme comprises of lectures and laboratory demonstration on following Analytical Techniques

(i)X-Ray Diffraction (ii) Spectroscopy (iii) NMR Spectroscopy (iv) Thermal Analysis (v) Elemental Analysis and (vi) Microscopy

Sincerely

SOPHISTICATED ANALYTICAL INSTRUMENT FACILITY
STIC, CUSAT
COCHIN

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L2L

Lecture to Lab Series 2018

X-Ray Diffraction

Powder and Single Crystal Analysis

Analytical Spectroscopy

UV-Vis -NIR-Mid IR

NMR Spectroscopy

NMR and Mass Analysis

Electron Microscopy

Scanning and Transmission

Elemental Analysis

Ultimate and Heavy metals

Thermal Analysis

TGA and DSC

Course Outline

L2L Series includes Theory Session and laboratory demonstrations of Following Analytical Methods

- **X-Ray Diffraction**
- **Electron Microscopy (Scanning and Transmission)**
- **Spectroscopy (UV Vis NIR & Mid IR)**
- **Thermal Analysis (Thermo Gravimetry and Differential Scanning Calorimetry)**
- **Nuclear Magnetic Resonance and Mass Spectroscopy**
- **Elemental Analysis (Ultimate and Heavy Metal analysis)**

Mode & Duration

Short Theory Sessions

Time	9.30 -10.30 AM	11.45- 12.30	2.30-3.30 PM
Day I	**X-Ray Diffraction**	**Analytical Spectroscopy**	**Electron Microscopy**
Day II	**Thermal Analysis**	**NMR and Mass Spectrometry**	**Elemental Analysis**

Laboratory Sessions

Time	10.30 -11.30 AM	1.30-2.30 PM	3.30 – 4.30 PM
Day I	**X-Ray Diffraction**	**Analytical Spectroscopy**	**Electron Microscopy**
Day II	**Thermal Analysis**	**NMR and Mass Spectrometry**	**Elemental Analysis**

Registration: Terms & Conditions

All applicants must complete the application form.

This is not a training course. This program envisages to create an awareness among graduates/postgraduates and research students on use of sophisticated analytical instruments. Persons from industries, teachers under FIP/TIP and other schemes are not permitted.

Hands own training may not be possible. But elaborate laboratory demonstration by expert technical persons will be available.

Each analytical facilities covered under the programme is a massive subject hence detailing in every aspects may not be possible in a short span.

Programme fee

Rs. 500 per student GST extra @18% Number of students permitted each time minimum 15 maximum 30

No course materials or refreshments will be provided. Students are permitted to carry lunch with them.

Please note that it is often difficult for us to find someone to fill your seat. You will not incur a refund but you can find a substitute to take your place.

We have done our best to create a favorable environment for teaching and learning. There is plenty of time for questions and discussions during each course.

INTRODUCTION

The need of precise , accurate and reliable analytical data has become relevant as the regulatory agencies scrutinizes every aspect in industries, medicine and environmental monitoring. In Research and development the application of a precise and accurate analytical instrument plays a vital role. The choice of an instrument for a specific application is crucial. Good working knowledge of the available technique and equipment is necessary. The technology is getting smart, fast and small day by day. This attempt is to familiarize the fundamentals, basic instrumentation, operation and analysis using instruments available at SAIF Laboratory STIC

Analytical Techniques can be divided into three broad categories given below which can be applied to Fundamental research, Quality Control, Product testing, Medical and Clinical Testing, Environmental Monitoring etc,

1. Qualitative analysis

Identifying one or more species present in a material.

2. Quantitative analysis

Determining the amount of a specie present in a material.

3. Structure Determination

Finding the spatial arrangement of atoms in a molecule

The scope of this short note includes the following Analytical Techniques

1. Analytical Spectroscopy

Fourier Transform Infrared Spectroscopy

UV-Vis NIR Spectroscopy

2. Thermal Analysis

Thermo gravimetric Analysis

Differential Scanning Calorimetry

3. X-Ray Diffraction

X-Ray Powder Diffraction

X-Ray Single Crystal structure determination

4. Electron Microscopy

Scanning Electron Microscopy

Transmission Electron Microscopy

5. Elemental analysis

Atomic Emission Spectrometry

Ultimate Analysis (CHNS)

Cold Vapour Atomic Absorption Spectrometer

6. **NMR and Mass Spectroscopy**

1.Analytical Spectroscopy

Spectroscopy is the study of the interaction of electromagnetic radiation with matter.

The electromagnetic radiation may be anywhere from very high energy gamma rays to low frequency radio waves.

The nature of the interaction depends upon the frequency or energy of the electromagnetic radiation and also on the properties of the matter.

The distribution of energy possessed by a molecule at any given moment, defined as the sum of the contributing energy terms

$E\ total = E\ electronic + E\ vibrational + E\ rotational + E\ translational$

The translational energy relates to the displacement of molecules in space as a function of the normal thermal motions of matter. Rotational energy, is observed as the tumbling motion of a molecule, which is the result of the absorption of energy within the microwave region.

The vibrational energy component is a higher energy term and corresponds to the absorption of energy by a molecule as the component atoms vibrate about the mean center of their chemical bonds.

The electronic component is linked to the energy transitions of electrons as they are distributed throughout the molecule, either localized within specific bonds, or delocalized over structures, such as an aromatic ring. In order to observe such electronic transitions, it is necessary to apply energy in the form of visible and ultraviolet radiation (190nm to 3000nm) The infrared regions are classified as follows:

Near Infrared 12,500 to 4,000 cm-1

(0.8 to 2.5 μm)

Mid Infrared 4,500 to 400 cm-1

(2.5 to 50 μm)

Far Infrared 400 to 12.5 cm-1

(50 to 800 μm)

2. Electron Microscopy

Most microscopes can be classified as one of three basic types: optical, charged particle (electron and ion), or scanning probe. Optical microscopes use visible light and transparent lenses to see objects as small as about one micrometer (one millionth of a meter), such as a red blood cell (7 μm) or a human hair (100 μm). Electron and ion microscopes, use a beam of charged particles instead of light, and use electromagnetic or electrostatic lenses to focus the particles. They can see features as small a tenth of a nanometer (one ten billionth of a meter), such as individual atoms. Scanning probe microscopes use a physical probe (a very small, very sharp needle) which scan over the sample in contact or near-contact with the surface. They map various forces and interactions that occur between the probe and the sample to create an image. These instruments too are capable of atomic scale resolution

3. X-Ray Diffraction

Solid matter can be described as:

Amorphous: The atoms are arranged in a random way similar to the disorder we find in a liquid. Glasses are amorphous materials.

Crystalline: The atoms are arranged in a regular pattern, and there is as smallest volume element that by repetition in three dimensions describes the crystal. E.g. we can describe a brick wall by the shape and orientation of a single brick. This smallest volume element is called a unit cell. The dimensions of the unit cell is described by three axes: a, b, c and the angles between them alpha, beta, gamma.

About 95% of all solids can be described as crystalline. An electron in an alternating electromagnetic field will oscillate with the same frequency as the field. When an x-ray beam hits an atom, the electrons around the atom start to oscillate with the same frequency as the incoming beam. In almost all directions we will have destructive interference, that is, the combining waves are out of phase and there is no resultant

energy leaving the solid sample. However the atoms in a crystal are arranged in a regular pattern, and in a very few directions we will have constructive interference. The waves will be in phase and there will be well defined x-ray beams leaving the sample at various directions. Hence, a diffracted beam may be described as a beam composed of a large number of scattered rays mutually reinforcing one another. X-Rays can be used as an analytical tool to see through a molecule since the wavelength of X-rays is compatible with inter atomic distances in a molecule.

4. Thermal Analysis

When sample of mass *m is* heated *, the sample receives energy.* In consequence, the state of the sample might change. Either a phase transition occurs or the internal energy changes. If the internal energy changes without a phase transition, the received quantity of heat, δq, *is proportional to the temperature* increase, δT:

$$\delta q = mC\,\delta T, \quad C = \textit{specific heat capacity}$$

The heat capacity depends on the conditions under which the system is treated. If the heat transfer is measured at constant volume, the heat capacity is defined as C_V. If the heat capacity is measured at constant pressure, the heat capacity is defined as Cp.

Thermo Gravimetric Analysis (TGA)

A technique whereby the weight of a substance, in an environment heated or cooled at a controlled rate, is recorded as a function of time or temperature. Thus, the data obtained from a TG experiment are displayed as a thermal curve with an ordinate display having units of weight (or weight percent) and the abscissa may be in units of either temperature or time.

In TG studies, mass loss is read directly in units of weight percent of the original sample quantity. The results from thermogravimetric analysis may be presented by (1) mass versus temperature (or time) curves, referred to as Thermogravimetric curve, or (2) rate of mass loss versus temperature curve, referred to as Derivative Thermogravimetric (DTG). The results of a TG experiment may be used, in many cases, as "compositional analysis". Measurements of changes in sample mass with temperature are made using a thermobalance. Thermogravimetric analysis relies on high degree of precision in three measurements: mass change, temperature, and temperature change.

Differential scanning calorimetry

Differential scanning calorimetry (DSC) monitors heat effects associated with phase transitions and chemical reactions as a function of temperature. In a DSC the difference in heat flow to the sample and a reference at the same temperature, is recorded as a function of temperature. The sample is sealed in an aluminum pan. The reference is an inert material such as alumina, or just an empty aluminum pan. The temperature of both the sample and reference are increased at a constant rate. Since the DSC is at constant pressure, heat flow is equivalent to enthalpy changes:

$$\frac{đq_p}{dt} = \frac{dH}{dt}$$

5. Elemental Analysis

CHNS elemental analysers have been used in analytical laboratories for over thirty years. The method is used extensively across a wide range of applications, including pharmaceuticals, chemicals, oil-related products, catalysts and food. In the oil industry, an important application is the regular monitoring of coke build-up on refinery catalysts to ensure that regeneration procedures (involving controlled burning of the carbon) are executed at optimal intervals. Since many of these catalyst systems involve large quantities of noble metals such as platinum, palladium and rhenium, mismanagement of this testing would entail serious financial losses. In food analysis, the determination of nitrogen (as a surrogate for protein) is very important for pricing grain and evaluating meat products, and is increasingly undertaken by combustion analysis.

Heavy metal detection is done mainly using AAS, ICPAES, Flame Ionization, and ICP-MS methods. The operation of an ICP-AES system relies upon the same interaction of molecules with electromagnetic radiation. in a flame-based system, not all of the atoms or elements present in the sample are excited, particularly if they exist in a polyatomic compound. Some elements readily form non-emitting and refractory oxides that result in an underestimation of their concentration. In plasma-based systems the temperature is considerably hotter (~6000 to 10 000 K) that results in more effective excitation of atoms(generally greater then 90%) of approximately 60 elements including some non metals. This intense heat prevents polyatomic species from forming, thus increasing the detection limits for many elements

6. NMR Spectroscopy

Nuclei with an odd mass or odd atomic number have "nuclear spin" (in a similar fashion to the spin of electrons). This includes 1H and 13C (but not 12C). The spins of nuclei

are sufficiently different that NMR experiments can be sensitive for only one particular isotope of one particular element. Since a nucleus is a charged particle in motion, it will develop a magnetic field. 1H and 13C have nuclear spins of 1/2 and so they behave in a similar fashion to a simple, tiny bar magnet. In the absence of a magnetic field, these are randomly oriented but when a field is applied they line up parallel to the applied field, either spin aligned or spin opposed. The more highly populated state is the lower energy spin state spin aligned situation

Structural factors cause changes in the magnetic field experienced by the nucleus. this changes the resonance frequency and hence the chemical shift. The structural factors mean that different types of proton will occur at different chemical shifts. This is what makes NMR so useful for structure determination.