

Value added course

on

Hands on training on Analytical Instruments

(24th and 25th of March – 2022)

Venue: 3rd floor of PG block,

Chemical Engineering Department.

B.M.S College of Engineering

Value-added Course on “Hands on training on Analytical Instruments”

APPLICATION FORM

Value Added Course on Hands on training on Analytical Instruments

24-25th March 2022

1. Name:
2. Department:
3. USN:
4. Mobile:
5. E-mail Id:

Online Registration Link

<https://bit.ly/3wnRUrZ>

DECLARATION

The information furnished above is true to the best of my knowledge. I agree to abide by the rules and regulations governing the course. If selected, I shall attend the course for the entire duration.

Place:

Signature of the applicant

Date:

Value Added Course On Hands on training on Analytical Instruments

24-25th MARCH 2022



Organized by

**Department of Chemical Engineering
B. M. S. College of Engineering
Bengaluru-560019**

B. M. S. COLLEGE OF ENGINEERING

B. M. S. College of Engineering was founded in the year 1946 by Late B. M. Sreenivasaiah, and nurtured by his illustrious son, Late B. S. Narayan. BMSCE is the first private sector initiative in Engineering Education in India. BMSCE is in the 75th year of dedicated service in the field of Engineering Education. BMSCE today offers 13 Under Graduate & 16 Post Graduate courses, both in conventional and emerging areas. At present, 15 Departments are recognized as Research Centers offering Ph.D./MSc. Engineering by Research in Science, Engineering, Architecture and Management. First Institution in the state bestowed with NBA Accreditation in Tier I Format (Washington Accord). World Bank funded TEQIP Phase I, Phase II, and Phase III Institute, and Partner Institution of the Melton Foundation, USA, which advocates Global Citizenship with exchange of culture. BMS is an Autonomous Institute (UGC approved) since 2008.

ABOUT CHEMICAL ENGINEERING

The department started in 1995 by the great visionary Late B. S. Narayan, with an intake of 40 students. Subsequently, in the year 2002, the intake was increased to 60 students. The Chemical Engineering Department has 14 number of teaching faculty, and NBA accredited for the duration of 5 years in the year 2004, and then

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later in 2014 for a period of 5 years accreditation from NBA, India in Tier-1 format under Washington Accord. Now, this accreditation is extended till 2022. The autonomous undergraduate course takes its cognizance from the program specific criteria established in all reputed institutes offering course in Chemical Engineering. The well-balanced curriculum, including the titles like Basic Sciences (BS), Engineering Sciences (ES), Professional Core (PC), Professional Electives (PE), Humanities and Social Sciences (HSS), and Project Work (PW). The Research Centre for Ph.D. and MSc. (Engg) by research was established during the year 2004. Under this research center, totally 3 students have completed their Ph.D.

COURSE CONTENT

1. Gas Chromatography
2. UV Spectrometry
3. Rotavapor
4. Bomb Calorimeter

OBJECTIVES AND OUTCOMES VAC

1. Able to operate the analytical instruments
2. Able to analyze the various analytes in the solution
3. Enables to measure the heat of combustion of a particular reaction.

5. Analyze the GC chromatograms
6. Analyze the Kubelka-Munk/Tauc plot from UV data
7. Able to determine the band gap energy of nano materials

RESOURCE PERSONS

Dr. Y. K. Suneetha, Associate Professor & Head
Department of Chemical Engineering, BMSCE, Bengaluru

Mr. Hari Prasad Uppara, Assistant Professor, Department of
Chemical Engineering, B.M.S. College of Engineering

CHIEF PATRONS

Dr. B. S. Ragini Narayan, Donor Trustee, BMSET
Dr. P. Dayanand Pai, Chairman, BMSCE
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Dr. Suresh Ramaswamyreddy, Vice Principal
(Administration)
Dr. Bheemsha, Vice Principal (Academic)

ORGANIZING CHAIR

Dr. Y. K. Suneetha, Associate Professor & Head
Department of Chemical Engineering, BMSCE,
Bengaluru

COORDINATORS

Dr. Y. K. Suneetha, Associate Professor & Head
Department of Chemical Engineering, BMSCE, Bengaluru

Mr. Hari Prasad Uppara, Assistant Professor, Department of
Chemical Engineering, B.M.S. College of Engineering

WHO CAN ATTEND?

Any student who is willing to learn the analytical instruments.

REGISTRATION FEE

Students/Research Scholars: Rs. 200

IMPORTANT DATES

Last date for receipt of Application & registration fee: 23rd
March 2022

HOW TO APPLY

Register online using the below link.

URL: <https://bit.ly/3wnRUrZ>

ADDRESS FOR CORRESPONDENCE

Mr. Hari Prasad Uppara
Coordinator, Department of Chemical Engineering, B. M. S.
College of Engineering, Basavanagudi, Bengaluru-560019
Phone: 8019275350, E-mail: hariprasad.che@bmsce.ac.in

Report

All the faculty members of chemical engineering department are attended for the inaugural function of this Value-added course. Firstly, the Head of the department (Dr. Y.K. Suneetha) welcomed all the participants and explained the importance and the outcomes of this course to the participants. And she suggested the participants to utilize this facility for full extent for their future endeavours.

Objectives:

Students are trained to understand the principle of working, learn and practice the standard protocol and analyze the data by conducting experiments on

1. Gas Chromatography
2. UV Spectrometry
3. Rotavapor
4. Bomb Calorimeter

Outcomes of the course:

At the end of the course the student will be

1. Able to operate the analytical above-mentioned instruments
2. Able to analyze the various analytes in the solution
3. Capable of measuring the heat of combustion of a particular reaction.
4. Capable to analyze the GC chromatograms
5. Capable to analyze the Kubelka-Munk/Tauc plot from UV data
6. Able to determine the band gap energy of Nano materials

This course was intended to expose the students to the analytical instruments that will be useful for their final year project work. In this course, the instruments such as GC, UV-vis-spectrometry, Bomb calorimetry and Rotavapor were covered and provided the hands-on-training to the students.

This Valued-added course was organized in two days. In first day, the instruments such as Rotavapor and Gas chromatography was demonstrated and performed the analysis of the

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prepared samples. And the participants were actively took-part and operated the instruments for the un-known sample mixtures. In second day of this course, the instruments such as Bomb calorimetry and UV-Vis spectrometry was operated for the prepared concentrations of the samples. In addition, the sample data collected from UV-vis spectrometry was plotted and analyzed using Tauc plots/Kubelka-Munk functions. Finally, the Vote of thanks given by the Head of the department. And the participants expressed their high satisfaction of this Hands-on-training course and they said that they will recommend to the fellow students to join this course if it is conducted again.

In GC analysis, four pure samples such as Hexane, methanol, ethanol and propanol were injected and analysed the peaks of these samples by integrating the peak areas using software provided with the instrument. The students were allowed to prepare the mixture of above samples. Moreover, each student was allowed to inject the prepared mixture samples into GC instrument and analysed the peak areas. In UV-vis-spectrometry, the four different concentrations of copper sulphate and potassium permanganate was analysed in this instrument and obtained the absorbance data for these prepared samples. And the students were allowed to operate the instrument and analysed the data obtained from this instrument. The similar kind of practice was also performed for Bomb calorimetry and Rotavapor.

Course Content

Gas Chromatography

Make of GC: Mayura Analytics PVT. Ltd. Bengaluru.

GC Model No: 9800

Column: Capillary or packed column

Detector: FID and TCD detectors

When a mixed solution containing the different compounds is injected into the GC. Then the mixture is heated and vaporized within the sample injection unit. In the GC system, the carrier gas (mobile phase) transports the vaporized mixture in the injection unit to the column, and then to the detector. However, the mixture of compounds is getting separated into the various components, and the amount of each compound is then measured by the detector. The detector converts the amount of each compound into an electrical signal, and sends these signals to a data processing unit. The data obtained enables to determine the amount of compounds contained in the sample.

There are three main GC system components:

1. **Injection unit:** Herein, the sample is heated and converted into vapours.
2. **GC column:** The column is used to separate each compound.
3. **Detector:** It detects the compounds and outputs their concentrations as electrical signals.

Compounds that cannot be analysed

- Compounds that do not vaporize (inorganic metals, ions, and salts)
- Highly reactive compounds and chemically unstable compounds (hydrofluoric acid and other strong acids, ozone, NO_x and other highly reactive compounds)

Compounds that are difficult to analyse

- Highly adsorptive compounds (compounds containing a carboxyl group, hydroxyl group, amino group, or sulfur)
- Compounds for which standard samples are difficult to obtain (Qualitative and quantitative analyses are difficult.)

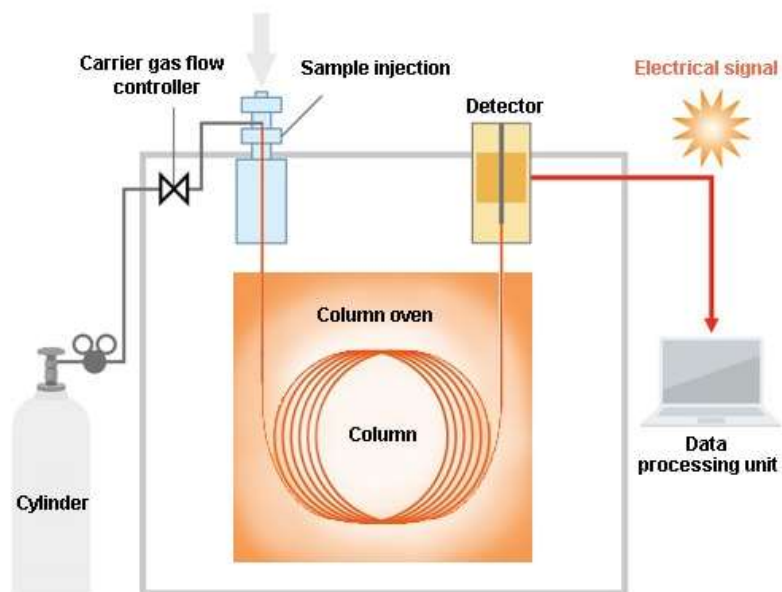


Fig. 1. Schematic representation of GC instrument.

Operating procedure:

- ✓ Switch on the instrument and the computer.
- ✓ Firstly, allow the inert gas flow to the GC instrument.
- ✓ Then, allow the flow of combustible gas (H_2 , O_2 and inert) to the GC instrument and confirm the flame at the detector point.
- ✓ Open the online GC software in PC and choose both channel1 and channel2.
- ✓ Go to windows panel in the software and choose the vertical tilt.
- ✓ Then, go to ESM and EZM software. Set the desired temperature in ID1 (Oven) and ID2 (injector/detector). Indeed, the injector/detector temperature should be maintained more than the oven temperature.
- ✓ After reaching the temperature to the set point temperature, do calibration with the standard sample.
- ✓ Finally, perform the analysis for the unknown sample.

UV-vis Spectrometry

UV-Vis spectrometry model no: Cary 8454

Make: Agilent Technologies

UV lamp: Deuterium lamps

Operating temperature: 0 to 40°C

Spectroscopy is the theoretical science dealing the interaction of electromagnetic radiation with the matter. Spectrometry is the application of spectroscopic technique in which the interaction of radiating energy (in terms of wavelength and frequency) with the matter can be measured and quantified to obtain the physio-chemical properties of the matter. The wavelength can either be fixed or scanned during the measurement. When the radiating energy interacts with the matter it undergoes various phenomena like absorption (photons absorbed by the matter), transmission (photons not absorbed by the matter), reflection (part of photons reflected) and refraction due to crystal structure or change in the electronic state.

UV-vis spectroscopy uses UV (1 - 380 nm) and visible radiation (380 – 780 nm) to carry the absorption process. According to Beer-Lambert law, when the incident light (I_0) passes through the sample having concentration c , a part of light is absorbed by the sample and the remaining light is transmitted with attenuated intensity (I_t).

$$\text{Absorbance, } A = -\log\left(\frac{I_t}{I_0}\right) \text{ or } A = \epsilon \cdot c \cdot l$$

Where ‘ ϵ ’ is molar absorption coefficient ($L \cdot mol^{-1} \cdot cm^{-1}$), ‘ c ’ is concentration of sample ($mol \cdot L^{-1}$) and ‘ l ’ is optical path length (cm).

Components of UV-visible spectrophotometer:

The major components of UV-visible spectrophotometer are source of light, wavelength selector, sample containers and detector (shown in **Fig.2**).

1. Source of light - The deuterium and hydrogen lamps are used for the UV region because it emits mostly in region of 160-375 nm. However, tungsten filament lamps are used in the visible and IR region because it emits in the wavelength region of 320-2500 nm.

2. Wavelength selector – the spectroscopic instruments use wavelength selector to provide specific range of wavelength to hit on the sample. Generally, two types of wavelength selectors are used namely filters and monochromators.
3. Sample containers made up of glass (340-2000 nm) or quartz (190-2500 nm) cuvettes. In UV region, quartz cuvette is used because glass container absorbs much of the radiation.
4. Detector – A detector which converts light signal to electrical signal. Examples of detectors are photomultiplier tube and photodiode detectors.

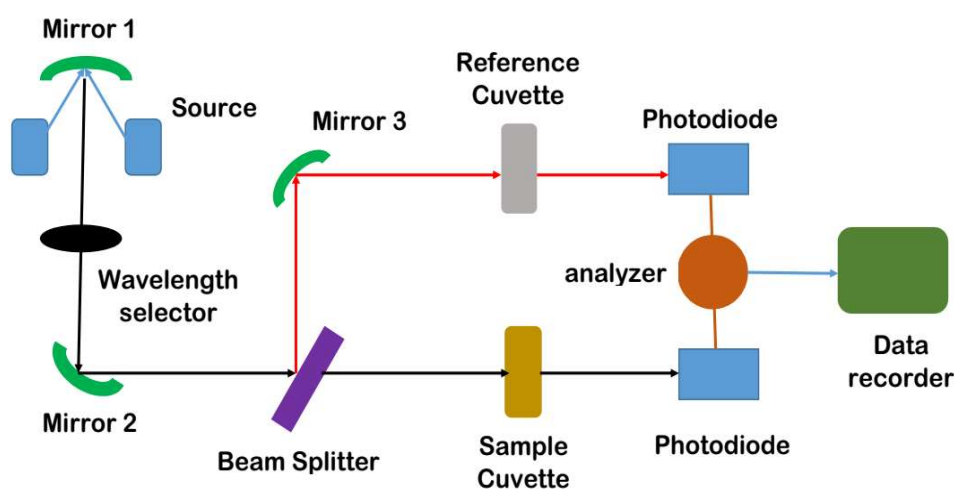


Fig.2. Schematic illustration of UV-vis spectrometry.

UV-visible spectroscopy is used to study the electronic transitions of the material from ground state to excited state upon absorption of radiation (190 – 780 nm). The possible electronic transitions are $\sigma \rightarrow \sigma^*$, $\pi \rightarrow \pi^*$, $n \rightarrow \sigma^*$, $n \rightarrow \pi^*$, $\pi \rightarrow \sigma^*$ and $\sigma \rightarrow \pi^*$; in which $\sigma \rightarrow \pi^*$, $\pi \rightarrow \sigma^*$ are forbidden transitions and the remaining are allowed transitions. Among all transitions, the $\sigma \rightarrow \sigma^*$ requires the greatest energy and lower wavelength to excite it. Similarly, the $n \rightarrow \pi^*$ requires the lowest energy and shows the absorption peaks at longer wavelengths.

UV-vis DRS is a useful technique for investigating d-d and d-f transitions of metal ions and to determine their oxidation states within a mixed oxide. In addition, band gap energies are also calculated to understand the property the material. Generally, the bandgap energies of the samples are calculated using the Tauc plot.

$$[F(R_{\infty})hv]^{\frac{1}{n}} = C (hv - E_g)$$

$$F(R_{\infty}) = \frac{k}{s} = \frac{(1-R_{\infty})^2}{2R_{\infty}}$$

Herein, $F(R_{\infty})$, R_{∞} , h , ν , E_g , C , n represents Kubelka-Munk function, diffuse reflectance, Planck's constant, incident light frequency, bandgap energy, a constant, and an integer. The integer 'n' is chosen as 0.5 for the bandgap is in direct transition. It is observed that a plot between $[F(R_{\infty})hv]^2$ ν/s hv was linear near the edge for the direct transition. The extrapolation of the straight line to abscissa provides information about bandgap energy. The value of n mainly depends on the electronic transition structure of different semiconductors. If the band gap is a direct transition, then $n = 1/2$; if the band gap is an indirect transition, then $n = 2$.

Rotavapor

Model: Rotavap R-210

Make: Buchi

Oil heating bath temperature: Max 180°C

Combi clip: Handling the evaporating flask.

A rotary evaporator is a device used in chemical laboratories for the efficient and gentle removal of solvents from samples by evaporation. Rotary evaporators (also called "rotavaps") are used to remove solvents from reaction mixtures and can accommodate volumes as large as 3 liters. They are found in almost every organic laboratory, since they allow performing this task very quickly. A typical rotary evaporator has a water bath that can be heated in either a metal container or crystallization dish. This keeps the solvent from freezing during the evaporation process. The solvent is removed under vacuum, is trapped by a condenser and is collected for easy reuse or disposal. Most labs use a simple water aspirator vacuum on their rotavaps, so a rotavap cannot be used for air and water-sensitive materials unless special precautions are taken i.e. additional traps are used. In the lab, the house vacuum line, a circulation bath or a membrane pump are used as source for the vacuum (40-50 torr). The fact that a vacuum is usually applied to the setup means that the boiling points of the solvents are going to be significantly lower than at ambient pressure (see table below).

Since the flask is rotated during the evaporation process, the surface area is larger than normal which increases the evaporation rate significantly. The solvent is collected in a flask and can properly be disposed off afterwards (organic solvent waste). In addition, this method also avoids overheating of the target compound i.e. oxidation because lower temperatures are used. The same rules like for vacuum filtrations apply here in terms of the glassware and other precautions i.e no cracks on the flask, etc.

Uses:

1. Distilling solvents
2. Vaporizing of solvents
3. Recrystallization

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4. Synthesis and cleaning of chemicals
5. Soxhlet extractions
6. Drying powders by means of the drying flask

Components:

Cooling area: the solvent vapor flows very quickly into the condenser. Here, the energy in the solvent vapor is transferred to the cooling medium, so that the solvent condensed.

Receiving flask: It receives the condensed solvent.

Vacuum: the vacuum reduces the boiling temperature and thus increases the distillation performance. The evaporating performance is influenced by the vacuum, heating bath temperature, and rotation speed and size of the evaporating flask.

Bomb calorimeter

The calorimeter used to determine the energy change during a reaction accurately is known as a bomb calorimeter. The modern Bomb calorimeter is a development of the original calorimeter of Berthelot. The bomb calorimeter is an instrument used to measure the heat of reaction at a fixed volume and the measured heat which is called the change of internal energy (ΔE). In chemistry, the changes of heat of a reaction can be measured at fixed pressure or volume.

The calorific value (heat of combustion) of a sample may be broadly defined as the number of heat units liberated by a unit mass of a sample when burned with oxygen in an enclosure of constant volume. I

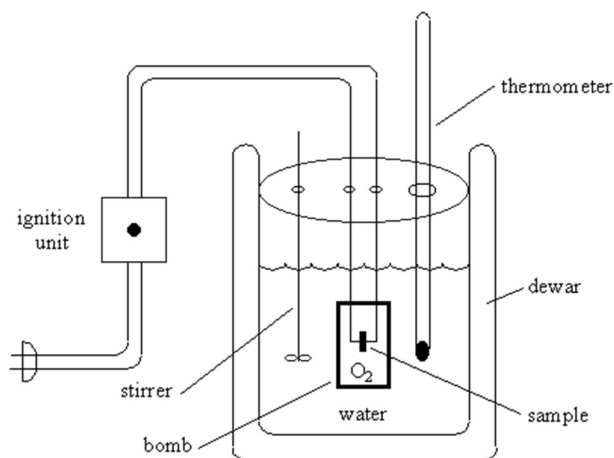


Fig. 3. Construction of bomb calorimeter.

The bomb calorimeter consist primarily of the sample, oxygen, the stainless steel bomb, and water. The dewar prevents heat flow from the calorimeter to the rest of the universe, *i.e.*,

$$q_{\text{calorimeter}} = 0$$

Since the bomb is made from stainless steel, the combustion reaction occurs at constant volume and there is no work, $w_{\text{calorimeter}} = -p dV = 0$

Thus, the change in internal energy, DU , for the calorimeter is zero

$$DU_{\text{calorimeter}} = q_{\text{calorimeter}} + w_{\text{calorimeter}} = 0$$

The thermodynamic interpretation of this equation is that the calorimeter is isolated from the rest of the universe.