



INDIAN INSTITUTE OF TECHNOLOGY BOMBAY

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BRIJRAJ CHANDRA MANSINGH CHEMICAL INSTRUMENTATION LABORATORY

ब्रिजराज चंद्र मानसिंह रासायनिक यंत्रीकरण प्रयोगशाला



INDIAN INSTITUTE OF TECHNOLOGY BOMBAY



ABOUT THE LABORATORY

The Chemical Instrumentation Laboratory in the Department of Chemistry, Indian Institute of Technology Bombay has been established in the memory of my father, Shri Brijraj Chandra Mansingh.

The family of Mr. Mansingh wishes to thank the Dept. of Chemistry and the Office of Alumni and Corporate Relations for their immeasurable help in the setting up of this Laboratory. This project involved a lot of hard work and dedication and it is very fulfilling to see this vision come to fruition.

My father retired as General Manager - Chemistry, Head of the Chemistry Division from the Oil and Natural Gas Corporation (ONGC) in 1993. He was a devoted lifelong student of Organic and Inorganic Chemistry. He completed his post-graduation in Oil Chemistry and Petroleum Technology from Harcourt Butler Technical University (formerly HBTI) - Kanpur, in 1957 and was awarded the gold medal for topping his batch. Over his long and distinguished career at ONGC, from 1958 to 1993, he made several vital contributions to the growth and success of the Chemistry Division at ONGC.

He was very proud of his association with the field of Oil Chemistry and he was a highly regarded expert in the study and development of Oil chemicals and drilling fluids.

The four years that I spent at IIT Bombay have left me with indelible experiences and fond memories. And IITB has played a foundational role in shaping my professional career. I am humbled to be a part of the extended family of alumni that stay connected to our beloved institution and that are motivated to give back in so many different ways. And I am blessed to have this opportunity to honour the memory of my father, who encouraged and inspired me in everything I do.

I sincerely hope that this Laboratory can help the next generation of young and brilliant students ... as they pursue their scientific curiosity and their passion for knowledge.

Sanjay Mansingh

(B. Tech, Electrical Engineering, Class '92)



The Department of Chemistry, IIT Bombay has grown, from the seed planted in 1965, into a premier center for advanced learning. The department has a faculty strength of 39 and offers Ph.D., M.Sc., and B.S programs. Over the years, the department has gained strength in both fundamental and applied aspects of chemistry, with emphasis on emerging areas like Green Chemistry, Biological Chemistry, Sustainable Chemical Practices and Chemistry of Materials. While the key focus of the department is on fundamental research, several research groups also interact regularly with industry and address problems related to chemical applications. The vibrant research atmosphere has attracted the crème-de-la-crème among students in Chemistry at all levels. As of now, we have more than 368 active Ph.D. students on roll, along with 105 M. Sc. and 128 B. S. students. In more recent times, we have become a popular destination for postdoctoral researchers as well, thanks to the attractive, highly competitive and prestigious Institute Postdoctoral fellowship (IPDF) and National Postdoctoral Fellowship (NPDF) of the Science and Engineering Research Board (SERB). The phenomenal research output of the department is manifested in the large number of research articles published every year in high impact, peer-reviewed journals. Faculty members serve as editors and members of editorial boards of prominent journals such as Chemical Science, WiRES, ACS Catalysis, Chemistry – A European Journal, Chemistry – An Asian Journal and more. Faculty members have also been recognized by several awards and fellowships, nationally as well as internationally. Students and postdoctoral fellows have gone on to become mature, independent researchers across the world.

Experiments facilities are most crucial for a research-intensive department like ours, which strives to ensure that the students are well-trained in the best practices of Chemistry and are capable not only of making new molecules and materials, but also of characterizing them precisely and devising applications thereof. Over the last couple of decades, the chemistry department at IITB has become well equipped with almost all instruments that are essential in a modern research-oriented center and students receive hands-on training on instruments such as 400 and 500 MHz NMR high-resolution mass spectrometers, chromatographs, bench-top AFM, scanning electron microscope (SEM), multi angle light scattering instrument (MALS), nanosecond, picosecond, and femtosecond laser spectroscopic setups and fluorescence microscopes capable of probing single molecules and particles, X-ray diffractometers, Electron Paramagnetic Resonance (EPR) spectrometer and various other instruments.

While the list of existing equipment is impressive, there is a constant need for removal of obsolescence and upgradation of our arsenal with new equipment, in order to keep pace with the growing demand due to the ever-increasing student strength. This is where the newly founded Brijraj Chandra Mansingh Chemical Instrumentation Laboratory is set to play an important role. Built with a generous funding from Shri Sanjay Mansingh, this state of the art facility will cater to more than 1000 B. Tech, 200 BS/MSc and more than 350 Ph. D. students. It will be available for use of all students and researchers of the institute, through an online booking portal. It is envisioned that the facility will go a long way in encouraging undergraduate students, who undertake research projects under the guidance of faculty members of the department. The advanced and sophisticated instruments of the facility will enable students to conduct thought-provoking experiments and cutting-edge research, asking questions that were never asked before and thus pushing the frontiers of our understanding of Chemical Sciences.

LIST OF EQUIPMENTS IN THE LABORATORY

Fast Kinetics:
Flash Photolysis

TLC- Mass
Spectrometer

Electron Spin
Resonance (ESR)
Spectrometer

Absorption
Spectrophotometer

Fluorescence
Spectrophotometer

ATR-IR
Spectrophotometer

Spin Coater

Contact Angle
Measurement
Apparatus

Cyclic Voltammetry

Polarimeter

Magnetic
Susceptibility
Measurement

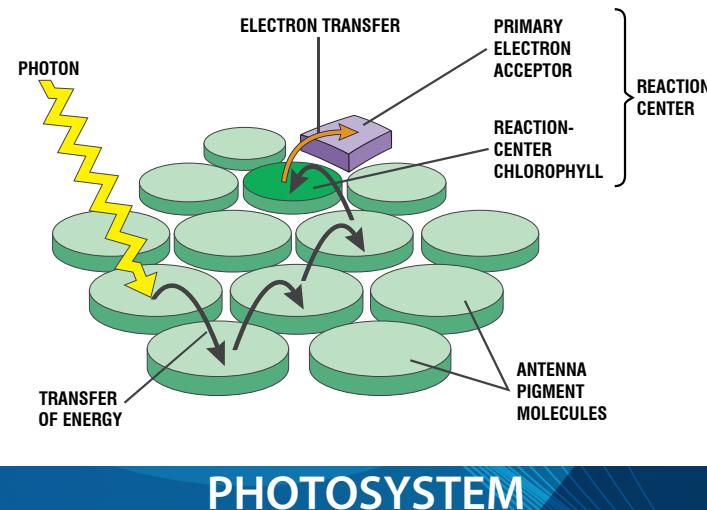
Gas
Chromatography

The instruments in blue are set up through the donation by Shri Sanjay Mansingh.
Those in black are procured from institute funds.

FAST REACTIONS: FLASH PHOTOLYSIS

Preamble

Many reactions in nature occur at an extremely fast time scale and are also complex. These reactions are not resolvable in the time scale that humans can act. For instance, the light-harvesting system of the photosynthetic reaction center, which is at the heart of the photosynthesis process, occurs in the femtosecond time scale (a femtosecond is one-millionth of one-billionth of a second). Similarly, the early chemical events in the process of vision occur in the picosecond (one-millionth of one-millionth of a second) time scale. The time scale of early events in the photosynthetic and vision process initiates a series of very complex processes, which ultimately leads to an event that occurs on the time scale that humans can decipher without any aids. New studies indicate that "the blink of an eye" could be as low as 15 milliseconds. Apart from these examples, a vast number of reactions, either initiated or uninitiated by light, occur at timescales that need special tools to investigate, to which this facility is geared.

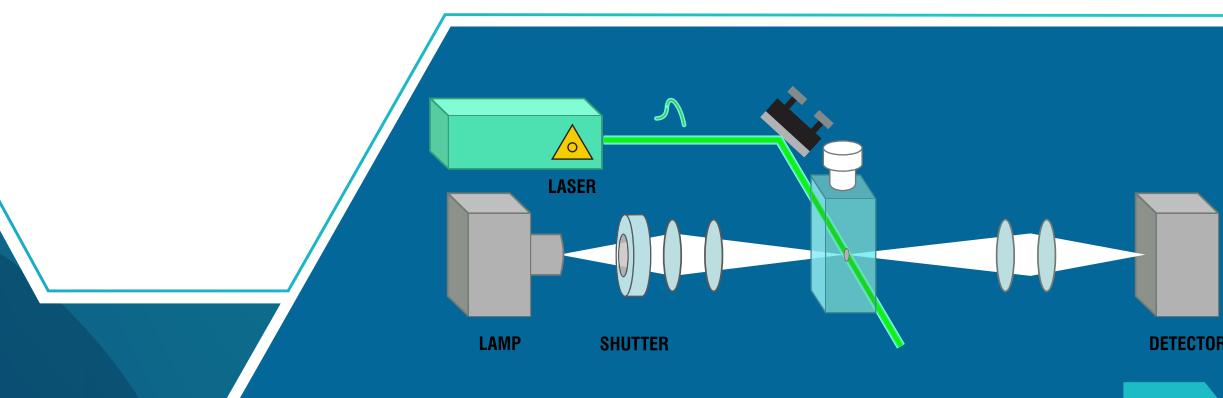


The instrument

The timescale of a reaction is measured relative to a triggering event, which could be as simple as adding two reacting chemicals. Alternatively, a large class of reactions occur due to a "photo-trigger", and are classified as photochemical reactions. The initial events in a photochemical reaction are known to cover a large time window at least of about ten orders of magnitude (femtoseconds to microseconds) thus making it impractical for a single instrument to cover this entire dynamic range. The LASER FLASH PHOTOLYSIS facility is capable of probing light-induced events that occur on the time scale of nanoseconds (one-billionth of a second) to microseconds (one-millionth of a second). A generic version of the instrument consists of three major components, which are (i) photolysis laser, (ii) probe unit and (iii) detection unit. The three units are in turn connected by a master controller which also acts as a time-keeper.

Experiments

One illustrative example, among many experiments that are planned for UG/PG/PhD students, is the photo-redox reaction of quinone coupled with proton transfer. In this reaction quinone upon excitation with light is converted to hydroquinone through an intermediate semiquinone. This is also a classical example of consecutive reactions, wherein the concentration of the intermediate semiquinone builds up in the first half and decays in the second half. This facility is aimed to teach and train students to understand the concepts in light-induced reactions.



Preamble

Thin layer chromatography (TLC) is one of the widely used tools by synthetic chemists to monitor organic and organometallic reactions in the laboratory. These reactions are widely used to access pharmaceuticals, perfumes, flavoring agents, agrochemicals, laboratory chemicals etc. Typically, TLC gives a qualitative idea on the product formation, however, to ascertain the identity of the products, further purification and spectroscopic (IR and NMR)/ spectrometric characterization are necessary. One of the widely used spectrometric technique used to determine the molecular weight of organic molecule is mass spectrometry (MS), which can be combined with TLC to accelerate reaction discovery. Therefore, exposing BS/MS students to such an important analytic tool will strengthen their skillset required for further academic training or securing industrial jobs. Moreover, the instrument will be useful for Ph.D. students in their research projects.

The instrument

The TLC MS is an automated system which analyses the compounds separated using TLC by mass spectrometry. The system procured from Advion, USA uses a patented technology. An in-built plate reader (Plate Express) provides a simple, automated means of visually pinpointing and

extracting compounds from a range of TLC plate formats into a compact mass spectrometer (CMS). The mass detector uses electrospray ionization technology (ESI) to detect molecular ions having m/z up to 1200 amu. Synthetic organic, natural product and biomolecules such as peptides quickly analyzed without additional sample preparation utilizing TLC/MS.

Experiments

The TLC/MS will be utilized in large number of UG and PG laboratory courses, which involves the synthesis, extraction and characterization of organic compounds. Some of the experiments include preparation of a fluorescent dye for freshman students across the institute; extraction of Caffeine from tea leaves; experiments involving Grignard reaction, Wittig reaction, Pechman Condensation, radical coupling reaction, Suzuki-Miyaura reaction, solid phase peptide synthesis etc. for BS/MS/Ph.D. students.



Understanding the Electronic Structure of metal complexes: Electron Paramagnetic Resonance (EPR)

Preamble

The structure-activity relationship is one of the important aspects to have control over the property of the targeted complexes and to fine-tune the structure of the complexes to realize the improved activity in many research areas such as Chemistry, Biology, solid-state physics, semi-conductors, display materials, catalysis, spin Tapping, etc. For example, to understand the mechanism of a catalytic reaction (either a radical or non-radical pathway), nature of the excited state involved in the various bio-mimetic and metalloenzyme oxidation reaction need to be understood. The transient intermediates that are generated in many reactions, protein folding, display devices (light or temperature or pressure-induced) are some of the cutting-edge research areas. Electron Paramagnetic Resonance (EPR) spectrometer helps to study these intermediates and various associated processes.

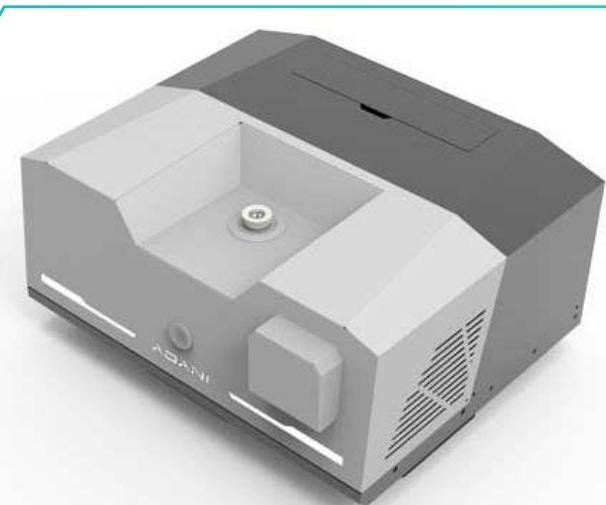
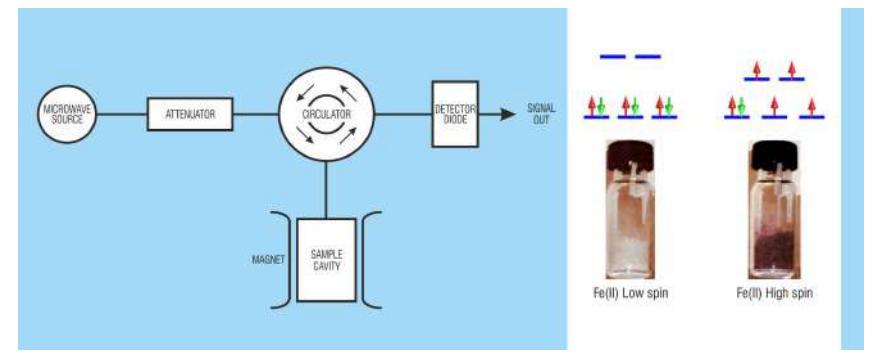
The instrument

EPR instrument is mainly used to characterize the paramagnetic complexes with unpaired electrons and organic radicals. Unlike nuclear magnetic resonance (NMR), there is always an

element of hesitation and hurdle among the students to understand, analyze and interpret the data that arises from the EPR, due to the complication arising from the g-anisotropy, hyperfine interaction, and magnetic anisotropy in certain transition metal complexes such as Fe(II). Therefore, we aim to introduce this sophisticated analysis at their preliminary level to make the students realize the potential applications of these instruments besides teaching the basic principles, analysis, and interpretation of the EPR data. The generic instrument consists of four major components 1) Microwave resonator 2) Microwave bridge 3) Magnet system 4) Control electronics.

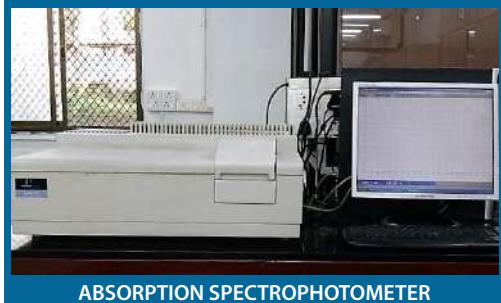
Experiments

Among the many experiments planned for UG/PG/Ph.D., students, one illustrative example is the temperature-dependent spin state change of the Fe(II) complex i.e. temperature assisted spin Crossover (SCO) of $[\text{Fe}(\text{NH}_2\text{trz})_3]\text{Br}_2$ (where NH_2trz = 4-amino-1,2,4-triazole) phenomenon. The SCO complexes are envisaged as molecular-based information storage devices, molecular switches, and display materials, etc.,. At room temperature, the Fe(II) complex exists as a high spin ($S = 2$), paramagnetic while at low temperature (77 K) the same complex exists as a low spin ($S = 0$), diamagnetic complex. EPR instrument is aimed at teaching how to determine the electronic structure of both spin states from their EPR spectral features, besides the fundamental theories of the instrument.

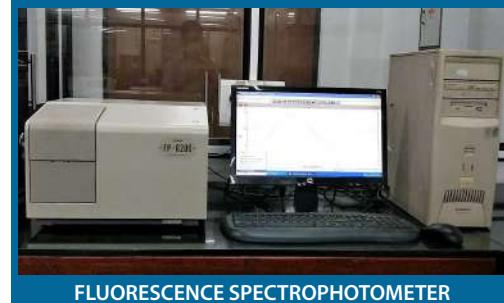


OTHER INSTRUMENTS IN THE LABORATORY

BRIJRAJ CHANDRA MANSINGH
CHEMICAL INSTRUMENTATION LABORATORY



ABSORPTION SPECTROPHOTOMETER



FLUORESCENCE SPECTROPHOTOMETER



POLARIMETER



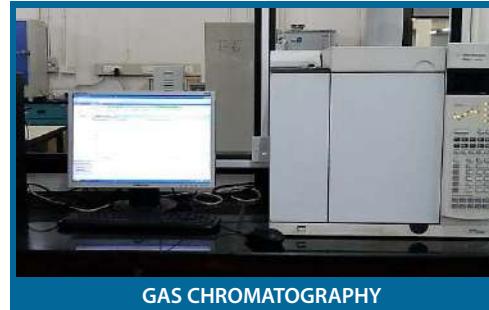
MAGNETIC SUSCEPTIBILITY MEASUREMENT



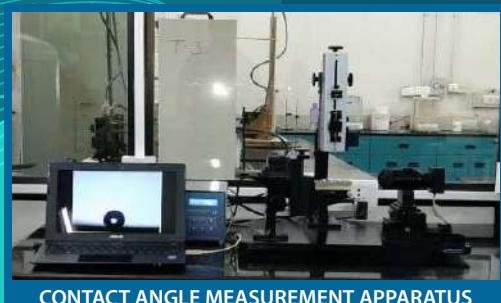
ATR-IR SPECTROPHOTOMETER



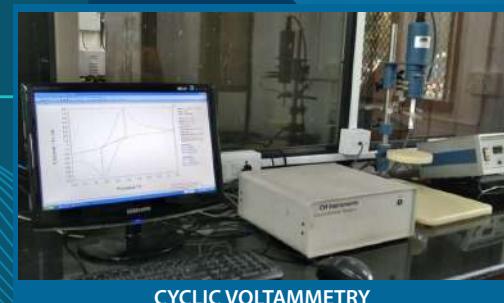
SPIN COATER



GAS CHROMATOGRAPHY



CONTACT ANGLE MEASUREMENT APPARATUS



CYCLIC VOLTAMMETRY

OUR LABORATORY

