MEETING REPORT

First refresher course in experimental chemistry*

In recent years, under the initiative of its Science Education Panel, the Indian Academy of Sciences, Bangalore has organized a number of Refresher Courses in Experimental Physics at various places to help the undergraduate and postgraduate teachers acquire hands-on experience in some experiments in physics. In these courses, which were mostly at the M Sc level, the participants assembled simple electronic kits and conducted experiments using them.

The First Refresher Course of the Academy in Experimental Chemistry was recently held at the University of Hyderabad. This course was formulated keeping in view the need to strengthen experimental chemistry programmes at the undergraduate and postgraduate levels in the country. Twenty university/college teachers were selected from different parts of India for this course. Sixteen of them actually participated in this course. All the resource persons of the course were from the School of Chemistry, University of Hyderabad. They not only designed the experiments, but also directly supervised the laboratory work and lectured on contemporary research topics in chemistry. The course involved experiments covering all major branches of chemistry. The experiments were designed such that the chemicals and minor equipments used in the course were inexpensive and amenable for easy implementation at the college/university level. The course comprised 12 laboratory experiments, 13 lectures and one demonstration experiment. The laboratory experiments were broadly categorized as follows: three in organic chemistry, three in inorganic chemistry, three in physical chemistry, two in materials chemistry and one in computational chemistry. Each day started with a lecture by a resource person giving a brief outline of his research activities followed by detailed instructions for the laboratory experiment scheduled for the day. Each participant was provided a laboratory coat, a pair of safety goggles and a book on experimental chemistry (*General Chemistry Experiments*, Anil J. Elias, Universities Press, 2002) prior to the commencement of the course.

Since it was the first time that the Academy was organizing a course in experimental chemistry, no structure/model was available. It was indeed a difficult exercise to decide upon the experiments to be included in the course that had a limited duration of two weeks. The problem was compounded by the general nature of the course, which was expected to cover all the major branches of chemistry and hence required active involvement of resource persons from all areas of chemistry. The timing of the course was decided taking into consideration the availability of the laboratory space (two weeks at a stretch) between two semesters. The summer vacation could have been the other alternative.

The course began with a welcome address by the Course Director, A. Samanta, who briefed the candidates about various activities of the Academy, including this new initiative and summarized the salient features of the course content. The course was formally inaugurated by M. Periasamy, Dean, School of Chemistry. Considering the utmost importance of safety in a chemical laboratory, the course began with a lecture on laboratory safety. K. C. Kumara Swamy discussed at length incidents of common accidents in the laboratory, citing several case histories and precautions to be taken to avoid such accidents. He stressed upon the practices that must be adhered to inside a chemistry laboratory (such as wearing a laboratory coat, safety goggles, shoes, etc.). He also emphasized the need for maintenance of proper laboratory records. The lecture was followed by an outdoor demonstration of the use of different types of fire extinguishers, blankets, etc. in extinguishing accidental fire in the laboratory.

The post-lunch session on the first day was devoted to the only demonstration experiment of the course in which the participants were acquainted with a single crystal diffractometer. In this experiment, P. Raghavaiah demonstrated the use of the diffractometer and explained the key steps involved in crystal structure determination. He also covered specialized applications such as determination of the Miller indices of crystal planes, a concept familiar in the undergraduate curriculum.

The first experiment, which was one of the three experiments in organic chemistry designed with the major theme of 'preparation, purification and characterization of organic compounds', started after the morning lecture by D. Basavaiah. This experiment consisted of preparation of 1,5-diphenyl-(E,E)-1,4-pentadien-3-one, a carbon–carbon bond formation reaction through aldol condensation between benzaldehyde and acetone. Progress of the reaction was monitored by TLC. The product was purified by recrystallization and yield of the reaction was determined. Purity of the sample was checked by TLC measurement and determination of R_f value. This was followed by determination of the melting point of the product and characterization of the product by infrared, ^{1}H NMR and ^{13}C NMR spectroscopic measurements. Apart from D. Basavaiah, L. Guruprasad, D. B. Ramachary and R. Nagarajan were associated with this experiment as resource persons.

The first inorganic chemistry experiment scheduled for the next day commenced after the lecture by Kumara Swamy. This experiment involved preparation of *cis*- and *trans*-bis(glycinato) copper(II) and tris(thiourea) copper(I) complexes. The experiment was introduced to highlight geometrical isomerism in coordination complexes, taking examples of simple square planar copper(II) compounds. The expectation that the less symmetrical *cis* form will have more number of observable vibrational modes compared to the *trans* form in the IR spectrum was also illustrated. Stabilization of softer Cu(I) [compared to Cu(II)] by a soft sulphur donor ligand was illustrated by means of the *tris*(thiourea)Cu(I) complex.

The second experiment of organic chemistry dealt with preparation of 2-phenylpent-4-en-2-ol. The reaction involved addition of allyl zinc, prepared *in situ* by treatment of zinc with allyl bromide in aqueous NH_4Cl solution, to acetophenone to yield the desired product. The morning lecture was delivered by Basavaiah and the experiment was supervised by Basavaiah, Ramachary and Nagarajan.

*A report on the Refresher Course in Experimental Chemistry of the Indian Academy of Sciences held at the University of Hyderabad during 27 November–10 December 2006 and organized by the School of Chemistry, University of Hyderabad, a Centre for Advanced Studies of the University Grants Commission.

Realizing the growing importance of computational tools in understanding the chemical problems and interpretation of experimental results, the next day was devoted to computational studies carried out on personal computers using commercially available software, Gaussian-03 run on LINUX platform. S. Mahapatra gave an introductory lecture on the basic aspects of theoretical and computational chemistry. M. Durga Prasad and Mahapatra supervised the computational chemistry experiments, which consisted of geometry optimization of molecules, determination of the stabilities of various possible conformers of molecules, calculation of the barrier involved in the transformation of geometrical and conformational isomers and calculation of the normal modes of vibration, their symmetries and vibrational frequencies. The calculations were performed at the B3LYP/6-31G level.

An experiment on synthesis of polymeric materials was included in the course. In this experiment, polystyrenes of three different molecular weights were synthesized by emulsion polymerization using different concentrations of sodium dodecyl sulphate as an emulsifier in the reaction mixture. T. Jana, who designed this experiment, also served as the resource person. In a subsequent experiment, the first in physical chemistry, the molecular weights of synthesized polystyrenes were determined using viscosity measurements. Again Jana served as the resource person.

The second inorganic chemistry experiment consisted of synthesis of alum from aluminum foil. In this simple experiment, which was supervised by S. K. Das, the amphoteric nature of aluminum was exploited. The steps involved in this synthesis were dissolution of aluminum in concentrated aqueous solution of KOH followed by its treatment with concentrated H_2SO_4 solution. The precipitate of $Al(OH)_3$, which formed on initial addition of H_2SO_4, redissolved in acid to yield a clear solution. On cooling, this solution yielded crystals of alum.

One of the physical chemistry experiments included in this course was determination of the pK_a value of an indicator, methyl red, using colorimetric/spectrophotometric measurements. The experiment involved determination of wavelengths at which the acidic and basic forms of the indicator had maximum absorbance, verification of Beer's law and determination of the relative amounts of the two forms of the indicator in solution as a function of pH. M. J. Swamy and A. K. Bhuyan were the resource persons. The former also delivered the morning lecture of the day.

The third physical chemistry experiment dealt with principles of chemical kinetics. In the classic experiment, 'iodine clock reaction', for which T. P. Radhakrishnan served as the resource person, the kinetics of the oxidation of I^- by H_2O_2 was studied. By an ingenious trick of converting the I_2 formed, back to I^- using $Na_2S_2O_3$, the concentration of I^- is kept constant so that the reaction rate was controlled essentially by the concentration of H_2O_2. By successive addition of small amounts of $Na_2S_2O_3$, several different runs of the experiment were mimicked in a single run.

The other experiment scheduled for the day was fabrication of silver nanoparticles by reduction of silver nitrate using glucose, followed by addition of starch as a stabilizing agent. In this experiment, which provided a direct experience of nanochemistry in a 'green' environment, the light-yellow colloidal solution obtained was characterized by recording its electronic absorption spectrum that shows the plasmon absorption band at ~ 415 nm. A control experiment where addition of starch is omitted, shows the formation of bulk silver in the form of a shining film, the well-known silver mirror reaction. This experiment was designed and supervised by Radhakrishnan.

Although experiments involving titrations are in the syllabi of most of the undergraduate courses, precipitation titration as a method of quantitative analysis has not found its place in most college curricula. Taking into consideration this aspect, we introduced an experiment on the estimation of silver by this method, which is known to give reliable results. Kumara Swamy gave a lecture on various aspects of quantitative analysis, including the structural aspects of colour changes of the indicator prior to commencement of the experiment. He also served as the resource person along with Das.

Synthesis of isoborneol and borneol by stereoselective reduction of D-camphor using $NaBH_4$ was the third organic chemistry experiment of this course. In addition to the preparation and characterization of these systems, the participants studied the optical rotation of the purified products. Ramachary and Nagarajan were the resource persons.

On the concluding day of the course Samanta delivered the morning lecture. In the interaction session that followed, he had a detailed discussion with the participants on various aspects of this course. The course was concluded after obtaining feedback from the participants. The feedback data suggested that the participants found this course novel, interesting and beneficial. Requests have been received to organize this type of course more frequently. Few suggestions on inclusion of additional experiments and to increase the duration of the course by one more week, have also been received.

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