Microscale Experiments in Chemistry – The Need of the New Millennium

1.Newer Ways of Teaching Laboratory Courses with New Apparatus

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Jonathan Swift was in a fantasy world when he wrote the old classic *Gulliver's Travels*. Perhaps, he knew that sometime in future, chemists would use the 'Liliput' scale for performing laboratory experiments. The Kaurava prince Duryodhana, denying any claims of territory to the Pandavas, categorically declared that he would not yield to them even that grain of dust, settled at the tip of a vibrating needle. Probably he realised that even that little particle could be used for doing many experiments! "Small is beautiful", it is said. "Green is more beautiful" – would be agreed upon more easily. While combining these two ideas in chemistry laboratories of teaching institutes, we recently realised that time has come to replace the regularly conducted chemistry experiments in our educational institutions, strictly to the smallest possible scales.

Chemistry has always been an experimental science. Even from the days of alchemists, it was anticipated that every statement of each scientist should be validated through experiments. In the schools, it is desired that teaching of every chapter of theory in chemistry must involve simultaneous confirmation by commensurate experiments in the laboratories. This was always found wanting in our present education system. The main reason for this lapse is large-scale operation of the experiments. Whenever students work in the laboratories they perform the tests on a large scale involving cumbersome assemblies of apparatus and huge amounts of chemicals. Besides, disposal of the products, excess reagents and solvents, without harming the environment, presents considerable difficulties. Even today, the chemistry laboratories in academic institutions are always stocked with huge flasks, big bottles and are fully replete with toxic gases and vapors of obnoxious chemicals. Health hazards in such laboratories to the students, teachers and laboratory staff have been a matter of concern to many of us for a long time. A tug-of-war had also been going on in recent years, to balance the budgets of running practical courses and yet maintaining the high standards of training by asking the students to do more complex experiments. Since the economy is always the winner, the number of experiments and the chemicals used therein, had been the losers! The increasing number of students and perpetually rising costs and consumption of chemicals and glassware add a heavy charge on the budgets of running the practical classes.

Sometime during the nineties, the concern about the tremendous wastage of chemicals in educational institutions became more vocal. A few books on small-scale experiments in chemistry and the apparatus required to do these began to appear. Amongst the chemistry periodicals, the *Journal of Chemical Education* even initiated a section entirely devoted to the microscale methods.

Approach to these problems in India was, however, very scanty. In 1992, one of us (SLK) during the tenure as a Visiting Professor at the University of Wisconsin – La Crosse, saw the laboratory experiments using microscale techniques. The tiny apparatus was attractive enough for immediate adaptation in India. Some apparatus, such as tiny 5ml RB flasks, Hickmann head and condensers with B10 ground joints were made in adequate numbers for the use of students. In 1993, the preliminary techniques of microscale were taught successfully to the final year students of M.Sc. organic chemistry in this department for the first time. Without exception, in each experiment, all the starting materials, which used to be 5 to 7 g earlier, were reduced to less than 250 mg. The students were found to be quite at ease with these apparatus, the experiments and the results. During the annual social gathering that year, they even presented a skit based on the new technique. It was thought then that the methodology must be introduced to as many teachers of other institutes as possible so that they can implement these for their own students.

Ever since, at least 400 teachers must have performed these 'microscale' experiments themselves during various refresher courses or four-day workshops specially organised for the purpose of inculcating the technique. It is heartening to note, that each one of them, is convinced that the time has come to adopt these methods in our curricula. An overwhelming majority opined that it is not very difficult to introduce these in laboratories at all levels – even in the high schools.

The first consequence of the adoption of the new technique in our laboratory was that the use of chemicals and subsequently the expenses of running the practical classes were drastically reduced. Earlier, 20 students in our laboratory used up around 5 liters of diethyl ether every day. Now 500 ml is more than adequate. With smaller amounts to handle, the students now perform the experiments with more care. Their skills in handling the equipment are markedly improved. Time required for completing each experiment also decreased. Even though the reaction time would essentially be the same irrespective of the amounts of reactants, the time in setting up the reaction assembly, measuring the reactants, work up of the reactions, filtration, purification and drying of the product would now be reduced to a few minutes only. After working with this technique for the past few years, and introducing these at both the undergraduate as well as post-graduate levels in our university, it can now be confidently stated that the students can easily perform each and every prescribed experiment on a much smaller scale. Some experiments such as the determination of boiling points in capillaries, doing some reactions in capillaries, vertical distillations using the Hickman head and use of Pasteur pipettes were incorporated for the first time in the laboratory. Under the new set-up, even for a multi-step synthesis, the quantities of starting compounds provided to the students were restricted to not more than 250 mg. Derivatives of functional groups were now prepared starting with not more than 100 mg and the separations of mixtures with maximum of 2.5 g of a three component mixture – less than one gram of each component. The students could still determine the type of mixture, separate it chemically, purify the components, determine the elements and functional groups on that much quantity and yet submit about 200 mg sample of each component for inspection by the laboratory instructor. To gain confidence and to verify the results more often, they can now repeat the experiments as many times as they wish without any loss to the exchequer or of time.

Although new glassware need to be provided for microscale experiments the cost is not high. Today, at least 4 glass blowing vendors in Pune are ready to supply these apparatus to the interested institutes. It should be noted that one set of new equipment costs approximately Rs.1000 less than the set currently used (*Table 1*). Each educational institute spends specified amount on chemicals and glass apparatus every year. If the microscale experiments are introduced, the existing stock of all the chemicals would be adequate for at least five more years. The money saved in chemicals can easily be used for the purchase of glass apparatus. The scenario started changing slowly. As of today, University of Mumbai and Goa University have formally adopted the technique at least partially.

The first lesson of microscale teaching really begins with a new significant word – 'transfer'. We insist that nothing should be poured – either from bottle to test tube or from test tube to filtration assembly. We seldom realise that indiscrete pouring of chemicals, solutions or solvents, amount to additions of huge excesses. The students are to be reminded every now and then that they must use droppers for transferring liquids.

It would be of interest to know some of the apparatus which are used in the microscale experiments.

Open Ended Capillaries (Figure 1): Usually used for the determination of melting points, these can be now utilised for deter-

Description	Number	Cost/Item in Rs.	Description	Number	Cost/Item in Rs.
Beaker (50 ml)#	2	60	Beaker 25 ml #	2	86
Test tubes # 20×125mm @Rs11each	2	22	Test tubes # 15×125mm @ Rs 8 each	6	48
Test tube holder *	1	20	Glass funnel *	1	12
Conical flasks # 25 ml	2	83	Conical flasks # 10 ml	2	80
Asbestos sheet*	1	10	Wire gauze *	1	8
Pair of tongs *	1	25	Test tube stand *	1	35
Brush *	1	12	Thermometer *	1	35
Measuring cylinder 5 ml*	1	105	Bosch head *	2	80
Clamps *	2	190	Porcelain dish *	1	30
Tripod stand *	1	160	Water condenser#	1	80
Hickman head	1	105	Stainless steel spatula*	1	30
Aircondenser #	1	65	RB flask 5ml #	1	80
RB flask 10 ml #	1	95	Hirsch funnel #	1	20
Filter tube	1	20	Graduated pipette 1 ml	1	55
Pasteur pipette	3	30	Waterbath *	1	35
Rubber bulbs	3	30	Beral pipettes	10	10
Sand bath dish*	1	15	Sample tubes 1ml	5	30

* these items are already being used in laboratories even today and as such do not add to the costs.

these replace the much more expensive old apparatus.

Grand Total = Rs. 2000/set approximately

Table 1. Microscale apparatus required to be issued to students.

mining the boiling points and performing some of the routinely performed reactions, such as colour or group tests. The major advantage is that only a few micrograms of the chemicals are consumed. We are also suggesting the use of capillaries to make solutions by taking a few crystals of the reagents/chemicals at the tip of the capillary and introducing the other solution of liquid by touching its tip with a drop of solvent taken through a dropper. Solutions thus made, could be diluted or transferred to another capillary by just touching its downward end with the tip of another capillary. In this manner only the amount required is

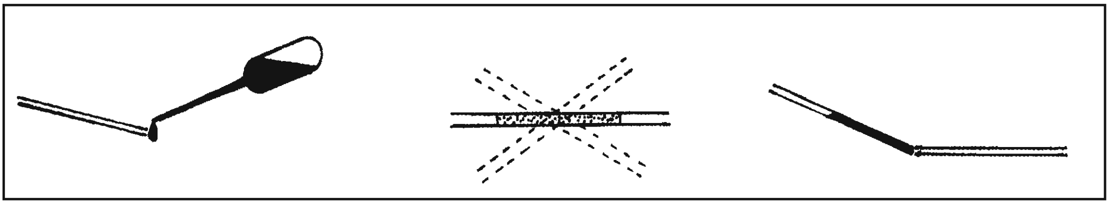

Figure 1. Use of capillaries.

made. This is extremely useful particularly in effective use of optimum quantities of expensive chemicals (e.g. as silver nitrate). A capillary of about 10 to 12 cm length made from hard glass would cost about 5 paise each. Even if a student uses 50 of these every day (which is very unlikely) during one laboratory class, the cost per student will be insignificant. Reusing the capillaries after washing can reduce this further.

Pasteur Pipettes (Figure 2): This is the most important apparatus in a microscale laboratory. These glass tubes with a rubber bulb at one end and a fine capillary drawn at the other are used in microscale laboratories for many purposes. These include transfer of solutions, filtration by either introducing a cotton wick at the narrow tip or by holding the tip tightly against the bottom of the test tube containing the liquid to be filtered and sucking the filtrate slowly, layer separation of two immiscible liquids, etc. They are more advantageous over the routinely used separating funnel as one can take out either the top or the

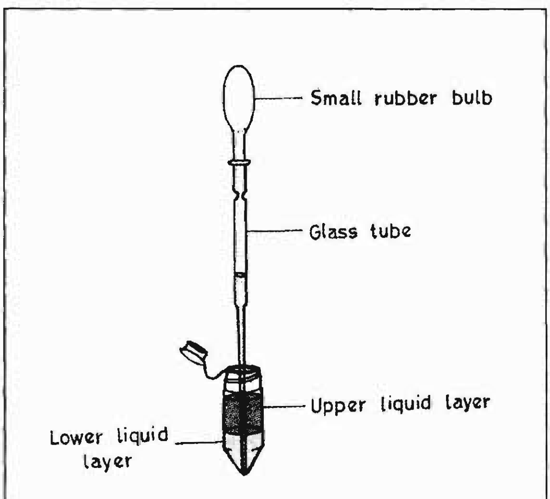

Figure 2. Use of Pasteur pipettes.

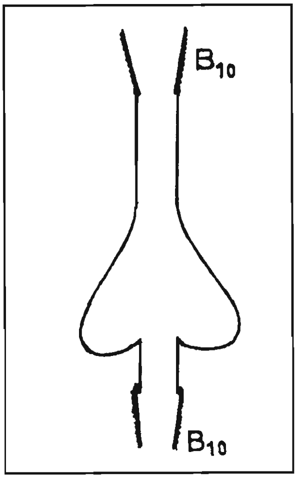

Figure 3. Hickman head.

bottom layer independent of each other. In a separating funnel, the bottom layer has to be taken out to retrieve the top one. The mixing of liquids is also done efficiently by agitating with the same Pasteur pipette. The students or laboratory staff can repair the broken tips by heating the narrow end of the tubes on a flame and pulling it, giving them first hand experience of glass blowing! A pipette with the attached rubber bulb costs Rs.10 as against a separating funnel costing Rs. 200.

Hickman Head (Figure 3): Formally it is used for collecting the distillate in the vertical distillation. It may be conveniently used to prepare reagents which are to be freshly prepared and distilled. Thus, just 1ml each of conc. sulfuric and conc. nitric acids are mixed in a 5 ml RB flask attached with Hickmann head and condenser and heated with a free flame. Fuming nitric acid is collected in the groove and excess sulfuric acid remains in the flask. The collected fuming nitric acid is taken out by a Pasteur pipette and used directly.

Beral Pipettes (Figure 4): These dropper-like pipettes made from low density polypropylene are manufactured locally at cost of just Re.1 per piece. These are unbreakable and very convenient for storing most chemicals particularly all table reagents. The capillary tubing at the tip does not allow the stored liquid to come out unless the bulb is squeezed. As such then the pipettes can rest horizontally on the working tables without any spilling. The volume of the drop that can be retrieved is just about 0.03 to 0.04 ml, thus making it possible to add very small quantities easily to any reaction vessel. The long drawn capillary can be inserted deep into the test tube and the contents delivered directly to the reactants. Occasionally these pipettes could be used to separate the layers of immiscible liquids and to remove last traces of liquids from flasks. Then usage of syringes and

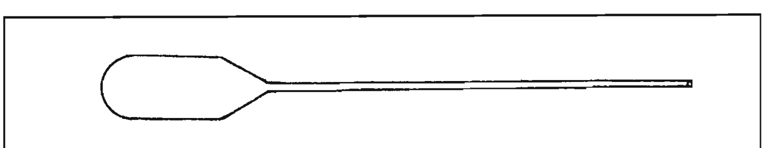

Figure 3. Beral pipette.

needles for transferring small amounts of liquids may be avoided. Thus it could be concluded that the regular chemistry experiments could be performed very easily on scales reduced to 10% of current usage saving in chemicals and the running costs, with less breakage, enhanced safety and a better environment.

The microscale chemistry is now the need, not only of the day but of this moment. In the subsequent parts of this series we would suggest a few experiments to illustrate this. If introduced at the proper level at proper times, the technique will lead to a great revolution in the activities in any chemistry laboratory.

An excellent review by Towse[2] published recently, includes most of the literature available on microscale experiments. K V Sane, S Bhanumati and Riyazuddin have also contributed to the development of microscale experiments.

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Suggested Reading

[1] S L Kelkar, D D Dhavale and B G Mahamulkar Ed., *J. Chem.*, 365, 2000.
[2] P J Towse, *Chemistry Education Research* (New Delhi), October 1998 and references cited therein.

"It seems to me that the integers have an existence outside ourselves which they impose with the same predetermined necessity as sodium or potassium"

C Hermite