

INSTRUMENTATION IN THE UNDERGRADUATE CHEMISTRY CURRICULUM*

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The word "instrumentation" is defined in Webster's New Intercollegiate Dictionary as, "a use of, or operation with, instruments." The dictionary includes several definitions of the word "instrument," but the one which is applicable to the present discussion is, "a measuring device for measuring the present value of the quantity under observation." Thus in considering instrumentation in the undergraduate chemistry curriculum we are concerned with the use of measuring devices.

RELATIONSHIP OF INSTRUMENTATION TO CHEMISTRY

Chemistry may be defined as the study of the composition, structure and properties of matter and the changes which it undergoes. Measurement is absolutely basic to all of chemistry for it is impossible to gain knowledge of matter without some means of measuring its composition, structure, and properties, and to recognize changes therein.

Measurement has been basic to the historical development of the knowledge which makes up the subject matter of chemistry. In fact, the rate of the development of this knowledge in each period of history bears a direct relationship to the variety, number, and precision of the tools of measurement which were available and which were employed in that period. The tremendous strides in developing and establishing chemical knowledge in the seventeenth century are directly attributable in large part to the availability and the use of the analytical balance, which is even today one of the most important instruments in chemical work. The greatly accelerated rate of chemical development in the current generation is due to a considerable extent to the variety, number, and accuracy of the tools of measurement which are now available and which are constantly being expanded and improved.

Measurement is basic, not only in the historical development of chemical knowledge, but also in virtually all areas of current research in chemistry. The idealized goal of research is the gaining of new knowledge, the refinement of current knowledge, or both. All scientific knowledge, with the possible exception of that on the most primitive levels, must be quantitative or, at least, semi-quantitative. And the gaining of quantitative knowledge is impossible without the tools whereby quantitative measurements can be made.

Measurement is, in addition, fundamental and basic in the practice and in the practical application of the knowledge which makes up the subject matter of chemistry. The tools of measurement are absolutely necessary in industry and in the home and wherever chemistry is practiced and chemical knowledge is applied. Therefore, instrumentation is intimately related to all of chemistry, in its historical development, in current research toward the expansion of chemical knowledge and in the application of that knowledge as well.

In viewing the very close relationship of instrumentation to chemistry, it is of utmost importance to maintain the proper perspective. Within chemistry, the instruments are the tools for achieving an end and are not the end in themselves. The instruments are merely the tools for use in the study of the composition, structure, and properties of matter and the changes which it undergoes.

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The role of instrumentation is easily subject to overemphasis on the one hand and to underemphasis on the other hand. Instruments are overemphasized in chemistry if they are considered to be the end in themselves. For example, the analytical balance, the spectrophotometer and x-ray diffraction instruments are not end-goals in chemistry, rather it is the knowledge to which measurements by means of these instruments can contribute that is chemistry. Instruments are underemphasized if chemical knowledge is studied without consideration of the tools whereby that knowledge has been gained. There is doubtless a proper place in scientific work for those specialist individuals who work solely in the theoretical realm with no consideration of the means whereby theory may be verified, refined, and applied. However, a general study of chemistry must include a study of the tools of measurement.

PLACEMENT OF INSTRUMENTATION IN THE UNDERGRADUATE CURRICULUM

In considering where instrumentation belongs in the undergraduate curriculum, it is well to base the consideration upon the core curriculum as adopted by the appropriate agency of the American Chemical Society. This does not mean that departures, even radical ones, from this recommended curriculum may not be in order either now or in the future, but it does represent a well-established basis for considering any particular part of the curriculum. The recommended program may be summarized briefly as follows:

- 1 year of general-inorganic chemistry
- 1 year of analytical chemistry
- 1 year of organic chemistry
- 1 year of physical chemistry
- advanced chemistry course(s) of 5 semester hours credit
- mathematics through calculus
- 1 year of physics
- foreign language
- social sciences, humanities, etc.

There are, in principle, three distinct means whereby instrumentation can be placed within this curriculum. First, instrumentation can be considered here and there throughout all of the courses in chemistry and physics. This is to say that, as the knowledge within the various areas of chemistry is studied, the tools of measurement whereby that knowledge has been developed and established can be studied simultaneously. This is done, of course, to a considerable extent in all of the science parts of the undergraduate curriculum. However this approach is hardly an adequate one insofar as instrumentation is concerned. The progress in this and recent generations in the development of unifying concepts has been so great that these concepts can be presented to good advantage early in the study of chemistry, even at times when the students are hardly ready to understand and to appreciate the means whereby those concepts have been developed.

The second means whereby instrumentation can be located within the undergraduate curriculum is to concentrate it in those courses which are particularly quantitative, that is, in physical chemistry and in quantitative analysis. Some attention is given to the tools of measurement in virtually all undergraduate courses in physical chemistry. However, the theoretical essence of the subject of physical chemistry precludes laying too much stress on the tools involved. Furthermore, the current trend in instruction in physical chemistry, particularly on the more advanced levels, is toward a more rigorously mathematical basis rather than to a more experimental basis. The place of instrumentation in the quantitative analysis course will be considered in a later section in some detail.

The third means whereby instrumentation can be located within the undergraduate curriculum is by introduction of a separate course in instrumentation, either as all or part of the recommended "advanced chemistry course" or as an

additional feature of the curriculum. This subject will also be considered in more detail in a subsequent section.

INSTRUMENTATION IN QUANTITATIVE ANALYSIS

The course in quantitative analysis is in a sense devoted to the methods of quantitative measurement of chemical composition and the practical and theoretical concepts underlying that which is measured. A quantitative determination generally includes a separation and a measurement. On the basis of the type of measurement, quantitative procedures are typically classified as gravimetric, volumetric, electrical and optical, often with a miscellaneous category added as well. In a one-semester course in quantitative analysis, it is customary to emphasize gravimetric and volumetric methods with relatively little consideration of electrical and optical methods. Therefore it is only proper to consider in some detail in this first course instrumentation as it applies to gravimetric and volumetric methods, particularly the analytical balance.

The second semester course in quantitative analysis does, in some institutions, continue the emphasis upon gravimetric and volumetric methods, perhaps with special consideration of more complex separations than are usually encountered in the first semester course. Even though much can be said in favor of a full year devoted primarily to gravimetric and volumetric methods, it seems that it should be entirely proper to give primary emphasis to electrical and optical methods in the second semester course in quantitative analysis. The designation of this course as one in "instrumental analysis" is somewhat of a misnomer, although this terminology is fairly well established already. Constant care is required lest the optical and electrical methods be considered as a subject apart from "classical" quantitative analysis, and the optical and electrical methods should, in fact, build upon the gravimetric and volumetric methods first encountered.

Many schools already offer a course of this type. It is usually placed in the curriculum after at least one semester of the course in physical chemistry. There seems to be no a priori reason, however, why this type of course could not directly follow the first semester of quantitative analysis. The earlier placement of the course would necessitate a less rigorous treatment of such topics as conductance of electrolyte solutions, but this should not be a prohibitive disadvantage.

If this course is designated as one of the two semesters of regular analytical chemistry, it must be taken by all full chemistry majors. If, however, the year of analytical work is filled by a separate course in qualitative analysis along with the first course in quantitative analysis, then the second course in quantitative work must be considered as part or all of the "advanced chemistry" recommendation in the American Chemical Society curriculum and must compete with several other courses in faculty planning and in student selecting. This topic will be discussed further in a later paragraph.

The subject matter of the course in optical and electrical methods becomes fairly well established, even though variation does properly exist from one institution to another. The optical methods include (1) those based upon the emission of radiant energy, as in arc, spark, and flame emission spectroscopy, (2) absorption methods as in colorimetry and spectrophotometry, (3) light scattering methods as in turbidimetry, nephelometry, fluorimetry, and phosphorimetry. All regions of the electromagnetic spectrum from radiowaves and microwaves to x-rays and gamma rays should be considered. Also, ion-optical methods such as mass spectrometry should be included. The electrical methods should include those based upon measurement of all common electrical quantities, such as potentiometric, conductometric, amperometric, polarographic, and coulometric methods. Along with the optical and electrical methods, it is well to include consideration of a number of miscellaneous methods such as chromatography (especially vapor

phase), thermal conductivity, radiochemical methods of analysis, and perhaps some others.

A wealth of literature is available in easily accessible forms for use in a course of this type. A half dozen regular textbooks are currently on the market. The annual review articles in the April issues of *Analytical Chemistry* provide excellent summaries of current work in each of numerous areas related to instrumentation as well as extensive listings of references. Regular feature sections on instrumentation have been included for several years in *Analytical Chemistry* and in *Industrial and Engineering Chemistry*. A new feature section has recently been established in the *Journal of Chemical Education* which provides excellent coverage of various areas of instrumentation which, perhaps, should be classed as required reading for all teachers and students of chemistry. In addition, the commercial development and manufacture of chemical instruments has become a very active field with the result that the several manufacturers provide excellent instructional information in their advertisements and in other literature which they freely provide.

The expense of equipment is often cited as a deterrent to the establishment of laboratory courses in optical and electrical methods of analysis. Much analytical equipment is expensive, to be sure, but it is possible to provide a reasonably adequate laboratory course in this subject within a budget entirely consistent with other instructional expenses. Relatively simple and inexpensive spectrophotometers for use in the visible region are available from some manufacturers, and the principles involved can be taught almost as well with instruments employing visible light as with ultraviolet light. Infrared spectrometers are still quite expensive for undergraduate teaching purposes, although less expensive instruments have recently come on the market. The simpler spectrophotometers can be used directly for turbidimetric measurements and, with some modification, for fluorescent and other types of work. A pH meter can be used for a variety of electrometric measurements, and need not be limited to simple potential measurements. Also, an inexpensive vacuum tube voltmeter which may be purchased in kit form for 26 dollars is very satisfactory for student use with several electrode systems and in numerous applications. A constant current supply for coulometry need consist of nothing more than a few batteries and a resistor. Homemade apparatus for gas chromatography can be very satisfactory, although a recorder such as the relatively inexpensive Varian models is very useful for this and some other types of work as well. Some of the equipment can be used for other courses and for research by students and staff, thus easing the financial burden attributable to this course. Furthermore, of course, it is not necessary to provide laboratory experience in every type of analysis discussed in the classroom.

Another possibility whereby the instruction can be carried out with a minimum of expense for laboratory equipment is to obtain actual data from other laboratories from which the students can make all necessary calculations and interpretations. For example, x-ray diffraction patterns, infrared spectra, and mass spectra form three types of data in which most of the instructional value lies in the treatment of the data, not in the running of the instruments. One company is currently planning to provide at low cost actual copies of sets of data of this type for instructional use.

Professors who have had experience with a course of this type almost invariably become enthusiastic about it. It provides much opportunity to teach chemistry, the initial fears of some persons notwithstanding. The quantitative approach to chemistry is extended beyond that of prior courses in the chemistry curriculum. Of considerable importance is the enthusiasm which the course generates among the students. Some students are "sold" on particular types of chemical work as a result of the course. All students, whether interested primarily in one area of chemistry or another, are able to recognize readily its importance to them.

SEPARATE COURSE IN INSTRUMENTATION

Two objections immediately arise when the possibility of a separate course in chemical instrumentation is suggested. The first is the feeling that chemistry courses should be courses in the subject matter of chemistry and not in the tools of measurement whereby that subject matter has been established and is applied. This viewpoint is generally valid, but it may be noted that the curriculum already does include "tool courses," such as those in mathematics and, in some respects, those in foreign language. Furthermore there is occasional precedence for a tool course within the chemistry curriculum as, for example, chemical microscopy. A second objection is the feeling that instrumentation is not strictly chemistry, so that if a special course is needed it should be provided in some other department just as the desired mathematics courses are provided by a separate department rather than within the chemistry department. This viewpoint is a reasonable one, but the fact is that such courses are generally not available in any other department. So if the chemistry faculty really feels the need of a separate course in instrumentation, it is almost forced to provide it. It may also be noted that the approach of physicists and engineers, for example, to instrumentation is not entirely the same as that of chemists, although there is no basic reason why this must be so.

Let us now consider from a positive standpoint the need for a separate course in chemical instrumentation. On a purely empirical and practical basis, much can be said in favor of such a course, simply because much of importance is left out of the regular courses in the curriculum. A chemistry student can successfully complete the usual undergraduate curriculum and have little or no familiarity with such topics as electronics, vacuum gauges, thermoregulators, self-balancing potentiometers, indicating meters, optic design in instrumentation, and even such terminology as "input impedance" and "transconductance." This deficiency points to a possible need for a separate course in instrumentation.

On a more fundamental basis, there are basic principles widely applicable in virtually all measuring operations. Unless these basic principles are considered and used, the whole subject of measurement and instrumentation is nothing more than a hodge-podge of largely unrelated items. It is hardly appropriate to take the necessary time in one of the regular courses in the basic areas of chemistry to consider the underlying principles of instrumentation, so the possible need for a separate course is indicated.

It may be claimed with considerable justification that there is a science of instrumentation. The time may come when separate departments or divisions of instrumentation will be established, with full professional majors therein. It is interesting to note the present existence on a high scientific level of societies and journals which specialize in instrumentation.

If a separate course is to be provided in chemical instrumentation, where does it fit into the curriculum? Inasmuch as instruments are tools of measurement, the course perhaps could come early in the curriculum. However, the student generally gains little appreciation of a tool until he has some concept of what it is for. Therefore it appears that the best place for the course in instrumentation is on the advanced course level, perhaps as all or part of the five hours recommended for advanced chemistry courses in addition to the four regular year-courses. This placement permits a proper building upon the material of the other chemistry courses, particularly with respect to the possible transducers encountered therein.

The instrumentation course must compete in faculty planning or in selection by the students with other advanced chemistry courses, or in both of these. The same place in the curriculum is often filled by such courses as organic qualitative analysis, advanced inorganic chemistry, a third semester of physical chemistry, biochemistry, senior research, and even by a course in "instrumental analysis" if the latter is not counted toward the two semesters of basic analytical chemistry. Every one of these courses is of considerable value. Yet it would be decidedly

unrealistic to claim that any one of them is absolutely the best one for all undergraduate students of chemistry. It does seem, however, that the case for a separate course in chemical instrumentation is sufficiently strong to warrant consideration along with the other possibilities when faculties plan which advanced course or courses are to be offered and when students and their advisers select which of the available courses shall be taken. All of these courses, including the one in chemical instrumentation, may be on what is commonly designated as the "advanced undergraduate-graduate" level. Those students who do go to graduate school generally get some of these courses in their graduate work. Some who do not continue into graduate school may profit more from one of these courses than another, but one in chemical instrumentation surely warrants some consideration. In no case is it feasible for a student to take every course from which he can receive some benefit. The responsibility to learn and to achieve must ultimately rest upon the individual, and no curriculum can in any sense provide a complete mechanism for the fulfillment of that responsibility.

The specific development of separate courses in chemical instrumentation has not yet proceeded very far. Circumstances would doubtless cause variation in the course outline from one situation to another. Among the main topics to be considered, however, would likely be transducers, electronics, servo-mechanisms, indicating meters, recorders and controllers, and perhaps some optics and pneumatics. A transducer may be defined as any device which will convert phenomena under investigation to an appropriate signal. Over 500 different types of analog transducers are now available, but there are relatively few types of output signal. The output signal is often electrical, consisting of a voltage, a current flow, a resistance, a capacitance, an inductance, although it need not be electrical. There possibly are millions of electronic circuits available in practice and in possibility, yet much is quite standard insofar as the fundamental operations are concerned. The course in instrumentation should include consideration of the major components of electronic devices and of the principle types of circuits encountered. A servomechanism may be considered to be a null or balancing device, whereby the effect to be measured is opposed by an equal and opposite effect automatically and continuously. In theory and in practice, a study of servomechanisms provides a unified and general method of attacking most classes of measurement. It should be noted that current trends in instrumentation go beyond the mere improvement of classical techniques of chemical measurement, and this fact accentuates the need for a separate course in the principles of instrumentation.

SUMMARY

Instruments are the tools of measurement whereby the knowledge which makes up the subject matter of chemistry is obtained and established and whereby chemical knowledge is applied. Therefore, instrumentation is intimately related to all of chemistry and must occupy a prominent position within the undergraduate curriculum. Instrumentation can be included in all of the courses in chemistry as an adjunct to the study of each part of the subject matter of chemistry, particularly in those courses which are predominately quantitative such as physical chemistry and quantitative analysis. The second semester in quantitative analysis can properly consist primarily of optical and electrical methods of analysis. In addition, however, it should be noted that there are basic principles which are widely applicable in virtually all measuring operations, and these principles can be treated adequately only in a separate course devoted to chemical instrumentation.