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INSTRUMENTAL ANALYSIS

IN THE UNDERGRADUATE CURRICULUM



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Do the instrument skills and techniques taught in school match what employers want in B.S. graduates?

At about the same time that the National Science Foundation (NSF) sponsored workshops to examine curricular developments in the analytical sciences (1), we asked industrial employers for their opinions on how well-prepared recent B.S. graduates were for analytical positions (2). In an attempt to evaluate changes in the undergraduate curriculum over the past 10 years, our initial 1993 survey of industrial employers was repeated in 2004 (3). We estimate that 26% of recent B.S. graduates go directly into chemistry-related employment in manufacturing and service industries; a significant fraction of those take analytical chemistry jobs. Our reports noted the differences between our interpretation of what em-

ployers want—the analytical chemistry knowledge and skills that they think their employees should have—and what the analytical chemistry curriculum consists of, according to the available literature (2, 3).

We got the distinct impression from the published literature that the analytical chemistry curriculum is changing: Topics that once would have been taught in the instrumental analysis (IA) course are now being introduced in the earlier quantitative analysis course. In addition, we concluded that the IA curriculum has changed since our 1993 survey so that the content is more consistent with what industrial employers are looking for. The blurring of the distinction between the former “quant” and

Table 1. Techniques for which students should have practical experience, according to industrial employers (from Ref. 3).

Group 1 (>66%)	Group 2 (33–66%)	Group 3 (<33%)
UV–vis absorption, potentiometer (pH measurement), GC, HPLC, IR absorption, atomic absorption, autotitration	MS, optical microscopy, flame atomic emission, microwave digestion, X-ray fluorescence	NMR, CHN analysis, CE, molecular fluorescence, gel electrophoresis, ICPMS, ICP-optical emission spectroscopy, surface analysis, electrochemical techniques, thermal methods

instrumental courses is in line with the American Chemical Society (ACS) Committee on Professional Training (CPT) recommendation that “both courses should include laboratory work and coverage of chemical/biological and instrumental methods of analysis” (4).

For two reasons, we were interested in further exploring the situation regarding IR absorption spectrometry, NMR spectrometry, and MS (other than as a detector for GC). These are the techniques that curriculum committees often consider part of the undergraduate organic chemistry component. First, we wondered whether the central role that MS now plays in analytical research and applications in life sciences is in any way reflected in where MS is taught in the undergraduate curriculum. Likewise, increasing numbers of inductively coupled plasma MS (ICPMS) instruments are used in clinical and environmental labs to determine multiple trace elements. Is that change represented in the curriculum? Second, for a school’s undergraduate chemistry program to receive ACS certification, the CPT mandates, for whatever reason, that students must have access to a working NMR spectrometer. How is this emphasis on NMR reflected in the curriculum?

Table 1 shows the extent to which industrial employers think students should have experience with various instruments, as determined by the 2004 survey (3). The categories were assigned according to the fraction of the respondents who indicated that students should have experience operating the instruments. The techniques in group 1 were selected by >66% of the respondents, and the techniques in groups 2 and 3 were selected by 33–66% and <33%, respectively. In the context of analytical work, 42% of the employers replied that experience with MS should be part of undergraduate training, almost all considered hands-on experience with an IR spectrometer important, and 30% deemed practical operation of an NMR spectrometer valuable.

To get a more accurate picture of the analytical chemistry curriculum, we felt that it was appropriate to ask faculty directly, rather than rely only on material in the literature.

Survey says . . .

We created a new survey for faculty members and circulated it in 2 stages during 2005. In stage 1, the questionnaires were distributed via the Council on Undergraduate Research listserv. Because the subscribers to this list include many faculty who are not chemists, or even scientists, and because we cannot determine

the total number of subscribers, a response rate for this mode of distribution cannot be provided. In stage 2, the questionnaire was emailed to faculty who were identified as “analytical” at 233 institutions across the U.S. A total of 64 completed questionnaires were returned from 60 institutions in 27 states. Although this response rate appears low, it is almost identical to the number that responded to Mabrouk’s survey of faculty who teach quantitative analysis (5); thus, subject to the same caveats she expressed, we consider our findings representative. Our respondents were almost equally divided between 4-year predominantly undergraduate institutions (29) and Ph.D.-granting institutions (27), with a few responses from M.S.-granting institutions (4). Copies of the surveys and cover letters may be obtained from the authors.

Respondents were asked to answer and comment on a total of eight questions relating to MS and IA. The first four questions concerned where MS is to be found in the chemistry undergraduate curriculum. The next three questions related to the teaching of IA or the institution’s equivalent course. Respondents were asked to indicate in which semester IA was taught, which techniques were taught, and whether students were given the

Table 2. Where students are introduced to topics in the IA curriculum.

Instrument	Lecture (%)	Lab (%)
GC	95	89
UV–vis	93	89
HPLC	89	87
MS	87	56
Atomic absorption (flame)	87	79
GC/MS	85	73
Molecular fluorescence	81	71
IR	79	77
Electrochemical methods	76	55
pH or plon	74	74
Flame atomic emission	72	29
Atomic absorption (furnace)	69	19
NMR	63	53
LC/MS	58	11
ICP-optical emission spectroscopy	58	21
CE	55	19
ICPMS	43	8
X-ray fluorescence	31	8
Surface analysis	26	5
Gel electrophoresis	22	6
CHN analysis	13	0
Thermal methods	11	8
Microwave digestion	6	3
Automatic titration	6	11
Optical microscopy	3	3

opportunity to actually use the instruments. The eighth question asked whether NMR and IR spectrometries were taught outside of the organic sequences. Additional questions covered independent research, safety, and communication skills; these topics are not discussed in this article. To obtain more information on the opinions of analytical educators, we checked recent editions of several analytical chemistry textbooks for their relative coverage of MS and of IR and NMR spectrometries (6–11).

The current status of commonly taught techniques is given in Table 2, which shows the percentage of respondents who include them in their IA lecture or lab courses. No technique—not even UV–vis absorption spectrometry, which must surely be taught in all programs—shows up 100% of the time. This finding suggests that some programs deal with some instrumental techniques in other courses, most likely the quantitative analysis course. To give some indication of the changes in the IA curriculum over the past 20 years or so, we also discuss some earlier data relating to the situation in 1981 and 1998 (12, 13). A survey from 1992 provides some additional data for trend analysis (14).

MS

Only 18% of respondents indicated that students first hear about MS in freshman (first-year) general chemistry, compared with 62% who indicated that the first introduction was in the sophomore (second year) organic classes; ~9% and 11% indicated that students did not find out about MS until the junior (third) and senior (fourth) years, respectively. Several respondents explained that MS was first introduced as a structure-determining tool in the organic courses, and more detailed coverage came later in the analytical course. Some 87% of respondents specified that MS was dealt with in the lecture part of the IA course, and 56% replied that students had access to MS in the lab part of the IA course. This number rose to 73% for GC/MS (85% of respondents include this topic in lecture). However, for LC/MS, only 58% of respondents deal with this in lecture and 11% in the lab; for ICPMS, the corresponding numbers are 43% and 8%.

These data may be compared with those from 1981 and 1998 (12, 13). In 1981, only 19% of respondents indicated that MS was included in the IA lab, a number that had grown to 24% by 1998. (In 1998, 69% reported that GC/MS was included in the IA lab.) In a 1992 survey, MS was not included in a list of the 13 techniques that appeared most frequently in the lab portion of the IA course (14). According to our data, MS now ranks eighth and GC/MS ranks seventh. We deduce that MS is increasingly being considered a part of the IA curriculum and that significantly more IA lab courses now include MS of all types, compared with the situation in 1998. LC/MS and ICPMS were not itemized in any of the other surveys. The textbook survey shows that authors are not yet in agreement about whether MS is part of the IA curriculum: Two of the five texts surveyed did not include a separate chapter on MS, though all mentioned it as a detection mode for GC. One text mentioned MS on only 4 of its 724 pages (11).

Table 3. Excerpts from ACS CPT guidelines on the role of laboratory instruction.

Laboratory instruction should include practical experience with instrumentation for spectroscopy, chemical separations, and electrochemical methods. It should give students hands-on experience with chemistry and the self-confidence and competence to keep legible and complete experimental records; synthesize and characterize inorganic and organic compounds; perform accurate and precise quantitative measurements; use and understand modern instruments, particularly NMR, FTIR, and UV–vis spectrometers and GC, GC/MS, and HPLC instruments for chemical separations and electrochemical instruments; interpret experimental results and draw reasonable conclusions; analyze data statistically and assess reliability of results; anticipate, recognize, and respond properly to hazards of chemical manipulations; design experiments; plan and execute experiments based on research and using the literature; communicate effectively through oral and written reports; and work effectively in small groups and teams.

NMR

Our results indicate that 63% of respondents include NMR in the lecture part of the IA course and 53% include it in the lab. The corresponding numbers for 1981 and 1998 are 48% and 33%, respectively, for proton NMR and 2% and 20%, respectively, for ^{13}C NMR. These numbers are perhaps more difficult to interpret than those for MS, but little change seems to have occurred in the percentage of lab courses offering NMR experiments. In the 1992 Harris and O'Brien data (14), NMR ranked 10th, which is the same position as in our data. In the 1998 Girard and Diamant survey (13), proton NMR ranked ninth. A mismatch would appear to exist, however, between the importance attached to NMR by the analytical chemistry teaching community and the views and opinions of the industrial employer community, which rated hands-on experience with NMR as relatively unimportant. The industrial employers seem to agree with the textbook authors about NMR—three of the five texts did not include the topic at all.

One possible reason for the mismatch in viewpoints is that some industrial analytical organizations consider NMR a “facility technique”—the entire NMR lab is viewed as a black box. Samples are delivered, and shortly afterward spectra and interpretations are returned. On the other hand, educators use the interpretation of NMR spectra as a means of teaching critical thinking skills and ensuring that students understand the principles on which the technique operates. The presence of a working NMR

Some programs deal with some techniques in other courses, most likely the quantitative analysis course.

Table 4. Excerpts from ACS CPT guidelines on chemical instrumentation.

Instruments and equipment now used in a good undergraduate chemistry program typically include, in addition to analytical balances, pH meters, desktop computers, and specialized glassware, most of the following:

Apparatus for inert atmosphere manipulations; atomic absorption spectrometer; computer workstations for computational chemistry and molecular modeling; FT-NMR spectrometer; gas and liquid chromatographs; gas chromatograph/mass spectrometer; multipurpose electrochemical instrumentation; optical spectrometers; and vacuum systems.

They may also include instruments or apparatus for the following purposes:

Calorimetry and thermal analysis; electrophoresis; kinetics measurements; laser-based applications; MS; molar weight measurements; radiochemistry (including counting equipment and sources); Raman spectroscopy; ultracentrifugation; and X-ray crystallography.

instrument, as required by the CPT, ensures that students gain an appreciation for the practice of NMR, its scope, and its limitations. In theory, a similar argument might be applied to MS for organic structure determination. However, some forms of MS, such as those for GC and LC detectors, are considered so robust and easy to use that everyone is expected to be able to carry out the analyses. Thus, the instruments are not housed in special facilities. This is also the situation for IR spectrometry.

IR

For the group 1 technique of IR spectrometry, 79% of the current respondents indicated that this was part of the IA lecture, and 77% indicated that it was part of the lab. The figures were 81% and 66%, respectively, in 1981 and 82% and 55%, respectively, in 1998. However, both of these earlier surveys reported separate results for FTIR. No coverage was reported in 1981, but in 1998, 76% included the topic in lecture and 61% offered the topic in the lab. Harris and O'Brien ranked IR in fifth place in 1992 (14), exactly the same as in our results. Good agreement seems to exist between industry's ranking of this technique as one to which students should have been exposed and the extent to which they indeed get practical experience. This is reflected in the textbook coverage: All authors either include a chapter on IR spectrometry or devote significant space in the spectrometry chapters to IR.

Role of the lab class

An exercise of this sort raises questions about the role of the lab course or the lab component of a course within the broader educational goals of the program. Some experienced chemical educators have recently voiced concerns. According to Wenzel, "The majority of undergraduate laboratories incorporate activities whose main focus is to support content from the lecture and to teach fundamental manipulative skills and techniques" (15). He goes on to state that "another feature that characterizes many undergraduate science curricula is a rigid set of requirements . . . so that most courses have their own associated laboratory. This format not only restricts interdisciplinary curricular initiatives but encourages students to compartmentalize material into unnecessary and often arbitrary sub-disciplines that may no longer have meaning in modern scientific investigations." One might add that the faculty may be similarly encouraged in their thinking about the composition of the curriculum.

Even Wenzel's characterization of the lab course as "supporting content from the lecture" may be optimistic. Hawkes writes that "laboratory classes do not help students to understand how chemical principles affect their universe" and that "they can help in promoting interpretation and design of experiments, but they are not useful in learning other aspects of chemistry" (16, 17). Hawkes focuses primarily on the role of the lab component of a course for nonmajors, but clearly not everyone agrees with him (18). Also, his position is somewhat at odds with that expressed by the convocation organized by the Center for Science, Mathematics, and Engineering Education of the National Research Council (NRC). That meeting resulted in the call for the development of introductory-level college science courses that are "problem-driven, emphasize critical thinking, provide hands-on experience, are relevant to topics students find in life, offer both the process and the concepts of a discipline, show links between related disciplines, place the subject in a broader personal historical, cultural, social or political context, and provide intellectual tools needed to explore new areas" (19). Hawkes's position would also seem to be in conflict with the recommendations of the Committee on Undergraduate Science Education, which, in its 1997 report, advocated strongly for the inclusion of lab experiences in introductory science courses and provided references to descriptions of exemplary courses (20). Nonetheless, the articulation of opinions about the impoverished nature of the chemistry lab experience may be indicative of a gap between the reality of undergraduate lab instruction and the possibilities indicated by research.

The CPT guidelines for the role of lab instruction are given in Table 3, which highlights the need for exposure to molecular absorption and NMR spectrometries, instrumental chromatographies, and electrochemistry. This emphasis is reinforced by the guidelines for equipment and instrumentation in Table 4. The CPT places particular emphasis on NMR spectrometry: "Nuclear magnetic resonance spectroscopy has become an indispensable experimental method for chemistry. An approved chemical pro-

The undergraduate analytical curriculum also has to serve students who go on to graduate school.

gram must have an operational NMR spectrometer" (21). Furthermore, the guidelines say, "The instruments available to the students should be reasonably recent models in current use by professional chemists. A department should have several pieces of sophisticated equipment suitable for undergraduate instruction as well as for research. One of these must be an NMR spectrometer" (21).

Although the CPT guidelines do not go so far as to specify which instruments students should use in formal lab courses and which they should encounter in research projects, the material provided in the supplements provides some indication of the CPT's views in the syllabus for each subdiscipline. The lab sections of the supplements in Table 5 indicate that the use of instrumental techniques for materials characterization is a common theme across the subdisciplines.

The analytical chemistry supplement contains "instrumental methods" to which students should have been exposed in "a systematic study of the entire sequence of steps of the analytical process." These are given in the analytical section of Table 5, from which it is clear that the CPT guidelines place the same emphasis on MS that industrial employers do. However, the relative importance of IR and NMR is not clear, unless one can deduce something from the order in which the techniques are listed.

A comparison of Tables 1 and 4 shows that several discrepancies exist regarding the importance of experience with techniques. For example, industry rates experience with an autotitrator, a microwave digestion system, and an optical microscope higher than do the faculty responsible for the teaching of analytical chemistry and the CPT. The reverse is true for molecular fluorescence, CE, and electrochemical techniques other than potentiometry—industrial employers rate these techniques as less important than do the teaching faculty and the CPT. In addition, results from the surveys of industrial employers indicate that they consider sampling, sample preparation, and interpretation of data to be important (2, 3).

However, we should remember that the undergraduate analytical curriculum also has to serve students who go on to graduate school in chemistry or a related discipline. The faculty in those graduate programs will expect doctoral students to have knowledge of relevant chemical measurement technology. Given the limitations of time and resources, tension will probably always exist between the requirements of industrial employers and those of graduate programs. Lab instructors have difficult choices to make about which techniques to include and which to exclude. Even with the most dexterous and creative use of the available time, students can probably not interact meaningfully with >10 different instrumental techniques in the typical one-semester (3-month) course.

As a further complication, employers with nonanalytical positions to fill might hold different views about which techniques students should have experienced hands-on. Budgetary constraints are a nontrivial factor. Many of the instruments under discussion are expensive, in terms of capital investment as well as operational and maintenance costs. Thus, if a department acquires an instrument such as an NMR spectrometer (to offer ACS-certified B.S. degrees) and makes it available for student

Table 5. Excerpts from ACS CPT guidelines supplements that relate to laboratory courses.

Analytical: The laboratory experience needs to reflect the entire "analytical process" and not focus only on the measurement step. The problems to which students are exposed should reflect the diversity of analytical problem-solving: biological, materials, environmental, and chemical systems; major to trace components; various physical states of matter; chemical specification; and qualitative and quantitative analyses reflecting a range of accuracy and precision.

The lab experience course should provide exposure to a diverse set of approaches that reflect the wide range of analytical tools available (equilibrium-based methods, kinetic-based methods, physical properties) using various families of instrumentation: spectroscopy (UV-vis, fluorescence, atomic absorption, ICP-atomic emission, IR, Raman, X-ray, NMR); separations (GC, HPLC, electrophoresis, ion chromatography, affinity chromatography); MS (including the distinction and utility of different ionization methods, including electron ionization, chemical ionization, ESI, MALDI); electrochemistry (ion selective electrodes, amperometry, voltammetry); hyphenated techniques (GC/MS, LC/MS); and thermal methods (thermal gravimetric analysis, differential scanning calorimetry).

Inorganic: Characterization methods that involve measurements of magnetic susceptibility, conductivity, X-ray diffraction, IR, UV-vis, NMR, Mössbauer, and mass spectra.

Organic: Spectroscopic analysis of starting materials and products; deducing structures and answering questions from spectroscopic data; analysis of experimental data using statistics.

Physical/spectroscopy: Analysis of a vibration-rotation spectrum; isotope effects (e.g., HCl/DCI); analysis of a polyatomic vibrational spectrum (e.g., SO₂); analysis of an electronic-vibration spectrum (e.g., I₂); analysis of electronic spectra (e.g., conjugated polyene dyes); atomic spectroscopy; Raman spectroscopy; NMR analysis of spin-spin coupling in a non-first-order case; laser applications.

Biochemistry: The experiments should emphasize techniques of general importance to biochemistry as described in the general guidelines. Some examples are error and statistical analysis of experimental data, spectroscopic methods, electrophoretic techniques, chromatographic separations, and isolation and identification of macromolecules.

use, it is perhaps not too surprising that as many lab courses as possible make use of the technique. Therefore, it turns up in the IA lab, despite the fact that industrial employers and most textbooks indicate that this technique has low priority in the analytical curriculum.

Roughly 76% of the respondents to our survey indicated that students encountered instrumental techniques in courses other than analytical chemistry and that many of these encounters involved using the techniques in both quantitative and qualitative chemical analyses. Thus, students are exposed to chemical measurements and instruments even if they do not get hands-on operating experience or detailed explanations of how the instruments work. Although analytical faculty may feel a little uncomfortable with this diffusion of IA into other parts of the curriculum, it opens up opportunities for the analytical courses to provide just what the CPT recommends: "an integrated view of chemical, biological methods and instrumental techniques, including their theoretical basis, for solving a variety of real chemical problems." These are encouraging signs that Wenzel's characterization of lab classes as merely places where students acquire "fundamental manipulative skills and techniques" may no longer be true.

Conclusions

Changes have occurred in the content of the IA lab course over the past 5 years or so that reflect a greater inclusion of NMR and MS in the analytical chemistry curriculum, even though this trend is not yet apparent in textbooks. This is somewhat unexpected, because the conventional wisdom is that the textbooks define the curriculum. We think that there should be a closer dialogue between industry and academia with regard to curriculum content and that maybe practicing industrial chemists should be better represented on the CPT. We discern a continued integration of the instrumental course with what used to be called the quantitative course, as well as the coverage of chemical instrumentation in other areas of chemistry. This trend may be driven by a greater integration of biological topics into the curriculum or the teaching of analytical chemistry by faculty other than traditional analytical chemists. We suggest that the CPT could usefully expand its deliberations to topics other than the content of the undergraduate course and offer commentary on exemplary educational practices, particularly lab instruction. However, we recognize that instructors may not wish to receive advice on best pedagogical practices from a committee consisting of a number of industrial chemists.

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