JOURNAL OF CHEMICAL EDUCATION
ARTICLE
pubs.acs.org/jchemeduc

A Survey of the Practices, Procedures, and Techniques in Undergraduate Organic Chemistry Teaching Laboratories

Christopher B. Martin,* Monica Schmidt, and Michael Soniat

Department of Chemistry and Physics, Lamar University, Beaumont, Texas 77710-0022, United States

ABSTRACT: A survey was conducted of four-year institutions that teach undergraduate organic chemistry laboratories in the United States. The data include results from over 130 schools, describes the current practices at these institutions, and discusses the statistical results such as the scale of the laboratories performed, the chemical techniques applied, the instrumentation available, the laboratory equipment used, the chemistry topics covered, the way chemical safety is presented, and how chemical waste is handled. These results provide a "snapshot" of the current state of the undergraduate organic teaching laboratory.

KEYWORDS: Second-Year Undergraduate, Curriculum, History/Philosophy, Organic Chemistry, Textbooks/Reference Books, Laboratory Equipment/Apparatus, Laboratory Management, Microscale Lab

Science is an ever changing and evolving field of study. Not only do the theories and techniques of a particular scientific field yield to newer and more refined ones, but these changes must be reflected in the classrooms as well. It may be argued that the main points of focus in an introductory course in organic chemistry have changed very little over the past 40 years. The same alcohol dehydration that occurred in a beaker in 1975 will still produce the same result today. Although the result of the reaction has not changed, the ways that the organic chemistry teaching laboratory has changed are tremendous. Gone are the days of the perpetual sodium fusion test, countless derivatives, and the fire extinguisher under the arm of every teaching assistant. Today the students are taught the practical and experimental skills in the field of organic chemistry using routine ^{1}H NMR, fractional distillations on a 2 mL scale, and the analysis of microliters of their product on a capillary GC.

A brief investigation of the available organic chemistry laboratory textbooks reveals the change in the emphasis in techniques and topics covered. For example, one textbook from the 1960s and two textbooks from the 1970s report the procedure to perform an electrophilic aromatic stubstitution reaction (nitration) of benzene using 40−60 mL of concentrated sulfuric acid and 35−50 mL of concentrated nitric acid in a 500 mL round-bottomed flask.[1−3] In the 1980s, this was replaced by only 8 mL of sulfuric acid and 5 mL of nitric acid in a 125 mL flask.[4] The 1990s saw the volumes reduced even further with the increasingly popular microscale techniques that used only 1 mL of each acid in a 5 mL conical vial.[5] Today, textbooks even provide "multiscale" procedures where the institutions may select the scale that best fits their individual needs.[6] Although the analysis of laboratory textbooks may help to reveal some of the changes in the scale of the laboratory and the trends of the experiments performed, it only addresses the trends of what is available in the textbooks and not what is actually being performed in the laboratories. Additionally, it fails to consider the practices at schools that develop and use their own laboratory experiments.

The American Chemical Society's Committee on Professional Training (ACS CPT) has outlined guidelines for institutions that wish to seek certification in their Bachelor of Science (B.S.) degrees.[7] The spring 2008 edition indicates that the (postgeneral chemistry) laboratory experience must include the "synthesis of molecules, measurement of chemical properties, structures, and phenomena; hands-on experience with modern instrumentation; and computational data analysis and modeling". The hands-on experience must include "spectrometers (NMR, FT−IR, and UV−visible spectroscopy), chemical separations instruments (such as those for GC, GC−MS, and HPLC), and electrochemical instruments". The CPT does not, however, provide any stipulations on how these are distributed throughout the curriculum except to say that they must cover 4 of the 5 foundation areas (organic, inorganic, biochemistry, physical, and analytical). For example, some institutions may use HPLC and GC in the organic chemistry lab whereas others may reserve these instruments for an (analytical) instrumentation lab. Many different approaches to the organic chemistry laboratory exist, all of which may successfully fulfill the guidelines of the ACS CPT. Therefore, it is clear that neither a survey of the organic chemistry textbooks nor relying on the published requirements from the CPT will necessarily provide an accurate view of the current trends being practiced in modern organic chemistry teaching laboratories. Therefore, a direct survey of the institutions is needed to effectively determine an accurate assessment of the organic chemistry labs.

The aim of this article is to report the results of a survey of the current practices of the organic teaching laboratories in the United States by four-year institutions. To our knowledge, this is the first survey of this type to measure the state of the undergraduate organic teaching laboratory in recent years. Hopefully, these results will serve as a method of measurement that will be beneficial to academic institutions both undergraduate and postgraduate, the chemical industry that aims to employ recent graduates, and to the students who are evaluating future education.

■ MATERIALS AND METHODS

The survey was administered via an online survey[8] between the fall semester of 2008 and the beginning of the spring semester 2009. Over 600 survey invitations were sent to four-year academic institutions that were eligible to grant an ACS-certified

dx.doi.org/10.1021/ed200234f | J. Chem. Educ. XXXX, XXX, 000–000

Bachelor's of Science degree in chemistry. Greater than 90% of all four-year institutions teaching organic chemistry laboratories in the country are represented by these qualifications. The voluntary survey was closed and the data analyzed once the completion rate of the survey reached a point of less than one new survey in a two-week time period. The results reflect over 130 different academic institutions. Although these statistics are by no means to be considered all encompassing, the trends and overall results appear to be an accurate reflection of the state of the current organic teaching laboratories across the United States. The results were anonymous and the statistics were not adjusted to reflect the number of students taking the organic chemistry laboratory at each institution. Therefore, the results are indicative of a "per institution" basis and not a "per student" basis.

RESULTS AND DISCUSSIONS

Laboratory Scale, Glassware, and Factors Influencing Laboratory Changes

The scale of the laboratory was defined as the typical size of the reactions that are performed in the organic teaching laboratory at the respondent's institution. For this survey, microscale was defined as reactions typically containing less than 3 mL of solvent combined with the usage of specialized glassware such as conical vials. Macroscale (or miniscale) reactions usually involve at least 25 mL of solvent and are performed in round-bottomed or Erlenmeyer flasks.

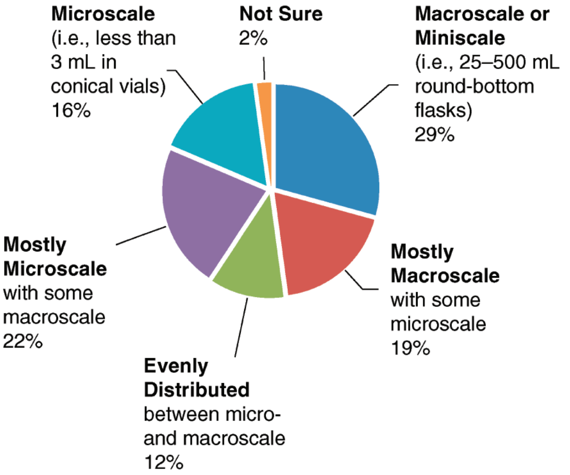

Figure 1. Distribution of the various experimental scales in current organic chemistry teaching laboratories.

The results indicate that the distribution of institutions that use microscale compared to macroscale is similar (Figure 1). Additionally, 53% of the institutions conduct their laboratories with a "hybrid" approach where a combination of micro- and macroscale techniques are used.

In an attempt to further understand the reasoning behind the use of the various reaction scales and glassware in the laboratories, the views of each institution for microscale and macroscale were investigated (Table 1).

From the results shown in Table 1, it can be concluded that the overwhelming majority of institutions surveyed believe that microscale laboratories reduce the waste generated compared to macroscale labs, but they also feel that this smaller scale fails to teach the students the appropriate required techniques that students need to learn in the organic lab. On the other hand, only about half of the respondents agree that the undergraduate microscale lab fails to prepare the students with the skills for performing reactions in the research laboratories. Surprisingly, almost the same percentage of faculty is in agreement that the microscale teaching labs do prepare students with the skills needed for research. Therefore, the results indicate that the majority of institutions believe that students who perform macroscale experiments have the opportunity to gain a greater understanding of the macroscale skills required in the organic teaching laboratory when compared to microscale labs (69% vs 38%), but neither scale is sufficient at the undergraduate teaching laboratory for preparing the students for the macroscale techniques used for performing research (50 ± 8% in all cases).

One of the common criticisms against microscale is that the students are unable to effectively comprehend the scientific concepts on such a small scale. As one respondent stated, "Seeing is believing. If you cannot see the product, it is not satisfying at the introductory level." Only 28% of the respondents felt that the scale of the experiment significantly affected the ability of the

Table 1. Views of Microscale and Macroscale

Microscale		Macroscale	
Statement	Agreement (%)	Statement	Agreement (%)
Microscale labs are a great way to reduce the waste generated in undergraduate teaching labs.	77	Macroscale labs generate a large volume of waste in undergraduate teaching labs.	67
Microscale labs provide the opportunity for students to learn the appropriate techniques needed.	38	Macroscale labs provide the opportunity for students to learn the appropriate techniques needed.	69
Microscale labs fail to teach students the macroscale techniques used in modern research settings.	52	Macroscale labs teach students the appropriate techniques used in modern research settings.	58
Microscale labs use quantities of chemicals that are so small that students are often unable to concentrate on the science.	29	Macroscale labs use quantities of chemicals that allow students to concentrate on the science rather than the quantities.	28
Microscale labs improve the safety in the lab.	53	Macroscale labs increase the safety risks in the lab.	39

Table 2. Influence of Various Factors Affecting Potential Changes to the Organic Teaching Labs

	Response (%)			
Factor	Not Important	Relatively Unimportant	Somewhat Important	Very Important
Cost of chemicals	1	16	**52**	30
Cost of waste disposal	3	22	**46**	29
Cost of glassware and equipment	3	20	**51**	25
Applicability of techniques beyond the course for use in other undergraduate labs	2	13	**53**	32
Conforms to current available glassware in the undergraduate organic labs at your institution	4	18	**50**	28
Conforms to current academic research standards and techniques	1	7	**55**	37
Conforms to current industrial, R&D, or private industry desires	6	38	**41**	15
Flexibility	6	14	**57**	23

students to concentrate on the scientific principles. In support of this statement, the exact same number of respondents felt that the larger scale benefitted the students' scientific comprehension. It should also be noted that some of the institutions indicated that macroscale is required to serve the need of their chemical engineering students. Others indicated that the degree of macroscale experimentation increased in the second semester where the concentration of chemistry majors was greater.

A common criticism of macroscale compared to microscale is that of safety. The possibility for large-scale fires and other accidents should be significantly lower when performed on a 3 mL scale when compared to a 100 mL scale. Nevertheless, there was a fairly even distribution in the view that macroscale labs increased the risks and microscale decreased the risks in the organic teaching laboratory. Therefore, it appears that safety is not a significant motivation for the selection of one scale over another.

If the practice of micro- and macroscale is fairly evenly distributed across the survey and it is the majority opinion that macroscale does not significantly increase the ability to focus on the fundamental science, transition to research setting, or increase safety, then it appears that the main consideration for microscale over macroscale is predominantly a matter of waste generation at the cost of attainment of nonresearch organic laboratory skills.

One could reasonably argue that the transition from macroscale to microscale in the organic teaching laboratory is significant considering that macroscale was effectively the only scale available for many years. One possible reason for some resistance to change scales is due to the pre-existing glassware and expertise available at an institution. In an attempt to determine the effect that various aspects may have on an institution's decision to alter the organic teaching laboratory, several questions that reflect these aspects were asked.

The data in Table 2 indicate that the greatest number of institutions believe that cost, applicability of techniques learned within academia and beyond, and flexibility are all somewhat important factors in determining what scale should be used in the laboratory. This result is not surprising, as it is expected that all of these factors would be reported as important from academic institutions. However, an interesting result is obtained when the results are retabulated with the statistics grouped into only two categories: mostly important or mostly unimportant. This was done to determine the relative ranking of the importance of these items by academic institutions when they consider changes to the organic teaching laboratories (Table 3).

It appears that the most important feature that may influence potential changes in the undergraduate organic chemistry teaching labs is the effect on academic institutions. The central consideration is the conformation to "internal" applicability, namely, current academic standards. Conversely, the least important aspect appears to be the conformation to "external" standards such as commercial and private industries. The origin of this polarity was not studied, but it can be hypothesized that this is an effect of the changing relationship between academia and industry in organic chemistry. Although the extent of the relationship is certainly an effect of region, changes in legal restrictions, economic considerations, the quantity of collaborations, internships, and involvement between academic institutions and private industry, the relationship is definitely not the same as it was 20−30 years ago.

Although academic institutions are often faced with the task of operating with a very limited budget, it appears that the consideration of cost is not a significant determining factor when deciding to change an organic laboratory curriculum. However, it is apparent that the cost of purchasing chemicals is a slightly more important factor than the cost of disposal of the chemical waste (82% vs 75% from Table 3).

Organic Laboratory Techniques

The main purpose of the organic chemistry teaching laboratory is to allow the student to gain the experience of actually performing a chemical reaction. Although the lecture portion is the place where students learn how one could theoretically perform a chemical transformation, the laboratory is the place where the student actually performs the reaction. Owing to obvious time restrictions within an academic term, there is often a significant difference between what laboratory instructors would like for students to learn and what they actually have the time to cover. In an attempt to quantify the relative importance of the multitude of techniques available for teaching purposes, the survey asked the respondents to determine the

Table 3. Retabulated Importance behind Factors Influencing Potential Change in Organic Teaching Laboratories

	Response (%)		
Factors	Not to Relatively Important	Somewhat to Very Important	Importance Ranking
Cost of chemicals	17	**82**	3
Cost of waste disposal	25	**75**	6
Cost of glassware and equipment	23	**77**	5
Applicability of techniques beyond the course for use in other undergraduate labs	15	**86**	2
Conforms to current available glassware in the undergraduate organic labs at your institution	22	**78**	7
Conforms to current academic research standards and techniques	8	**93**	1
Conforms to current industrial, R&D, or private industry desires	44	**56**	8
Flexibility	20	**80**	4

Table 4. The Importance of Various Techniques in the Undergraduate Organic Teaching Laboratories

	Response (%)					
Technique	Not Considered	Relatively Unimportant	Important	Very Important	Absolutely Required	Rank
Recrystallization	0	0	8	26	**66**	1
Extraction	0	0	10	24	**66**	2
Thin-layer chromatography (TLC)	1	2	11	25	**60**	3
Washing liquids and work-up	0	2	17	26	**55**	4
Melting point determination (m.p.)	0	2	17	26	**54**	5
Drying liquids	0	2	16	29	**53**	6
Simple distillation	1	2	18	24	**55**	7
Vacuum filtration	0	1	20	33	**46**	8
Solvent removal	0	7	23	33	**38**	9
Fractional distillation	5	11	20	25	**39**	10
Column chromatography	4	16	21	24	**34**	11
Gravity filtration	2	12	**41**	19	26	12
Drying solids	7	19	**28**	22	24	13
Boiling point determination (b.p.)	6	**30**	22	16	26	14
Rotary evaporation (rotovap)	16	**25**	23	15	20	15
Moisture-sensitive techniques (drying tube)	16	15	**34**	25	9	16
Optical rotation	26	**31**	25	13	5	17
Vacuum distillation	21	**37**	28	9	5	18
Steam distillation	24	**35**	26	10	5	19
Sublimation	22	**46**	21	8	2	20
Paper chromatography	38	**48**	11	2	2	21
Air-sensitive techniques (Schlenk line)	**52**	29	12	4	2	22

importance of each of the following laboratory techniques in the organic teaching laboratory (Table 4).

The results of Table 4 show that the first 11 techniques listed are absolutely required in an organic teaching laboratory. These include (in order) recrystallization, extraction, TLC, liquid washing and work-ups, melting points, liquid drying, simple distillation, vacuum filtration, solvent removal, fractional distillation, and column chromatography. The vast majority of organic laboratory textbooks have all of these items listed as skills to be included within the first term of a multiple-term organic laboratory sequence.

There may be several reasons why the remaining techniques were not considered as important by respondents. For example, techniques such as vacuum distillation are essential for research laboratories, but are difficult to perform with a large number of students simultaneously. Individual vacuum distillations are typically performed using dedicated vacuum pumps, which can operate at very low and consistent pressures. Vacuum distillations in the teaching labs would probably require house vacuum, which cannot operate at very low pressures and is often very inconsistent. In decades past, steam-distillations were commonly used to co-distill desired organic products by safely supplying

Table 5. Importance of Students' Exposure to a Particular Instrumental Technique in an Organic Teaching Laboratory

	Response (%)				
Exposure	Not Considered	Relatively Unimportant	Important	Very Important	Absolutely Required
Gas chromatography (GC)	20	11	16	16	**36**
High-performance liquid chromatography (HPLC)	28	**34**	24	8	7
Mass spectrometry (MS)	20	13	**26**	20	21
Nuclear magnetic resonance (NMR)	13	3	12	13	**59**
Infrared spectroscopy (IR)	18	6	10	13	**54**
Ultraviolet visible spectrometry (UV−Vis)	26	**29**	28	5	11

Table 6. Importance of Students' Hands-On Experience to a Particular Instrumental Technique in an Organic Teaching Laboratory

	Response (%)				
Hands-On	Not Considered	Relatively Unimportant	Important	Very Important	Absolutely Required
Gas chromatography (GC)	6	7	17	23	**47**
High-performance liquid chromatography (HPLC)	29	**32**	22	11	7
Mass spectrometry (MS)	**26**	24	18	19	14
Nuclear magnetic resonance (NMR)	9	7	11	21	**52**
Infrared spectroscopy (IR)	2	2	10	17	**69**
Ultraviolet visible spectrometry (UV−Vis)	18	**33**	32	10	8

both the heat and water using steam cones, but these have proven to be increasingly unpopular since the introduction of nonflame-based heating sources. Sublimation, Schlenk lines, and optical rotation are probably not as popular because some of these techniques are suited better to other courses (i.e., inorganic chemistry laboratory) or they require equipment that is typically used for one laboratory and therefore would not be as useful for the remainder of the course. Other techniques listed by the universities included glassware manipulation, centrifugation, theoretical calculations, polarimetry, microwave synthesis, library or literature searching, and green methods, none of which contributed greater than 4% of the total responses.

Instrumentation

In the organic laboratory, an increasing degree of reliance on instrumentation has been observed. The decrease in cost and time coupled with an increase in instrument sensitivity and availability has led the organic lab of today to be very different from the organic lab of 20 years ago. To better understand the role that instrumentation plays in the organic teaching laboratory, institutions were surveyed as to the importance that various instrumentation has in two specific areas. The first area is simply exposure to an instrument. Exposure is defined as the requirement that a student be introduced to a technique, but they will not actually gain hands-on experience. This includes student samples that are analyzed by a technician, simulated results, or exercises that originate from real chemical analysis but not necessarily from their own experimental results. The second area is defined as actual hands-on experience. This includes any time the student performs an analysis on the actual instrument and then uses the results to analyze their experimental results. The results of the instrumentation portion of the survey are shown in Tables 5 and 6.

The results of the survey indicate the most important instrumental techniques in the undergraduate organic teaching laboratory are NMR, IR, and GC. At least 70% of the institutions surveyed indicated the students get actual hands-on experience on each of these instruments. NMR is much more expensive than IR or GC, but nevertheless, it still remains an important instrumentation technique in organic teaching laboratories today. Even when the survey asked the respondents to limit their opinion to only instrument exposure instead of hands-on experience, greater than 50% of the institutions indicated they felt these three instruments were very important to the course. HPLC, UV−Vis, and MS were not ranked as important to the organic teaching laboratory probably because these techniques are more appropriately introduced in higher-level instrumental laboratories, which are also smaller classes better suited to these instruments. Other instrumental techniques the respondents mentioned were circular dichroism (CD), fluorescence, theoretical calculations, polarimetry, and microwave synthesis. None of these contributed more than 4% of the total responses.

Laboratory Equipment

Just as instrumentation is important to the undergraduate organic teaching laboratory, so is the presence of certain pieces of laboratory equipment. Here, the four-year colleges and universities were asked to indicate which of the following pieces of instrumentation and equipment are currently being used in their organic teaching laboratories. It is important to clarify that this is different from the previous section where the respondents were asked their opinions of the importance of the instrument and its use. This question was specifically aimed at the current usage and availability of equipment in the respondents' respective laboratories.

The results from Table 7 indicate nearly three-quarters of all institutions currently use melting point apparatus, IR, fume

Table 7. Percentages of Institutions Reporting Use of Various Types of Instrumentation and Equipment in the Undergraduate Organic Teaching Laboratories

Instrumentation	Institutions (%)	Instrumentation	Institutions (%)
Melting point apparatus	98	Rotary evaporators	63
IR spectrometer	94	Computers NOT dedicated for instrumentation	61
Chemical fume hoods	93	UV−Vis spectrometer	43
NMR spectrometer	84	Refractive index	33
Gas chromatograph	74	Other types of fume hoods	16

Table 8. Most Common Organic Chemistry Topics Covered in Current Organic Chemistry Undergraduate Teaching Laboratories

Topic	Laboratory (%)	Topic	Laboratory (%)
Solubility	86	Kinetics	50
Electrophilic aromatic substitution	86	Rearrangements	39
Reduction reactions	85	Solvent effects	38
S_N1	81	Williamson ether synthesis	37
S_N2	75	Wittig	35
Oxidations	74	Free radical reactions	31
Fisher esterification	71	Lewis dot	30
E_1	66	Dehydrohalogenation	30
Electrophilic addition to an alkene	63	Cis−trans cyclyhexane reactivity	29
Stereoselective or specific products	61	Markovnikov alkene hydration	25
Diels−Alder	61	Protecting groups	23
Molecular models	59	Thermodynamics	22
E_2	56	>15 other topics	<20

hoods, NMR, and GC in their teaching laboratory. It is interesting to note a majority of colleges and universities have computers for use in the organic teaching laboratories that are not dedicated for instrumentation. The survey did not investigate whether these are for general use in a computer lab, writing of laboratory reports, theoretical calculations, or other purposes.

Organic Chemistry Topics Presented in the Undergraduate Teaching Laboratory

Ideally, the laboratory and the lecture portions of a chemistry course help to reinforce each other. The typical yearlong organic chemistry sequence covers the first 20−24 chapters in the modern organic chemistry textbooks. The laboratories, however, are not as standardized. Some institutions have the lectures and the labs running as embedded and concurrent (one single course containing both), whereas others have the lecture sections and the laboratory sections as separate courses. Some institutions have an entire semester of lecture as a prerequisite with a longer laboratory (sometimes 2 or more credit hours) that encompasses the lab experience intended to span both semesters of the organic chemistry sequence. With such variation, it is not as straightforward to predict a standard set of experiments for the organic chemistry laboratories compared to the lecture material.

This survey presented the respondents with a wide variety of potential chemical topics and asked each institution to indicate any of the topics currently being used in the undergraduate teaching laboratories at their school. Owing to the vast number of possibilities, only the responses reported by at least 20% of the schools are presented here. A total of 25 different organic chemistry topics are listed below in order of occurrence (Table 8).

Chemical Safety

The practices of chemical safety in the organic teaching laboratory have changed greatly over the history of the discipline. It was a common practice in the early part of the 20th century to report the taste of any new chemicals synthesized. Pipetting by mouth is still an accepted practice in organic laboratories in some developing countries. An increase in the understanding of chemical hazards and new legal responsibilities has helped to change the way safety is viewed in the organic teaching laboratory. The following table reports the percentage of organic chemistry laboratories that require each of the following safety related items (Table 9).

The data indicate the safety requirements can be divided into two groups: the first four items are required by nearly three-quarters of the academic institutions, whereas the remaining seven items are required by half of the colleges and universities or less. Therefore, it can be concluded a majority of organic chemistry labs require that students know the location of all the safety equipment in the lab, know how to operate basic safety equipment, wear approved safety glasses, and undergo a one-time safety training session for the laboratory course.

Once safety training is conducted and the laboratory commences, chemical spills still may occur. The event of a typical chemical spill can be handled several different ways for various reasons. The instructor may elect to handle the entire spill themselves for safety considerations, time, and to ensure the spill is handled correctly. Another approach would be to treat the

Table 9. Percentage of Organic Teaching Laboratories That Require the Indicated Safety Items

Safety Item	Laboratory (%)
Know location of safety equipment in lab	96
Know how to operate safety equipment (e.g., eyewash station)	82
Wear ANSI approved lab glasses	72
Have lab safety training	72
Explore MSDS reports	52
Wear protective gloves	49
Have specific experiment training (safety training specific to each lab)	41
Wear lab glasses (any type)	36
Know how to operate a fire extinguisher	27
Wear long sleeves	20
Wear lab coat	12

hazard as a teaching situation where guidance is provided as the student deals with their chemical spill. The student may also be expected to completely handle their own spill to encourage personal chemical responsibility (providing that sufficient training has been conducted). The table below shows the practices of the colleges and universities when presented with the following options for a chemical spill in the organic teaching laboratory (Table 10).

Table 10. Percent of Colleges and Universities That Handle Chemical Spills in the Organic Chemistry Teaching Laboratories in Each Respective Way

Way to Handle Chemical Spill	Laboratory (%)
The instructor handles the chemical spill.	64
A teacher's assistant handles the chemical spill.	45
The student is supervised by an instructor or teacher's assistant while cleaning the spill.	42
The student is instructed on how to handle the chemical spill on their own.	9

The results of the survey indicate it is common practice is to have the instructors or the teaching assistants to handle chemical spills. The survey did not differentiate between large institutions that would contain hundreds of undergraduate students for one instructor or small institutions that may contain a very low student-to-teacher ratio with no teaching assistants. Nevertheless, the trend is to not allow the student to handle chemical spills. Possible reasons for this practice may include safety concerns for the student or the desire to prevent the mishandling of chemical waste.

Chemical Waste and Environmental Considerations

One of the greatest concerns for any academic institution conducting organic chemistry teaching laboratories is the chemical waste generated. This is evident by the popularity of microscale techniques and the move away from macroscale techniques. All waste generated in the organic chemistry teaching laboratory is not the same and as a result should not be treated equally. Generally, waste is categorized as neutralized aqueous waste, nonhalogenated organic waste (solvent), halogenated organic waste (solvent), solid organic waste, and heavy metals. There are several methods for minimizing waste and other methods for handling waste once it is generated (Table 11).

Many organic laboratories generate aqueous waste from reactions or extractions. Often, this waste can be safely disposed of down the drain once it has been neutralized to a pH between 7−9. When presented with the opportunity to have undergraduate students neutralize their own aqueous waste for safe disposal down the drain, only 46% of the respondents indicated that students are encouraged to do so whereas 54% were not. The reluctance to allow the students to handle their own chemical waste may arise from many different reasons. It is possible that the instructors do not feel the students will neutralize the waste correctly; either they will dispose of all waste down the drain without neutralizing it properly or they believe the neutralization process itself introduces unnecessary risks. A counter-argument is that by not allowing students the opportunity to handle their own chemical waste they do not gain a sense of responsibility for their own waste generation. The students do not see the effort and time involved in taking care of their waste products properly and so they may not realize how important it is. This latter approach may encourage an "out of sight; out of mind" philosophy.

The cost of disposal of halogenated organic waste is much greater than that of nonhalogenated organic waste. Therefore, it is not surprising 78% of the academic institutions participating in the survey require students segregate their halogenated and nonhalogenated waste. Of the 22% who do not require waste solvent segregation, it is not known if the laboratories only use nonhalogenated waste, labs containing both halogenated and nonhalogenated within the same experiment are not present (so the student never realizes that these are segregated), or if the institutions simply treat all waste the same when disposed.

Another practice that may help undergraduate students become conscious of the quantities of chemical waste generated is to require them to record the quantity of waste upon disposal. This practice has legal implications and will differ based upon the status of the academic institution as a large- or small-scale generator of chemical waste. Legal and other classifications aside, only 17% of undergraduate institutions require their organic chemistry students record the quantity of waste generated. Although the practice of the recording of waste is almost certainly preempted by other mitigating factors, the low percentage of students who know how much waste is generated may suggest an alarming lack of knowledge as to the impact their reaction and waste products can have.

Another method used to minimize waste generated in the organic chemistry teaching laboratory is to attempt to reuse organic solvents. This can be achieved either by recovering solvents removed from the trap on a rotary evaporator or by solvent removed by simple distillations. Unfortunately, this practice is not always possible or practical. This difficulty is reflected by the fact that only 15% of the schools responding to the survey participate in solvent recovery in their organic teaching laboratories.

Just as halogenated and nonhalogenated solvents pose a chemical segregation issue, so does the presence of solid wastes containing heavy metals. The presence of these metals poses both health considerations as well as special disposal requirements due to federal regulations. The use of these substances (largely mercury and chromium in the organic laboratory) is becoming less frequent to avoid these issues. Of the schools surveyed, 32% of the institutions do not use any heavy metals in their organic chemistry teaching laboratories. Of the remaining

Table 11. Handling and Segregation of Waste and Re-Use of Chemicals

	Response (%)	
Method	Yes	No
Are the students encouraged to neutralize their own aqueous waste for disposal down the drain?	46	54
Are students required to segregate the waste organic solvent into separate containers as halogenated or nonhalogenated?	78	22
Are students required to record the quantity of organic waste generated?	17	83
Does your institution recover and reuse any organic solvents?	15	85
Does your institution require students to segregate solid waste into materials containing heavy metals and those that do not?	58	10
Does your organic lab sequence contain any multistep syntheses?	85	15
Other than multistep syntheses, does your organic laboratory sequence use any products from any experiment as the starting materials for another experiment?	50	50

68% of colleges and universities, 58% require the students to segregate their solid waste separating heavy metals from waste not containing heavy metals. Surprisingly, 10% of the academic institutions do not require any heavy metal segregation by the students.

Finally, organic waste can be minimized by reusing products from one reaction as the starting materials for another reaction. This practice is inherent when a laboratory sequence involves a multistep synthesis, but it may also be designed into a laboratory course to help reduce the quantity of waste generated. Multistep syntheses are very common among undergraduate teaching laboratories; they are present in 85% of the schools responding to the survey. On the contrary, only 50% of the academic institutions use the product from one laboratory experiment as the starting materials for another (other than for multistep syntheses).

CONCLUSIONS

The practices in the undergraduate organic teaching laboratories have changed significantly over the past several decades. This study reports the results of a survey to determine the current practices and procedures in the undergraduate organic teaching laboratories in the United States.

Overall, the surveyed programs show a trend toward microscale techniques, both to reduce the cost of reagents and to reduce the waste production. However, greater than 50% of respondents feel microscale labs fail to prepare the students for the modern industry in that macroscale techniques are used. With regards to changes in the current curricula, most participants felt conformation to current academic research standards and techniques and flexibility were most important. Also of great importance was adherence to current glassware and instrumentation available in their laboratories.

Although there is less emphasis placed on techniques used in the industrial field, respondents feel organic chemistry laboratory techniques should be applicable to later undergraduate courses. There are 11 such techniques that are absolutely required, including but not limited to recrystallization, extraction, thin-layer chromatography, liquid washing and work-up, melting point, liquid drying, simple distillation, vacuum distillation, solvent removal, fractional distillation, and column chromatography. Instrumentation techniques considered absolutely necessary include nuclear magnetic resonance, infrared spectroscopy, and gas chromatography, with NMR being the most important requirement. This includes both simulated spectra and hands-on experience with the instrument. Despite the higher cost, many institutions believe hands-on experience with NMR is very important. Current equipment in the laboratories other than instrumentation includes melting point apparatus, chemical fume hoods, rotary evaporators, and computers not dedicated to instrumentation.

Safety in the laboratory is always a primary concern of instructors and staff, and it is interesting to note that most accidents and spills are handled by the instructor or a student under faculty supervision. Perhaps the respondents feel as if the students, if left on their own, would fail to properly dispose of the hazard. It is interesting to see 28% of respondents did not report the requirement of ANSI safety glasses.

With waste hazards students seem to be encouraged to separate their wastes into halogenated and nonhalogenated wastes. Some are not encouraged to neutralize their wastes and most are not required to record the quantity of waste produced by their procedures. This is discouraging because the students might not realize the considerable impact of their waste. The current laboratories are also not reusing any of the products as starting reagents for further labs, except for usage in multistep synthesis. This creates more waste that must be disposed by the department, either in-house or by outside treatment facilities. With rising costs of waste disposal, it is imperative more research be performed in the area of reusable products and waste reduction.

AUTHOR INFORMATION

Corresponding Author

*E-mail: christopher.martin@lamar.edu.

ACKNOWLEDGMENT

The authors would like to thank the individuals who participated in the survey. Acknowledgment is made to the donors of the American Chemical Society Petroleum Research Fund for support of this research. Support by the Welch Foundation (Grant number V-0004) is also gratefully acknowledged.

REFERENCES

(1) Pavia, D. L.; Lampman, G. M.; Kriz, G. S. *Introduction to Organic Laboratory Techniques*; Sounders:Philadelphia, PA, 1976.
(2) Robertson, G. R.; Jacobs, T. L. *Laboratory Practice of Organic Chemistry*; 4th ed.; Maclillan Company: New York, 1962.
(3) Vogel, A. I. *Practical Organic Chemistry*; 3rd ed.; Longman Group Ltd.: London, 1970.
(4) Wilcox, C. F. *Experimental Organic Chemistry*; Macmillan Publishing Co.: New York, 1984.

(5) Pasto, D. J.; Johnson, C. R.; Miller, M. J. *Experiments and Techniques in Organic Chemistry*; Prentice Hall: Englewood Cliffs, NJ, 1992.

(6) Lehman, J. W. *Multiscale Operational Organic Chemistry*; Prentice-Hall: Upper Saddle River, NJ, 2002.

(7) *ACS Guidelines and Evaluation Procedures for Bachelor's Degree Programs*; American Chemical Society: Washington, D.C., 2008.

(8) SurveyMonkey Home Page. www.surveymonkey.com (accessed Sep 2011).