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Selection, Evaluation, and Modification of a Standard Operating Procedure as a Mechanism for Introducing an Undergraduate Student to Chemical Research: A Case Study

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ABSTRACT: In an effort to broaden the selection of research opportunities available to a student registered in a one-semester, upper-level independent study course at a primarily undergraduate institution (PUI), a highly motivated student was asked to select, evaluate, and modify a standard operating procedure (SOP). The student gained valuable experience in the decision-making processes involved with selecting an SOP based on discernment of which procedures could be performed at our institution. Once a procedure was selected, the student set up and evaluated the performance of the procedure, enabling mastery of the process. After demonstrating competence, the student was asked to suggest and perform modifications to the procedure thereby providing an opportunity to gain a better connection between the procedure and results. In this work, the QuEChERS (quick easy cheap effective rugged and safe) method for the analysis of hexachlorobenzene (HCB) was examined demonstrating the effectiveness of using an SOP as a tool for introducing an undergraduate to research at a PUI.

KEYWORDS: *Upper-Division Undergraduate, Analytical Chemistry, Inquiry-Based/Discovery Learning, Problem Solving/Decision Making, Agricultural Chemistry, Collaborative/Cooperative Learning, Gas Chromatography, Quantitative Analysis, Undergraduate Research*

BACKGROUND

Technological advancements are often the direct result of research activities, and as a consequence, continued progress requires introducing and training students in the area of scientific research. Traditionally, these activities are encountered at the graduate level, but much attention has been given to the training of undergraduates in research within recent years. A literature review of this journal reveals that there are numerous articles focused on research orientated toward a specific analytical experiment[1−10] and are often referred to as "guided-inquiry" or "problem-orientated" labs.[11,12] Further review shows that many have developed thematic lectures and/or laboratories designed to stimulate interest in research.[13−17] Ultimately, it is vitally important to prepare the undergraduate chemistry major for a career within the discipline.[18−27]

Within the last few decades, there have been numerous changes to the chemistry major which have enhanced student exposure to research methods. An early 2000s report by the Council on Undergraduate Research (CUR) stated that "PUI's have not responded quickly enough to the changing landscape of higher education and research", and as a result, a summit was convened in 2003 which produced 10 white papers on undergraduate research issues along with a clear set of recommendations.[27] Clauss, Blackwell et.al,[24] provided a new model for an undergraduate course which contains components of several previous publications (see ref 1 within this work). Furthermore, Richter-Egger et.al,[25] offered clear sets of their expectations for students regarding their involvement in the "development and refinement of instrumental methods" used by third-year and general chemistry students. The ACS Committee on Professional Training (CPT) highlights the importance of undergraduate research in their statement that "research can be the most rewarding and educationally valuable aspect of an undergraduate chemistry degree."[28] The CPT described the characteristics for undergraduate research, outlined the development of student skills, and emphasized the importance of the written research report. In 2008, the ACS Guidelines for Bachelor's Degree Programs was updated allowing "the use of undergraduate research both as in-depth course work, as well as a means of meeting 180 of the 400 laboratory hours required for certification provided that a well-written, comprehensive, and well-documented research report is prepared at the end of a project."[28] CPT is developing the next revision of the Guidelines, which are anticipated to be adopted in 2014.

While much progress has been made in this important area of chemical education and many PUIs now have active research components incorporated into their curriculum, there continues to be a need for new innovative methods of exposing undergraduate students to research and that is the focus of this paper.

COURSE DEVELOPMENT AND GOALS

The objective of this work was to provide research experience for an upper-level undergraduate chemistry student within an independent study research methods course. Considering that analytical method selection and implementation are common expectations of students who graduate with a degree in chemistry, whether entering graduate school or industry, this course was designed to mimic this experience by asking the student to carry out the following series of actions.

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1. Select an analyte and review the purpose and methods of analysis.
2. Select a standard operating procedure (SOP) based upon equipment availability.
3. Acquire the necessary, samples, standards, and equipment to perform the experiment.
4. Demonstrate experimental competence by producing reliable and reproducible results.
5. Suggest a modification of the procedure and carry out experiments using their alteration.
6. Compile and analyze results.
7. Write a traditional lab report, with special emphasis placed upon the discussion section.
8. Present their findings at an appropriate venue.

A student was asked to carry out these activities, and the process will be presented in this paper so that student expectations and outcomes can be evaluated.

SELECTION OF AN ANALYTE AND METHOD OF ANALYSIS (SOP)

Analyte selection is an important first step that forces the student to connect it to the scientific literature and the various methods of analysis. In this study, the student was interested in pesticide analysis and during her investigations she learned about the QuEChERS (quick easy cheap effective rugged and safe) method of analysis.[29,30] After investigating the health impacts that led the U.S. to ban use of hexacholobenzene (HCB) as a pesticide, the student selected this compound as the analyte of interest. While this compound has been prohibited from use for many years, the compound may persist in certain soils and as a consequence root-vegetables such as carrots are routinely tested for its presence. In this experiment, a 2007 FDA trial version[31] of the QuEChERS method was utilized. The method was originally developed in 2003[29] by the Association of Official Analytical Chemists (AOAC) and has since been adopted by both the U.S. FDA and the European Food Safety Authority (EFSA) for the analysis of pesticides in fruits and vegetables. The method involves the liquid extraction of the pesticide followed by a cleanup procedure and subsequent solvent exchange before sample analysis by gas-chromatography/mass-spectrometry (GC/MS).

In this section, the student was expected to select an analyte and an SOP. It quickly became clear to the student that she had to make a number of choices based upon practical considerations. From the mentor's perspective, it was important to maintain good communication with the student during the early stages of project development so that a feasible well-grounded study could emerge. In addition to educational objectives of analyte and SOP selection, the student also gained a valuable appreciation for the vast number of compounds and procedures employed for the analysis of pesticides.

ACQUISITION OF EQUIPMENT AND MATERIALS

As is often the case in analytical work, the student was forced to make many selections based on limitations of the immediate situation. This student's desire to perform a pesticide analysis necessitated the selection of a method that used equipment which was readily available. Fortunately, a tandem gas-chromatography/mass-spectrometer (Varian 431-GC and Varian 210-MS), centrifuge (Thermoscientific CL-2), vortex mixer (BioRad BR-2000), and a rotary evaporator were available for this study. The student was required to obtain cleanup columns from 3M (Empore High Performance Extraction Disk Cartridges with Mixed Phase Cation (MPC) filters), HPLC grade (≥99.9%) acetonitrile, acetone, and HCB from Sigma-Aldrich, a triphenyl phosphate (TPP) standard solution (501.5 ± 2.5 μg/mL dissolved in acetonitrile), graphitic carbon black (GCB), and primary-secondary amine (PSA) from Agilent Technologies. The student made use of toluene from Matheson Coleman and Bell, magnesium sulfate ($MgSO_4 \cdot 7H_2O$) from Mallinckrodt, and reagent grade (≥98%) sodium chloride (NaCl) from Sigma-Aldrich. The student also purchased the Gerber brand of carrot baby food from a local grocery store to be used as the analyte matrix.

While making everyday decisions may seem routine for the seasoned veteran, students are not often forced to evaluate their surroundings and make a decision based on what they find. A search of the literature reveals important techniques for how to guide students in decision-making processes.[11,12,32,33] The early work of Walters highlights the importance of project ownership and communication while students encounter decision-making steps.[34−36] Overall, the exercise of gathering equipment and materials proved to be both a challenge and a new experience for the student, which mimics work typically encountered within an analytical laboratory.

DEMONSTRATION OF EXPERIMENTAL COMPETENCE

While the QuEChERS method has proven to be effective, like many SOPs, it requires an optimization step for the purposes of minimizing analysis time while maintaining adequate recoveries. The first optimization involved the evaluation of two cleanup methods: a column versus a dispersive cleanup. Column methods routinely require lengthy elution times and a dispersive method is investigated to reduce the time involved with the cleanup step. The second and third optimizations involve modifications to the Gas Chromatographic (GC) and Mass Spectroscopic (MS) analysis parameters, respectively. Inspection of the literature[37−45] provides a rich source of parameters for the GC portion of the experiment. Further, the MS used in this study is equipped with an ion trap which can be operated in Selective Ion Storage (SIS) mode.

During this experiment, the 2007 FDA trial version[31] of the QuEChERS method was carefully followed. The procedure begins with pesticide extraction into acetonitrile and is followed by a cleanup using PSA and GCB. In this work, both columns and individually weighed portions of PSA/GCB were prepared. Extractants were either passed over the columns or had the cleanup materials added directly to the sample vial and were dispersed throughout the solution by vortex mixing. The dispersive clean up required an additional centrifugation and decantation step, and despite the extra steps, the dispersive cleanup procedure was much faster than the column cleanup method. After the cleanup procedures, all of the samples underwent a solvent transfer process where acetonitrile was replaced by acetone. Several additions of acetone were added using a rotary evaporator between each addition. In total, eight samples were examined: four by the dispersive and four by the column cleanup. Of the four, two were spiked with HCB, one contained the matrix standard (HCB and TPP), and the last served as a blank. Table 1 provides a summary of the compositions of the eight samples.

While the process of extraction and sample clean up are well-defined, undergraduate students are not accustomed to acquiring all of the materials needed to carry out the

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Table 1. Descriptions of the Samples Analyzed

Sample Names	Final Compositions
dispersive cleanup matrix blank	no HCB or TPP
dispersive cleanup matrix standard	0.6 ppm of HCB and TPP
dispersive cleanup spike 1	0.6 ppm of HCB and TPP
dispersive cleanup spike 2	0.6 ppm of HCB and TPP
column cleanup matrix blank	no HCB or TPP
column cleanup matrix standard	0.6 ppm of HCB and TPP
column cleanup spike 1	0.6 ppm of HCB and TPP
column cleanup spike 2	0.6 ppm of HCB and TPP
solvent standard	0.6 ppm of HCB and TPP
solvent	HPLC grade Acetone

experiment. It took the student in this work several weeks (working about 9 h per week) to go through the process of ordering chemicals, setting up equipment, and carrying out the extraction procedure. During this early experimental stage, the students understanding of the procedure was evaluated by engaging in biweekly meetings in which the every aspect of the SOP was scrutinized. On one such occasion, the student was asked to explain the role of the GCB and PSA in the cleanup. From the limited response, the mentor encouraged the student to investigate the nature of these substances and their role in sample cleanup procedures. From an educational perspective, this example demonstrates how the mentor pushed the student toward an understanding of the procedure rather than viewing it as a simple recipe that was to be followed. Evidence of student understanding emerged as she began to suggest additional modifications to the extraction and cleanup portions of the experiment.

Once it was clear that the student had a thorough understanding of the extraction and cleanup portions of the experiment, the student was asked to begin the optimization and execution of the GC/MS portion of the experiment, which offered an entirely different research experience. After providing a refresher of material presented in the instrumental analysis course, the GC parameters were set using information from Gamon et al.[37] The mentor worked with the student during the first run, which yielded usable data in 57.9 min, and demonstrated the importance of mastering the manual injection technique. The student was encouraged to intentionally change the injection technique and to observe the effects on peak shapes. Once the student mastered the manual injection technique and demonstrated to the mentor that she could generate reproducible results with the solvent standard, she was encouraged to research the effects of a variety of operational parameters with the goals of achieving data in a shorter time with improved signal-to-noise (S/N) ratios.

The development of an instrumental procedure requires an understanding of the fundamental phenomenon at play and how parameters such as temperature variation affect results. It

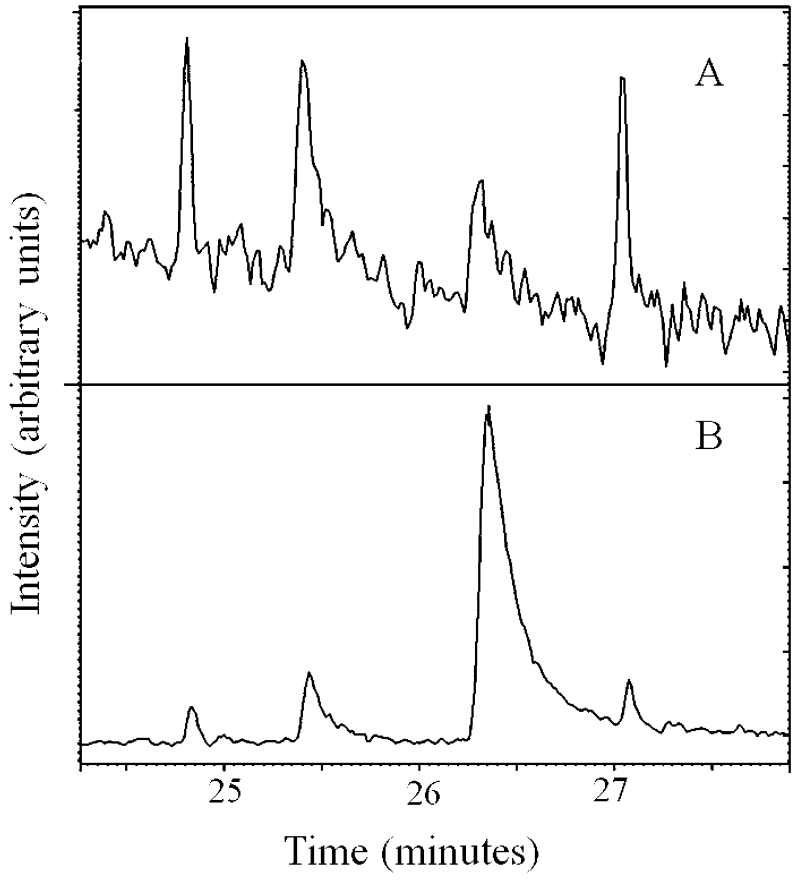

Figure 1. GC/MS analysis of TPP containing solvent standards. (A) Reconstructed total ion chromatogram and (B) chromatogram resulting from Selected Ion Storage of TPP fragments. GC Conditions: Varian VF-5 ms column (30 m × 0.25 mm × 0.25 μm), 5.00 μL injection, injector 275 °C, split injection ratio (5:1), column flow 1.0 mL/min. Oven program: hold 90 °C for 3.5 min, 25 °C/min ramp to 180 °C, hold 250 °C for 6 min, 5 °C/min ramp to 260 °C, hold 260 °C for 1 min, 25 °C/min ramp to 300 °C, hold 300 °C for 10 min. MS Conditions: EI Auto, 100 μs prescan (A) scan range 10−650 m/z at 1.0 s/scan and (B) SIS of the parent ion 326 m/z, identifying ions 325, 170, and 215 m/z, ionization storage level 35 m/z.

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Table 2. Gas Chromatographic Peak Areas and Peak Area Ratios for Each Sample

	Column Cleanup			Dispersive Cleanup		
	HCB (Counts)	TPP (Counts)	HCB/TPP (unitless)	HCB (Counts)	TPP (Counts)	HCB/TPP (unitless)
Solvent Standard 1	5920	16300	0.364	5830	17500	0.334
Matrix Standard 1	5070	16500	0.307	1720	13800	0.124
Spike 1	4050	12300	0.330	5560	24600	0.226
Matrix Standard 2	4350	17400	0.250	4000	16600	0.241
Matrix Standard 3	5470	19000	0.288	3760	18100	0.208
Spike 2	5240	14900	0.351	16200	31200	0.518
Matrix Standard 4	1030	5820	0.177	3990	19200	0.208
Matrix Standard 5	3310	13030	0.254	5680	24200	0.235
Solvent Standard 2	5350	22950	0.233	2580	16900	0.153

was vitally important for the mentor and student to review such relationships so that the student could apply logic to their parameter modifications. Further, it was important for the mentor to remain in close contact with the student to ensure a proper interpretation of the results. The first parameter that was examined was the split-mode ratio (i.e., carrier gas to sample ratio), which has the effect of adjusting the S/N ratio. The student quickly and easily optimized this parameter (5:1) by adjusting it until a maximum S/N ratio was observed. The second parameter the student attempted to adjust was the temperature program, which required significantly more time to optimize. The temperature program was changed 10 times before a final ramping procedure was found which reduced analysis time by 16.2 min per sample (41.7 min/sample). The importance of this improvement became clear to the student when she was asked to consider an industrial or other large sample throughput environment.

In addition to optimizing the GC, the operation of the mass spectrometer was also considered, specifically the selective ion storage modes. When the SIS mode is turned off (Figure 1A), the S/N ratio is about 5, which improved to about 20 when SIS mode was used (Figure 1B). Ten different SIS settings were attempted before arriving at the optimal chromatograph in Figure 1B. The process of optimizing the GC and MS experiments provided several opportunities for discussions to occur between the mentor and student. The conversations allowed the student to develop a better understanding of the physical principles which govern these techniques and afforded the mentor the opportunity to perform routine informal assessments of student learning.

PROCEDURE MODIFICATION, DATA ANALYSIS, AND INTERPRETATION OF RESULTS

As previously discussed, the modifications of the QuEChERS method performed in this work involved a variation of the sample cleanup procedure and optimizations of the GC/MS experiment. Once the analysis techniques were optimized, data could be collected to examine effects that the different cleanup procedures had of the recovery of HCB. With the use of the optimized parameters for the GC/MS experiment, all of the samples were analyzed in the following order: solvent rinse, solvent standard, matrix blank, matrix standard, spike 1, matrix standard, matrix standard, spike 2, matrix standard, matrix standard, and solvent standard and the results for HCB and TPP are shown in Table 2.

The solvent standard provides a baseline for the amounts of HCB and TPP found within each sample. The matrix standards allow one to assess how much if any HCB or TPP is lost in the extraction and cleanup processes. The matrix standards also allow one to assess cross contamination issues, which do not appear to be an issue in this study. There does appear to be reduced signals for dispersive matrix standard 1 and column matrix standard 4 samples and an increased signal for the dispersive spike 2 sample. To better assess the validity of the data set, the student was asked to perform a statistical analysis.

The 99% confidence interval ($N = 5$, $t = 4.60$) for the matrix standard peak ratios from the column and dispersive cleanup methods was calculated to be 0.255 ± 0.102 and 0.203 ± 0.096, respectively. For a sample spiked with 0.6 ppm of HCB and 0.6 ppm TPP, one would expect a peak ratio in the range of 0.153 and 0.357 for the column cleanup and 0.107 and 0.299 for the dispersive cleanup. The column cleanup spike 1 and spike 2 samples have peak ratios that fall within the expected range as does spike 1 of the dispersive method while spike 2 of this method falls above the expected range. For the three samples that fall within the confidence limits, this means that there is 99% confidence that the baby food samples contain only HCB from the spiked addition and that no additional HCB is present. For spike 2 of the dispersive method, the data suggests that there may have been additional HCB in this sample beyond the spiked addition. At this stage, it is important to mention that this study only examines two spiked samples while the QuEChERS method actually recommends a minimum of four spiked samples. It is believed that averaging of additional samples may have allowed one to rule out spike 2 of the dispersive method as an outlier. Considering that the baby food sample was obtained from a local grocery store, there is no reason to expect HCB in this sample, leading the authors to believe that the spike 2 sample from the dispersive cleanup procedure to be an outlier.

Despite this limited size of the data set, it is possible to calculate percentage recoveries for each sample using the following relationship recommended by the QuEChERS method.[31]

$$\frac{\left[\frac{\text{Peak response of pesticide in sample}}{\text{Peak response of internal standard in sample}}\right]}{\left[\frac{\text{Peak response of pesticide in matrix standard}}{\text{Peak response of internal standard in matrix standard}}\right]} \times 100 = \%\ \text{Recovery}$$

The percentage recoveries for the column cleanup are 107 and 114% for spike 1 and 2, respectively, and 94 and 215% for the dispersive cleanup. Spike 2 of the dispersive cleanup clearly overestimates the percentage recovery, but as was previously discussed, this sample is considered an outlier. Both of the column cleanup spikes and spike 1 of the dispersive method

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produce results that are considerably higher than the 47.3% recovery reported within the QuEChERS Trial Method[30] followed in this work.

The data compilation and analysis required several mentor-student conversations, which allowed the student to develop a better understanding of how to critically evaluate data and again offered the opportunity for the mentor to informally assess student comprehension.

SUMMARY

This study demonstrates how a previously established SOP was used as a mechanism for introducing an undergraduate student to chemical research and that the process was meaningful to the student in the following ways:

1. Selection of an analyte connected the student with the literature.
2. Selection of an SOP forced the student to evaluate equipment availability, which is an uncommon experience for undergraduates.
3. Acquisition of samples, standards, and equipment again forced the student to face experimental design and setup realities that are uncommon for undergraduates.
4. Demonstration of experimental competency provided the student with the opportunity to experience a student−mentor relationship.
5. Suggesting and carrying out a modification of the procedure allowed the student to gain confidence in her capacity to effect experimental results. This arguably might the most important aspect of this work because it provided an undergraduate student with the opportunity to fully recognize her influence of the experimental process. The student commented that she had developed from a "passive" follower of experimental procedures to an "active" participant in the experimental work.
6. The compilation and analysis of results tested the student's knowledge of previously learned information and reinforced the importance of comprehensive learning as opposed to memorization.
7. The production of a detailed laboratory report and subsequent contribution to this work provided the student with an opportunity to critique and reflect upon the experiment as a single process rather than a series of steps to be followed.
8. Presentation of the student's research at a Regional ACS meeting provided an opportunity to internalize the material and gain ownership of the research.

This work illustrates how an SOP can be used to expose students to analytical work and creates an atmosphere that enhances student learning and allows mentors to informally assess the depth of student understanding. In addition, there are only a few undergraduate experiences, namely, term papers, which enable students to undergo long-term development of thought. The process of simultaneously exposing students to both the depth and breadth of material simulates the work of analytical chemists and supports the suggestion that use of an SOP is an appropriate mechanism for introducing undergraduates to chemical research. Upon the basis of the positive impact of this work on student learning, additional work is underway.

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Notes

The authors declare no competing financial interest.

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REFERENCES

(1) Hage, D. S.; Chattopadhyay, A.; Wolfe, C. A. C.; Grundman, J.; Kelter, P. B. Determination of Nitrate and Nitrite in Water by Capillary Electrophoresis: An Undergraduate Laboratory Experiment. *J. Chem. Educ.* **1998**, *75* (12), 1588−1590.

(2) Seymour, P. Chromium Pollution: An Experiment Adapted for Freshman Engineering Students. *J. Chem. Educ.* **1999**, *76* (7), 927−928.

(3) Volker, E. J.; DiLella, D.; Terneus, K.; Baldwin, C.; Volker, I. The Determination of Ergosterol in Environmental Samples: An Interdisciplinary Project Involving Techniques of Analytical and Organic Chemistry. *J. Chem. Educ.* **2000**, *77* (12), 1621−1623.

(4) Selco, J.; Roberts, J. L.; Wacks, D. B. The Analysis of Seawater: A Laboratory-Centered Learning Project in General Chemistry. *J. Chem. Educ.* **2003**, *80* (1), 54−57.

(5) Giokas, D. L.; Paleologos, E. K.; Karayannis, M. I. Micelle-Mediated Extraction of Heavy Metals from Environmental Samples: An Environmental Green Chemistry Laboratory Experiment. *J. Chem. Educ.* **2003**, *80* (1), 61−64.

(6) Xia, K.; Pierzynski, G. Competitive Sorption between Oxalate and Phosphate in Soil: An Environmental Chemical Laboratory Using Ion Chromatography. *J. Chem. Educ.* **2003**, *80* (1), 71−75.

(7) Stock, N. L.; Martin, J. W.; Ye, Y.; Mabury, S. A. An Undergraduate Experiment for the Measurement of Perfluorinated Surfactants in Fish Liver by Liquid Chromatography-Tandem Mass Spectrometry. *J. Chem. Educ.* **2007**, *84* (2), 310−311.

(8) Miller, A. L.; Vaughan, P.; Sirvent, T. M. Effects of Storage Conditions on Lycopene Stability in Tomato Extracts: An Undergraduate Experiment. *J. Chem. Educ.* **2009**, *86* (11), 1304−1306.

(9) Mead, R. N.; Seaton, P. J. GC-MS Quantitation and Identification of Bisphenol-A Isolated from Water. *J. Chem. Educ.* **2011**, *88* (8), 1130−1132.

(10) MacNeil, J.; Gess, S.; Gray, M.; McGuirk, M.; McMullen, S. Mushroom Magic: Analysis of Metals in a Familiar Food. *J. Chem. Educ.* **2011**, *89* (1), 114−116.

(11) Wilczek-Vera, G.; Salin, E. D. Understanding Fluorescence Measurements through a Guided-Inquiry and Discovery Experiment in Advanced Analytical Laboratory. *J. Chem. Educ.* **2011**, *88* (2), 216−219.

(12) Boyce, M. C.; Singh, K. Student Learning and Evaluation in Analytical Chemistry Using a Problem-Oriented Approach and Portfolio Assessment. *J. Chem. Educ.* **2008**, *85* (12), 1633−1637.

(13) Cancilla, D. A. Initial Design and Development of an Integrated Laboratory Network: A New Approach for the Use of Instrumentation in the Undergraduate Curriculum. *J. Chem. Educ.* **2004**, *81* (12), 1809−1813.

(14) Mihok, M.; Keiser, J. T.; Bortiatynski, J. M.; Mallouk, T. E. An Environmentally Focused General Chemistry Laboratory. *J. Chem. Educ.* **2006**, *83* (2), 250−252.

(15) Adami, G. A New Project-Based Lab for Undergraduate Environmental and Analytical Chemistry. *J. Chem. Educ.* **2006**, *83* (2), 253−256.

(16) Engelen, D. L. V.; Suljak, S. W.; Hall, J. P.; Holmes, B. E. Undergraduate Introductory Quantitative Chemistry Laboratory Course: Interdisciplinary Group Projects in Phytoremediation. *J. Chem. Educ.* **2007**, *84* (1), 128−131.
(17) Shultz, M. J.; Kelly, M.; Paritsky, L.; Wagner, J. A Theme-Based Course: Hydrogen as the Fuel of the Future. *J. Chem. Educ.* **2009**, *86* (9), 1051−1090.
(18) Libes, S. M. Learning Quality Assurance/Quality Control Using U.S. EPA Techniques: An Undergraduate Course for Environmental Chemistry Majors. *J. Chem. Educ.* **1999**, *76* (12), 1642−1648.
(19) Lindsay, H. A.; McIntosh, M. C. Early Exposure of Undergraduates to the Chemistry Research Environment: A New Model for Research Universities. *J. Chem. Educ.* **2000**, *77* (9), 1174−1175.
(20) Headrick, K. L. Want Ads, Jobs, and Curriculum: A Survey of 1998 Chemistry Help-Wanted Ads. *J. Chem. Educ.* **2001**, *78* (9), 1271−1282.
(21) Kaliva, J. H. Realizing Workplace Skills in Instrumental Analysis. *J. Chem. Educ.* **2005**, *82* (6), 895−897.
(22) Weisshaar, D. E.; Earl, G. W.; Hanson, M. P.; Viste, A. E.; Kintner, R. R.; Duggy-Matzner, J. L. Instrumental Proficiency Program for Undergraduates. *J. Chem. Educ.* **2005**, *82* (6), 898−900.
(23) Vitha, M. F.; Carr, P. W.; Mabbott, G. A. Appropriate Use of Blanks, Standards, and Controls in Chemical Measurements. *J. Chem. Educ.* **2005**, *82* (6), 901−902.
(24) Hollenbeck, J. J.; Wixson, E. N.; Geske, G. D.; Dodge, M. W.; Tseng, T. A.; Clauss, A. D.; Blackwell, H. E. A New Model for Transitioning Students from the Undergraduate Teaching Laboratory to the Research Laboratory: The Evolution of an Intermediate Organic Synthesis Laboratory Course. *J. Chem. Educ.* **2006**, *83* (12), 1835−1843.
(25) Richter-Egger, D. L.; Hagen, J. P.; Laquer, F. C.; Grandgenett, N. F.; Shuster, R. D. Improving Student Attitudes about Science by Integrating Research into the Introductory Chemistry Laboratory: Interdisciplinary Drinking Water Analysis. *J. Chem. Educ.* **2010**, *87* (8), 862−868.
(26) Schaber, P. M.; Dinan, F. J.; St. Phillips, M.; Larson, R.; Pines, H. A.; Larkin, J. E. Juicing the Juice: A Laboratory-Based Case Study for an Instrumental Analytical Chemistry Course. *J. Chem. Educ.* **2011**, *88* (4), 496−498.
(27) Karukstis, K. K.; Wenzel, T. J. Enhancing Research in the Chemical Sciences at Predominantly Undergraduate Institutions: Recommendations of a Recent Undergraduate Research Summit. *J. Chem. Educ.* **2004**, *81* (4), 468−469.
(28) Committee on Professional Training: Undergraduate Programs. https://www.acs.org/content/dam/acsorg/about/governance/committees/training/acsapproved/degreeprogram/undergraduate-research-acs-cpt-supplement.pdf (accessed May 2014).
(29) Anastassiades, M.; Lehotay, S. J.; Stajnbaher, D.; Schenck, F. J. Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce. *J. AOAC Int.* **2003**, *86* (2), 412−431.
(30) Schenck, F. J.; Hobbs, J. E. Evaluation of the Quick, Easy, Cheap, Effective, Rugged, and SAFE (QuEChERS) Approach to Pesticide Residue Analysis. *Bull. Environ. Contam. Toxicol.* **2004**, *73*, 24−30.
(31) Schenck, F.; Wong, J. *Quechers Method Trial*; Technical report for the FDA-SERL: Atlanta, GA; November 2007.
(32) Jackson, P. T.; Walters, J. P. Role-Playing in Analytical Chemistry: The Alumni Speak. *J. Chem. Educ.* **2000**, *77* (8), 1019−1025.
(33) Iimoto, D. S.; Frederick, K. A. Incorporating Student-Designed Research Projects in the Chemistry Curriculum. *J. Chem. Educ.* **2011**, *88*, 1069−1073.
(34) Walters, J. P. Role Playing Analytical Chemistry Laboratory Part 1: Structural and Pedagogical Ideas. *Anal. Chem.* **1991**, *63* (20), 977A−985A.
(35) Walters, J. P. Role Playing Analytical Chemistry Laboratory Part 2: Physical Resources. *Anal. Chem.* **1991**, *63* (22), 1077A−1087A.
(36) Walters, J. P. Role Playing Analytical Chemistry Laboratory Part 3: Experimental Objectives and Design. *Anal. Chem.* **1991**, *63* (24), 1179A−1191A.
(37) Gamon, M.; Lleó, C.; Ten, A.; Mocholi, F. Multiresidue Determination of Pesticides in Fruit and Vegetables by Gas Chromatography/Tandem Mass Spectrometry. *J. AOAC Int.* **2001**, *84*, 1209−1216.
(38) Cripps, D. J.; Peters, H. A.; Gocmen, A.; Dogramici, I. Porphyria Turcica due to Hexachlorobenzene: A 20 to 30 Year Follow-Up Study on 204 Patients. *Br. J. Dermatol.* **1984**, *111*, 413−422.
(39) Hercegová, A.; Dömötörová, M.; Hrouzková, S.; Matisová, E. Study on Pesticide Residues in Apples, Apple-Based Baby Food, and Their Behavior during Processing Using Fast GC-MS Multiresidue Analysis. *Int. J. Environ. Anal. Chem.* **2007**, *87*, 957−969.
(40) Hercegová, A.; Húšková, R.; Matisová, E. Evaluation of Different Calibration Approaches in Pesticide Residues Analysis in Non-Fatty Food Using Fast GC-MS. *Int. J. Environ. Anal. Chem.* **2010**, *90*, 188−204.
(41) Húšková, R.; Matisová, E.; Ondreková, S.; Ďurčanská, J. Fast GC-MS of Endocrine Disrupting Chemicals. *Int. J. Environ. Anal. Chem.* **2010**, *90*, 173−187.
(42) Lesueur, C.; Gartner, M.; Mentler, A.; Fuerhacker, M. Qualitative and Quantitative Analysis of Polar Pesticide Multiresidues in Leaf Samples with a Liquid Chromatography-Ion-Trap Mass-Selective Detector. *Int. J. Environ. Anal. Chem.* **2007**, *87*, 1013−1032.
(43) Mercer, G. E. Determination of 112 Halogenated Pesticides Using Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring. *J. AOAC Int.* **2005**, *88*, 1452−1462.
(44) Paíga, P.; Morais, S.; Correia, M.; Delerue-Matos, C.; Alves, A. Determination of Carbamate and Urea Pesticide Residues in Vegetables Using Microwave-assisted Extraction and Liquid Chromatography. *Int. J. Environ. Anal. Chem.* **2009**, *89*, 199−210.
(45) Wang, M. J.; Jones, K. C. Occurrence of Chlorobenzenes in Nine United Kingdom Retail Vegetables. *J. Agric. Food Chem.* **1994**, *42*, 2322−2328.