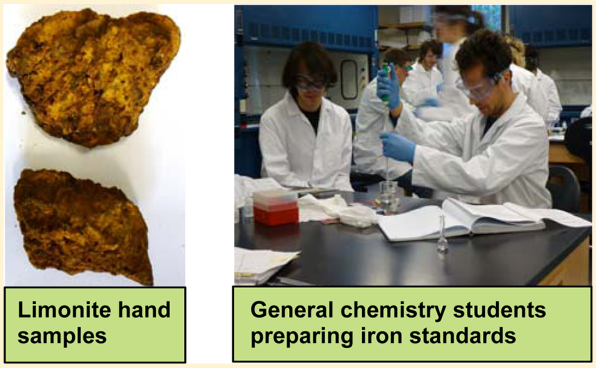
JOURNAL OF CHEMICAL EDUCATION
Laboratory Experiment
pubs.acs.org/jchemeduc

Quantitative Determination of Iron in Limonite Using Spectroscopic Methods with Senior and General Chemistry Students: Geology-Inspired Chemistry Lab Explorations

A. M. R. P. Bopegedera,* Christopher L. Coughenour,† and Andrew J. Oswalt‡

Department of Chemistry, The Evergreen State College, Olympia, Washington 98505, United States

S *Supporting Information*



ABSTRACT: Limonite is the field term for a mixed assemblage of ferric oxyhydroxides, often containing nonferric silicate impurities. It is abundant on Earth's surface, possesses variable iron content, and is easily recognized by distinctive yellow and ochre hues. Limonite is a unique centerpiece for undergraduate chemistry laboratories because each sample represents a true unknown to faculty and students alike, and because limonite does not digest readily with common methods. Senior students were guided through a primary literature review to assess and establish an appropriate digestion method. Students then constructed the procedure for determining the iron content in limonite using atomic absorption spectroscopy. A final report, produced in the style of this *Journal*, completed the start-to-finish process used by scientists, helping students learn how novel problems are solved in the laboratory. General chemistry students were provided with limonite extracts and used UV—vis spectroscopy to determine their iron contents, gaining proficiency in wet-lab, data collection, and analysis skills. Lab reports included an interdisciplinary discussion/interpretation of results from chemistry and geologic perspectives. The method used by students for digestion and iron extraction was validated using goethite and iron(III) oxide as standards (<3% error). The two spectrometric methods provided comparable results for the iron content in limonite. Despite challenging all students, these experiments were rated favorably in written evaluations, and students self-rated their learning gains as very high. Limonite experiments promote curiosity, discussion, and departure from laboratory exercises with predetermined results. Students become vested in analyzing a geologically important material that is inherently complex and heterogeneous.

KEYWORDS: *First-Year Undergraduate/General, Upper-Division Undergraduate, Interdisciplinary/Multidisciplinary, Physical Chemistry, Communication/Writing, Inquiry-Based/Discovery Learning, Geochemistry, Instrumental Methods, Quantitative Analysis, Spectroscopy*

■ BACKGROUND

Limonite is a general name given to a mixed assemblage of hydrated solid ferric oxyhydroxides that also frequently contains the anhydrous species goethite [α-FeO(OH)] and lepidocrocite [γ-FeO(OH)].[1] Limonite is common geologically and can be found in wetlands, where it forms "bog iron", and streams affected by acid-mine drainage (Figure 1), as a component of "yellow boy".[2] Limonite can also be found in modern and ancient soils, sometimes yielding hard concretions (iron pans) that inhibit root growth. The red (and yellow) hues of rocks and soil are often determined by iron constituents, even in low concentrations (several percent by mass),[3] and for thousands of years limonite has been a pigment used in yellow ochre and raw sienna[4] (Figure 2). Due to its low grade and often restricted occurrence, limonites such as "bog iron" are rarely used in modern iron and steel production.

Limonite is an important component of the iron cycle at Earth's surface. Iron, at over 4% by mass, is the fourth most common element in Earth's crustal lithosphere.[6] Most terrestrial iron occurs in the divalent state and is sequestered geologically, primarily in silicate and sulfide minerals often deeply buried in the crust and mantle. Once brought to the surface, iron-bearing minerals tend to undergo geologically rapid weathering. The typical steps are dissolution of the parent mineral, the subsequent oxidation of iron, and hydrolysis that yields ferric oxides or oxyhydroxides.[7] Depending on conditions, any of at least 10 well-known iron compounds may form that may be anhydrous or variably hydrated.[2] Most of these consist of close-packed sheets of oxygen with Fe(III) in the interstitial spaces.[8] Ultimately, oxyhydroxide minerals are formed that include ferrihydrite, goethite, and lepidochrocite. Amorphous oxyhydroxides are also formed.

Received: February 22, 2016
Revised: August 10, 2016

ACS Publications

DOI: 10.1021/acs.jchemed.5b00776
J. Chem. Educ. XXXX, XXX, XXX−XXX

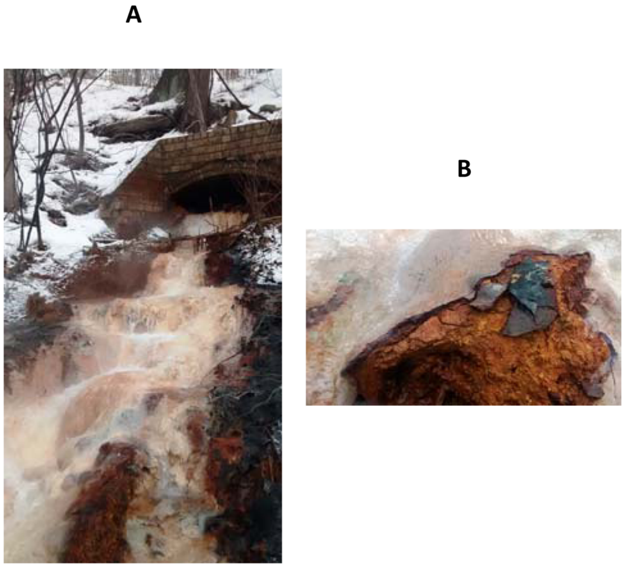


Figure 1. Limonite deposited from acid-mine discharge in a tributary of Solomon Run near Johnstown, Pennsylvania. (A) The white precipitate is aluminum hydroxides.[5] (B) Close-up of the limonite deposit in Solomon Run.

Figure 2. Streak plate revealing the characteristic yellow-brown streak of limonite (the compound color in powdered form).

Ferric oxides, an integral part of the reaction series forming the oxyhydroxides, and other iron-bearing products such as jarosite,[9] are common components of limonite. In reality, silicates and other non-iron-bearing minerals are also often present in association with limonite. The chemical constitution of natural limonite samples is, thus, highly heterogeneous, and its schematic chemical formula is $FeOOH \cdot nH_2O$.[10] Identification of the individual mineral species and other components of the limonite assemblage is generally not possible in the field and can be difficult in the laboratory, requiring techniques beyond X-ray diffraction due to adsorption effects and the poorly crystalline and/or disordered nature of some of the constituents.[2]

■ PEDAGOGICAL RATIONALE

Laboratory analyses of materials that are common in students' lives have been increasingly embraced by chemical educators to make learning relevant and to engage students' interest. Sherren presented compelling arguments for using real-life samples for unknowns in the chemistry curriculum since such samples inherently contain "impurities" that must be taken into consideration as professional chemists would have to do.[11] Determination of metals in real-world samples using spectrometric methods have been published in this *Journal*.[12−26] Although the connections between chemistry and geology have been explored,[27−47] publications on the analysis of rock/mineral samples have been rare.[48] The analysis of limonite, a complex and common Earth material, presented a valuable teaching opportunity to engage students at the first-year and senior levels. The experiments used spectrometric techniques while also developing wet-lab, data analysis, spreadsheet use, and technical writing skills.

The rich interdisciplinary learning environment at The Evergreen State College[49−53] inspired the invention of this experiment in a course that integrated general chemistry with physical geology where studying geologically important samples using chemistry was a recurring theme. Other interdisciplinary experiments included quantification of carbonate in marble,[54] extraction of copper from malachite,[55] analysis of lake water samples[56] (collected during a geology field trip), and flame tests and thin-layer chromatography to identify metals in minerals.[57]

The true unknown nature and heterogeneous chemical composition of limonite makes the determination of its iron content a sophisticated, yet feasible, research problem to teach senior students the process scientists use to solve novel problems in the laboratory. By guiding students' efforts through a series of steps with measurable outcomes (see Learning Outcomes Part 1 below), we helped them acquire valuable transferable skills. Limonite served to motivate student engagement due to its natural variability (results are not known *a priori*) and environmental significance.

We also developed a method to quantify the iron content in limonite using optical spectroscopy in the general chemistry laboratory to teach multiple, transferable skills (see Learning Outcomes Part 2 below). It was conducted during the second half of a year-long laboratory sequence so students could work independently to demonstrate competency as well as reinforce previously acquired skills. This experiment, also feasible at institutions with large enrollments, engaged all students and challenged even the most motivated. It allowed students to explore geologic samples that, unlike those given to most general chemistry students, possessed complexity and heterogeneity such as that encountered in nature. It is suitable for a laboratory practical[58,59] or for an inquiry-based lab.

■ LEARNING OUTCOMES

Grading rubrics (Supporting Information) with the following learning outcomes were given to both groups of students prior to the laboratory exercises.

Part 1. Senior Chemistry Laboratory

Students' work was collected and graded for outcomes 1−5 below, to ensure mastery of each step before proceeding to the next.

1. *Literature search:* Perform a literature search to identify suitable method to quantitatively digest limonite. Learn literature search and tracking tools (including Zotero[60]) in workshops conducted by the reference librarian.
2. *Understand the literature:* Describe the chemistry of the digestion process as detailed in the article selected by the instructor[61] from those submitted by students.[61−70]
3. *Prepare solutions for analysis:* Determine the desired concentrations of five iron standards using the AA spectrometer manual for guidance. Describe the process of their preparation, and those of the limonite digest, method blank, matrix blank, quality control (QC) standard, and recovery standard and their functions.

4. *Chemicals and glassware:* Make lists of chemicals and required glassware to be acid washed to prepare the above solutions.
5. *Method development:* Modify the experimental procedure to use graphite (not platinum) crucibles and air-acetylene (not nitrous oxide-acetylene) flame in the AA spectrometer[71] in response to the class discussion on the benefits, challenges, and safety concerns presented in refs 61 and 71.
6. *Proficiency with the AA spectrometer:* Attend instructional workshops and earn the "driver's license" to independently operate this instrument. Learn the theory of AA spectroscopy in lectures.
7. *Data collection and analysis:* Working in pairs, collect data and generate a class data set. Analyze the data set individually.
8. *Technical writing:* Submit a formal paper of results following guidelines of this *Journal.*[72] Edit and rewrite papers iteratively with support from the writing instructor for final evaluation by the chemist (for scientific merit) and the writing instructor (for technical writing skills).

Part 2. General Chemistry Laboratory

Students were required to work independently through the following steps, demonstrating competence in previously acquired skills.

1. *Quantitative dilution using volumetric glassware:* Prepare quantitative dilutions of the limonite extract and the iron stock solution following written instructions.
2. *Proficiency with the UV−visible spectrometer:* Collect absorbance data for the iron solutions and generate a class data set.
3. *Using spreadsheet software for calculations and graphs:* Plot graphs and analyze the class data set using spreadsheets (this involved using unfamiliar units, multiple unit conversions, and stoichiometric calculations).
4. *Formal report:* Write report detailing procedures for quantitative dilutions, data, analysis, detailed calculations, descriptive statistics, accuracy and precision of data, and sources of error.
5. *Interdisciplinary connections:* Include discussions of observed limonite variability in the context of geology.

■ EXPERIMENTAL METHODS

Part 1. Senior Chemistry Laboratory

The iron in limonite[73] was extracted into solution by fusing with lithium metaborate ($LiBO_2$) and lithium tetraborate ($Li_2B_4O_7$) in graphite crucibles at 1000 °C and dissolving the resulting beads (Figure 3A) in nitric acid.[71] Two different limonite hand samples (limonite X and limonite Y) were used. Each student pair prepared five iron standards, a QC standard (to verify the quality of iron standards), a matrix blank (instrument blank), a method blank (to ensure that iron was not externally introduced during experiment), and a recovery standard (to test the efficacy of the experimental method) and collected data with the AA spectrometer[74] (248.3 nm wavelength).

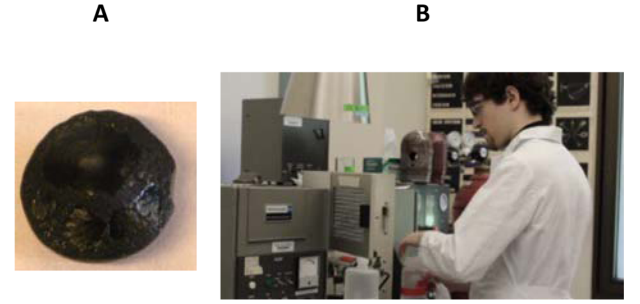


Figure 3. (A) Limonite fusion bead, ∼1 cm in diameter. (B) Student collecting data with the AA spectrometer.

Part 2. General Chemistry Laboratory

Students were provided with the limonite extract and a 10 ppm iron(II) stock solution. Each student prepared quantitative dilutions of these solutions adding 0.1% 2,2′-bipyridyl during the process to form a strawberry red Fe(II) complex[75] (Figure 4A). Absorption spectra of this red complex were recorded with UV−vis spectrometers[76] at a λ_{max} of 518 nm (Figure 4B). Individual student data were gathered into a class spreadsheet for analysis. The experiment, completed within a 3 h lab period, furthered students' skill with this instrument. Laboratory staff prepared extracts of goethite (Figure 4C) and iron(III) oxide (as recovery standards) following the same procedure as limonite. UV−vis spectra of these recovery standards were also recorded (Table 1).

Digestion Process. The mixture of lithium tetraborate (mp 920 °C) and lithium metaborate (mp 845 °C) flux melts at the oven temperature (1000 °C). Limonite dissolves in this melt, mobilizing the iron ions. After cooling, the resulting bead (Figure 3A) is digested with nitric acid to extract iron ions into aqueous solution. Orthoboric acid (inhibits the polymerization of silica preventing the solution from turning cloudy) and cesium chloride solutions (only for AA analysis) are added, and the solution is filtered. Cesium chloride (ionization suppressant), orthoboric acid, and the lithium borates prevent interelement interferences in the AA spectrometer. This technique made clear solutions from limonite samples containing silicates,[61,64,77,78] for analysis in the spectrometers. It is preferred to hydrofluoric acid for metal extraction due to safety concerns. See Supporting Information for detailed experimental procedures.

■ HAZARDS

Hydrochloric and nitric acids are strong acids. They, along with lithium tetraborate, iron(III) oxide, 2,2′ bipyridyl, hydroxylamine hydrochloride, and sodium acetate can cause serious damage to eyes, skin, and the respiratory system. Cesium chloride is harmful to skin and eyes. Boric acid may impair fertility or harm an unborn child. Lithium metaborate is a skin irritant. Ferrous ammonium sulfate hexahydrate is corrosive, fatal if ingested, and harmful if inhaled or absorbed through skin. Use oven mitts to remove hot crucibles from the muffle furnace.

Reading the Material Safety Data Sheets of all chemicals; understanding the hazards; and gaining familiarity with proper handling, disposal, and response to possible exposures were integrated into the prelaboratory assignments. All chemicals (CAS numbers in Supporting Information) were commercially purchased at 99.999% purity.[79]

■ RESULTS AND ANALYSIS

Part 1. Senior Chemistry Laboratory

Each student pair generated a standard curve with AA spectrometric data for iron standards (Figure 5A).

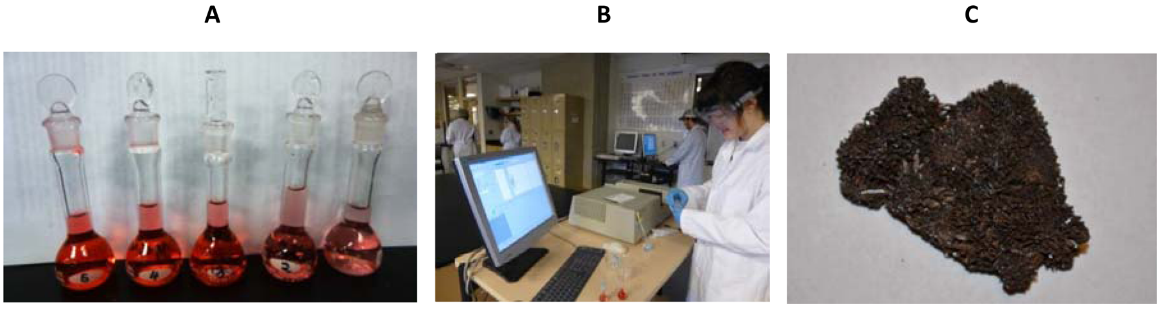


Figure 4. (A) Iron standards prepared by students. (B) Students working with UV–vis spectrometers. (C) Goethite sample used as a recovery standard.

Table 1. Analytic Results of Determination of Iron in Limonite by Spectrometric Methods

Sample	Mean Mass of Iron, % ±SD
AA Spectroscopy	
Limonite sample "X" (mean, $N = 7$)[a]	24.1 ± 1.5
Limonite sample "Y" (mean, $N = 7$)[a]	21.5 ± 0.9
Recovery standard (Fe_2O_3)	68.1
Theoretical value (Fe_2O_3)	69.9
UV–Vis Spectroscopy	
Limonite sample "X" (mean, $N = 41$)	21.8 ± 2.2
Limonite sample "X"[b]	21.9
Recovery standard (Fe_2O_3)[b]	71.1
Theoretical value (Fe_2O_3)	69.9
Recovery standard (Goethite)[b]	61.9
Theoretical value (Goethite)	62.9

[a] Limonite samples X and Y represent the two different hand samples used in the AA spectroscopic study. [b] Data collected by laboratory staff for comparison.

Part 2. General Chemistry Laboratory

Each student prepared a Beer–Lambert graph from the class data set collected with the UV–vis spectrometer for iron standards (Figure 5B).

Spreadsheet software was used for graphing and data analysis with both groups. Iron content in samples (Table 1) was determined using standard curves in Figure 5. Data, calculations, and analysis are available in the Supporting Information.

■ DISCUSSION

Part 1. Senior Chemistry Laboratory

The instrument response for the QC standard (Figure 5A) confirms the quality of the iron standards. Iron was not detected in the method blank, confirming that it was not introduced externally during experimental procedures. The low percent error (2.6% compared with the theoretical value in Table 1) for the recovery standard (Fe_2O_3) is evidence that the fusion method quantitatively extracted iron from solid samples into solution.

The iron contents in limonite hand samples X and Y are similar, perhaps because the commercial supplier collected them from the same location. The standard deviation of students' data was high, most likely due to errors introduced during the multiple quantitative transfer steps. Data in Table 1 were collected by one student (A.J.O., coauthor on this paper).

The burner head of the AA spectrometer clogged several times during the experiment (most likely due to the formation of iron oxides) and required cleaning. As a result, students gained experience in instrument maintenance. This experiment required five 3 h lab periods primarily because all students earned their driver's licenses on a single AA instrument. With the experience gained, laboratory time could be reduced to four periods.

Part 2. General Chemistry Laboratory

The scatter in the Beer–Lambert graph (Figure 5B) may be attributed to general chemistry students' lack of experience in preparing standards and the use of multiple spectrometers.[76] About 10% of students' data were discarded due to errors from poor technique. However, access to the class data set enabled every student to complete the data analysis.

The low percent errors for the two recovery standards (1.7% for Fe_2O_3 and 1.5% for goethite respectively, compared with their theoretical values in Table 1) reflect that the extraction method was successful for both solids. Goethite was used because it is often the most common mineral species present in limonite,[80] and it is valuable to assess the method's efficacy in

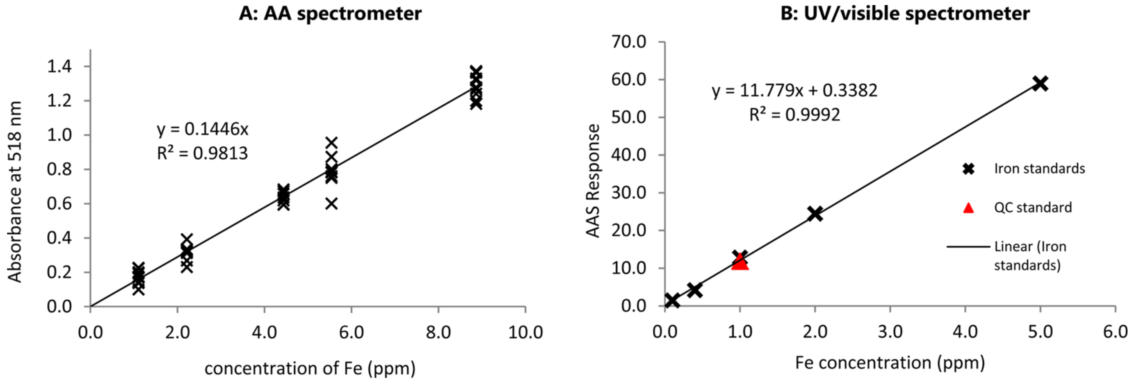


Figure 5. (A) Standard curve from iron standards generated by a student (A.J.O., coauthor on this paper) via the atomic absorption spectrophotometer. (B) Class data set ($N = 47$) from the UV–vis spectrometer on a Beer–Lambert graph. The regression lines represent the best fits.

DOI: 10.1021/acs.jchemed.5b00776
J. Chem. Educ. XXXX, XXX, XXX–XXX

digesting and recovering iron from its structure. Limonite has the additional caveat of possessing silicate "impurities" that must also be digested; however, the fusion method has been shown to be effective with these minerals.[71] In future trials the cheaper hematite (Fe_2O_3) can be solely used as the recovery standard.

Within experimental error, the percent masses of iron in limonite determined by the two different spectrometric methods are similar. This validates the use of UV−vis spectroscopy as a low cost option, particularly with general chemistry students. In future trials, we hope to use limonite and soil samples collected from our locality or field trips to make this experiment even more relevant for students.

Student Feedback and Evidence of Learning Gains

Part 1. Senior Chemistry Laboratory. Every student successfully achieved the learning outcomes. The process of iterative writing and editing greatly improved the quality of the lab reports even though students initially resisted this process. The reference librarian and the writing instructor, a veteran at the college, wrote:

> *This is the best teaching experience I have had in my career. The writing portion was dramatically enhanced by the collaboration of the faculty: a chemist and a rhetorician/librarian. Co-teaching allowed for substantive discussion of scientific conventions as practiced in laboratory reports and research papers. I was able to develop a sustained relationship with each student as I responded to their work individually and drew them into group discussions of scientific rhetoric: the effective uses of passive voice, the role of storytelling, and the art of description. Students were persuaded of the importance of these literacies because they were evaluated on their performance.*

Students' feedback included the following:

> *Deriving an experimental procedure for the limonite laboratory was very rewarding, even though it precipitated many problems over the course of several weeks. Our professor could have provided us the procedure for this laboratory but instead we got to experience firsthand the process used by scientists. That kind of experience is invaluable.*
>
> *We spent what felt like a month researching and refining a method for the limonite laboratory. Although this was very frustrating at the time, it was the first time a teacher had put me in a situation to dedicate myself to improving my research skills, double check my answers, and do my best to make sure that the method was as accurate as possible. I realized how important this skill is later in the year as I moved on to a research project.*

Part 2. General Chemistry Laboratory. Students worked with real samples, large data sets, and used unfamiliar units (and unit conversions), experiences uncommon in the general chemistry laboratory. Students' proficiency with transferable skills such as using spreadsheet software and quantitative reasoning skills were reinforced. The overall quality of students' lab reports was very good showing their investment and interest. A discussion following this laboratory, co-led by the chemist and the geologist, on the properties of limonite, its geological origins, solid state structure, chemical composition, and possible impurities further enriched this interdisciplinary learning experience. Students gained firsthand appreciation of the contributions chemical analysis makes to study geological samples.

Students' feedback included the following:

> *We were able to begin applying new knowledge to real world situations through laboratory work on field samples.*
>
> *The limonite laboratory connected geology and chemistry in a way that kept me engaged and wanting to learn new concepts in these subjects.*
>
> *This laboratory was creative; the instructors allowed me to figure things out on my own, giving me better insight into what it is like to be a scientist.*

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00776.

CAS numbers for all chemicals used in the limonite labs (PDF, DOCX)
Grading rubric for senior chemistry lab (PDF, DOCX)
General Chemistry lab notebook evaluation rubric (PDF, DOC)
General chemistry laboratory instructions and data analysis (PDF, DOCX)
Senior chemistry laboratory experimental instructions and data analysis (PDF, DOCX)

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: bopegedd@evergreen.edu.

Present Addresses

†Department of Energy and Earth Resources, University of Pittsburgh at Johnstown, Johnstown, Pennsylvania 15904.
‡Department of Chemistry, Oregon State University, Corvallis, Oregon 97331.

Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

Sara Huntington, Reference Librarian and Writing Instructor at The Evergreen State College, taught library research tools and supported the writing component. One of us (A.M.R.P.B.) was funded by the Evergreen State College Foundation to write this paper. Dan Cygnar is acknowledged for Figures 3A and 4C.

■ REFERENCES

(1) Schaetzl, R. J.; Anderson, S. *Soils: Genesis and Geomorphology*; Cambridge University Press: New York, 2005; pp 58−60.

(2) Jambor, J. L.; Dutrizac, J. E. Occurrence and Constitution of Natural and Synthetic Ferrihydrite, a Widespread Iron Oxyhydroxide. *Chem. Rev.* **1998**, *98* (7), 2549−2586.

(3) Schwertmann, U. The Effect of Pedogenic Environments on Iron Oxide Minerals. In *Advances in Soil Science*; Springer-Verlag; New York, 1985; Vol. *1*, pp 171−200.

(4) Bikiaris, D.; Daniilia, S.; Sotiropoulou, S.; Katsimbiri, O.; Pavlidou, E.; Moutsatsou, A. P.; Chryssoulakis, Y. Ochre-Differentiation through Micro-Raman and Micro-FTIR Spectroscopies: Application on Wall Paintings at Meteora and Mount Athos, Greece. *Spectrochim. Acta, Part A* **2000**, *56* (1), 3−18.

(5) Brasted, R. C. Nature's geological paint pots and pigments. *J. Chem. Educ.* **1971**, *48* (5), 323−324.

(6) Wedepohl, K. H. The Composition of the Continental Crust. *Geochim. Cosmochim. Acta* **1995**, *59* (7), 1217−1232.

(7) Schwertmann, U. *Encyclopedia of Soil Science*; Springer: Dordrecht, The Netherlands, 2008; pp 363−369.

(8) Bernal, J. D.; Dasgupta, D. R.; Mackay, A. L. The Oxides and Hydroxides of Iron and their Structural Inter-Relationships. *Clay Miner.* **1959**, *4* (21), 15−30.
(9) Brown, J. B. Jarosite-Geoethite Stabilities at 25 °C, 1 ATM. *Miner. Deposita* **1971**, *6* (3), 245−252.
(10) Klein, C. *Mineral Science*, 22nd ed.; John Wiley and Sons: Inc.: New York, 2002; pp 626.
(11) Sherren, A. T. The Use of Real Life Samples for Unknowns in Analytical Chemistry. *J. Chem. Educ.* **1991**, *68* (7), 598−599.
(12) Quigley, M. N. Determination of Calcium in Analgesic Tablets Using Atomic Absorption Spectrophotometry. *J. Chem. Educ.* **1994**, *71* (9), 800.
(13) Strohl, A. N. The Flame Spectrometric Determination of Calcium in Fruit Juice by Standard Addition. *J. Chem. Educ.* **1985**, *62* (4), 343.
(14) Kostecka, K. S. Atomic Absorption Spectroscopy of Calcium in Foodstuffs in Non-Science-Major Courses. *J. Chem. Educ.* **2000**, *77* (10), 1321−1323.
(15) Bazzi, A.; Kreuz, B.; Fischer, J. Determination of Calcium in Cereal with Flame Atomic Absorption Spectroscopy: An Experiment for a Quantitative Methods of Analysis Course. *J. Chem. Educ.* **2004**, *81* (7), 1042−1044.
(16) Laswick, P. H. The Iron Content of Breakfast Cereals. *J. Chem. Educ.* **1973**, *50* (2), 132.
(17) Paschal, D. C. An Atomic Absorption Experiment in Food Chemistry. *J. Chem. Educ.* **1976**, *53* (3), 165.
(18) Pinnell, R. P.; Zanella, A. W. Determination of Iron by Atomic Absorption in Vitamin-Mineral Tablets. *J. Chem. Educ.* **1981**, *58* (5), 444.
(19) Hooker, P. Mineral Analysis of Whole Grain Total Cereal. *J. Chem. Educ.* **2005**, *82* (8), 1223−1225.
(20) Atkins, R. C. Colorimetric Determination of Iron in Vitamin Supplement Tablets: A General Chemistry Experiment. *J. Chem. Educ.* **1975**, *52* (8), 550.
(21) Adams, P. E. Determining Iron Content in Foods by Spectrophotometry. *J. Chem. Educ.* **1995**, *72* (7), 649−651.
(22) Sheffield, M.-C.; Nahir, T. M. Analysis of Selenium in Brazil Nuts by Microwave Digestion and Fluorescence Detection. *J. Chem. Educ.* **2002**, *79* (11), 1345−1347.
(23) Donais, M. K.; Whissel, G.; Dumas, A.; Golden, K. Analyzing Lead Content in Ancient Bronze Coins by Flame Atomic Absorption Spectroscopy: An Archaeometry Laboratory with Nonscience Majors. *J. Chem. Educ.* **2009**, *86* (3), 343−346.
(24) Pomeroy, R. K.; Drikitis, N.; Koga, Y. The Determination of Zinc in Hair Using Atomic Absorption Spectroscopy. *J. Chem. Educ.* **1975**, *52* (8), 544−545.
(25) Markow, P. G. Determining the Lead Content of Paint Chips: An Introduction to AAS. *J. Chem. Educ.* **1996**, *73* (2), 178−179.
(26) Coleman, M. F. M. The Determination of Lead in Gasoline by Atomic Absorption Spectrometry. *J. Chem. Educ.* **1985**, *62* (3), 261−262.
(27) Bye, R. A Simple Extraction Procedure for the Determination of Lead in Gasoline by Atomic Absorption Spectrometry. *J. Chem. Educ.* **1987**, *64* (2), 188.
(28) Pezaro, P.; Mazor, E.; Samuel, D.; Ben-Zvi, N. Chemistry of Rocks and Minerals: A New Interdisciplinary Curriculum for Secondary Schools. *J. Chem. Educ.* **1978**, *55* (6), 383−385.
(29) Hostettler, J. D. Geochemistry for Chemists. *J. Chem. Educ.* **1985**, *62* (10), 823−831.
(30) Streng, E. F. Geology as a Part of the General College Chemistry Course. *J. Chem. Educ.* **1955**, *32* (12), 611−613.
(31) Schobert, H. H. The Geochemistry of Coal. Part I: The Classification and Origin of Coal. *J. Chem. Educ.* **1989**, *66* (3), 242−244.
(32) Schobert, H. H. The Geochemistry of Coal. Part II: The Components of Coal. *J. Chem. Educ.* **1989**, *66* (4), 290−293.
(33) Neff, T. R. Chemistry and the Modern Prospector. *J. Chem. Educ.* **1981**, *58* (9), 699−703.
(34) Morita, H. Peat and Its Organic Chemistry. *J. Chem. Educ.* **1980**, *57* (10), 695−696.
(35) Petrakis, L.; Grandy, D. W. Coal Analysis, Characterization and Petrography. *J. Chem. Educ.* **1980**, *57* (10), 689−694.
(36) Hocking, M. B. The Chemistry of Oil Recovery from Bituminous Sands. *J. Chem. Educ.* **1977**, *54* (12), 725−729.
(37) Nelson, T. W. The Origin of Petroleum. *J. Chem. Educ.* **1954**, *31* (8), 399−402.
(38) Fleischer, M. The Abundance and Distribution of the Chemical Elements in the Earth's Crust. *J. Chem. Educ.* **1954**, *31* (9), 446−455.
(39) Hulme, R. Mineral Sources and Extraction Methods for the Elements: A Correlation Based on Elements' Positions in the Periodic Table. *J. Chem. Educ.* **1956**, *33* (3), 111−113.
(40) Adler, I. Geochemical Exploration of the Moon. *J. Chem. Educ.* **1984**, *61* (1), 36−39.
(41) Howard, W. A. The Relationship between Balancing Reactions and Reaction Lifetimes: A Consideration of the Potassium−Argon Radiometric Method for Dating Minerals. *J. Chem. Educ.* **2005**, *82* (7), 1094−1098.
(42) Fairhall, A. W. Dating of Uranium Minerals by the Specific Radioactivity of Lead. *J. Chem. Educ.* **1963**, *40* (12), 626−633.
(43) Rowe, M. W. Radioactive Dating: A Method for Geochronology. *J. Chem. Educ.* **1985**, *62* (7), 580−584.
(44) Walker, L.; Lee, P. J. The Rocky Road to Chemistry. *J. Chem. Educ.* **1990**, *67* (4), 325−326.
(45) Yaalon, D. H. Weathering Reactions. *J. Chem. Educ.* **1959**, *36* (2), 73−76.
(46) Stubbs, M. F. Geochemical Problems as Student Projects. *J. Chem. Educ.* **1958**, *35* (11), 557−558.
(47) Willey, J. D.; Avery, G. B., Jr; Manock, J. J.; Skrabal, S. A.; Stehman, C. F. Chemical Analysis of Soils: An Environmental Chemistry Laboratory for Undergraduate Science Majors. *J. Chem. Educ.* **1999**, *76* (12), 1693−1694.
(48) Bopegedera, A. M. R. P. Analysis of Copper-Bearing Rocks and Minerals for Their Metal Content Using Visible Spectroscopy: A First Year Chemistry Laboratory Exploration. *J. Chem. Educ.* **2016**, *93* (9), 1616−1622.
(49) Tabbutt, F. D.; Kelly, J. J.; Cole, R. S.; Barlow, C. H.; Middendorf, D. V. Introductory Laboratory-A New Exploration. *J. Chem. Educ.* **1989**, *66* (11), 940−943.
(50) Tabbutt, F. D. Water: A Powerful Theme for an Interdisciplinary Course. *J. Chem. Educ.* **2000**, *77* (12), 1594−1601.
(51) Bopegedera, A. M. R. P. The Art and Science of Light: An Interdisciplinary Teaching and Learning Experience. *J. Chem. Educ.* **2005**, *82* (1), 55−59.
(52) Bopegedera, A. M. R. P. An Inquiry-Based Chemistry Laboratory Promoting Student Discovery of Gas Laws. *J. Chem. Educ.* **2007**, *84* (3), 465−468.
(53) Bopegedera, A. M. R. P. A Guided-Inquiry Lab for the Analysis of the Balmer Series of the Hydrogen Atomic Spectrum. *J. Chem. Educ.* **2011**, *88* (1), 77−81.
(54) Bopegedera, A. M. R. P. Teaching Analytical Skills and Techniques at the General Chemistry Level within an Interdisciplinary Framework: The Quantitative Determination of Anion and Cation Concentrations in Dolomitic Marble. *Society of Western Analytical Professors Symposium*, 2012, Oral Presentation.
(55) Yee, G. T.; Eddleton, J. E.; Johnson, C. E. Copper Metal from Malachite circa 4000 B.C.E. *J. Chem. Educ.* **2004**, *81* (12), 1777−1779.
(56) Bopegedera, A. M. R. P.; Coughenour, C. L. Investigating Geologically Important Samples in the General Chemistry Laboratory: Analysis of Alkaline Lake Waters for the Quantitative Determination of Alkalinity Dissolved Solids, Calcium and Magnesium Ion Contents. *Biennial Conference in Chemical Education*, 2012, Oral Presentation.
(57) Rioux, F.; Foster, J. C. *Paper Chromatography of Selected Transition-Metal Cations*; Chemical Education Resources, Inc., 1989.
(58) Bopegedera, A. M. R. P. Putting the Laboratory at the Center of Teaching Chemistry. *J. Chem. Educ.* **2011**, *88* (4), 443−448.
(59) Jones, M. M. Simple practical lab test for freshman students. *J. Chem. Educ.* **1977**, *54* (3), 178−179.

DOI: 10.1021/acs.jchemed.5b00776
J. Chem. Educ. XXXX, XXX, XXX−XXX

(60) Zotero Home Page. https://www.zotero.org/ (accessed Oct 2016).

(61) Verbeek, A. A.; Mitchell, M. C.; Ure, A. M. The Analysis of Small Samples of Rock and Soil by Atomic Absorption and Emission Spectrometry after a Lithium Metaborate Fusion/nitric Acid Dissolution Procedure. *Anal. Chim. Acta* **1982**, *135* (2), 215−228.

(62) Boar, P. L.; Ingram, L. K. The Comprehensive Analysis of Coal Ash and Silicate Rocks by Atomic-Absorption Spectrophotometry by a Fusion Technique. *Analyst* **1970**, *95*, 124−130.

(63) Delijska, A.; Blazheva, T.; Petkova, L.; Dimov, L. Fusion with Lithium Borate as Sample Preparation for ICP and AAS Analysis. *Fresenius' Z. Anal. Chem.* **1988**, *332* (4), 362−365.

(64) Ingamells, C. O. Lithium Metaborate Flux in Silicate Analysis. *Anal. Chim. Acta* **1970**, *52* (2), 323−334.

(65) Meyer, M. L.; Bloom, P. R. Lithium Metaborate Fusion for Silicon, Calcium, Magnesium, and Potassium Analysis of Wild Rice. *Plant Soil* **1993**, *153* (2), 281−285.

(66) Salles, L. C.; Curtius, A. J. Atomic Absorption Determination of Minor Elements in Rocks and Soils from Lithium Metaborate Flux-Fusion Dissolution. *Microchim. Acta* **1983**, *80* (1−2), 125−130.

(67) Van Loon, J. C.; Parissis, C. M. Scheme of Silicate Analysis Based on the Lithium Metaborate Fusion Followed by Atomic-Absorption Spectrophotometry. *Analyst* **1969**, *94* (1125), 1057−1062.

(68) Aruscavage, P. J.; Crock, J. G. Atomic Absorption Methods. In *Methods of Geochemical Analysis*; Baedecker, P. A., Ed.; U.S. Geological Survey: Denver, CO, pp C1−C8. http://pubs.usgs.gov/bul/1770/report.pdf (accessed Oct 2016).

(69) Cremer, M.; Schlocker, J. Lithium Borate Decomposition of Rocks, Minerals, and Ores. *Am. Mineral.* **1976**, *61*, 318−321.

(70) Fulford, G. D.; Chinloy, D. R.; Cousineau, P. G.; Ostap, S. Process for Producing Alumina from Bauxite. US Patent 4994244, February 19, 1991.

(71) Horowitz, A. J. Major Element Analysis of Rocks and Sediments by Atomic Absorption Spectroscopy. In *Open File Report 84-607*, Peck, D. L., Ed.; U.S. Geological Survey: Doraville, GA, 1984; pp 1−15. https://pubs.er.usgs.gov/publication/ofr84607 (accessed Oct 2016).

(72) Author Guidelines. *J. Chem. Educ.*http://pubs.acs.org/paragonplus/submission/jceda8/jceda8_authguide.pdf (accessed Oct 2016).

(73) Limonite samples were purchased from Ward's Science, https://www.wardsci.com/store/ (accessed Oct 2016).

(74) We used a Perkin-Elmer model 305 B atomic absorption spectrometer. Other comparable instruments could be used.

(75) Moss, M. L.; Mellon, M. G. Colorimetric Determination of Iron with 2,2′-Bipyridyl and with 2, 2′, 2″-Terpyridyl. *Ind. Eng. Chem., Anal. Ed.* **1942**, *14* (11), 862−865.

(76) We used Hewlett-Packard model 8452A and Agilent model 8453 diode array spectrometers. Other comparable instruments could be used.

(77) Suhr, N. H.; Ingamells, C. O. Solution Technique for the Analysis of Silicates. *Anal. Chem.* **1966**, *38* (6), 730−734.

(78) Ingamells, C. O. Absorptiometric Methods in Rapid Silicate Analysis. *Anal. Chem.* **1966**, *38* (9), 1228−1234.

(79) We purchased CsCl from VAr-Lac-Oid Chemical Co. Inc. (Bergenfiled, NJ, 07621); H_3BO_3 from Aldrich Chemicals (http://www.sigmaaldrich.com/); lithium metaborate, lithium tetraborate, and $(NH_4)_2Fe(SO_4)_2\cdot 6H_2O$ from Alfa Aesar (https://www.alfa.com/); iron(III) oxide and metals grade nitric acid and hydrochloric acid for VWR International (https://us.vwr.com/); the iron standard stock solution from High Purity Standards (http://highpuritystandards.com/); and the QC iron standard for the AA spectrometer from Inorganic Ventures (https://www.inorganicventures.com/). Goethite was purchased from Colorado Prospector (http://www.coloradoprospector.com/) (all accessed Oct 2016).

(80) Bonewitz, R.; Carruthers, M. W.; Efthim, R. *Rock and Gem*; DK Publishing: New York, 2008; p 169.

DOI: 10.1021/acs.jchemed.5b00776
J. Chem. Educ. XXXX, XXX, XXX−XXX