

Seeing the “Unseeable,” A Student-Led Activity to Identify Metals in Drinking Water

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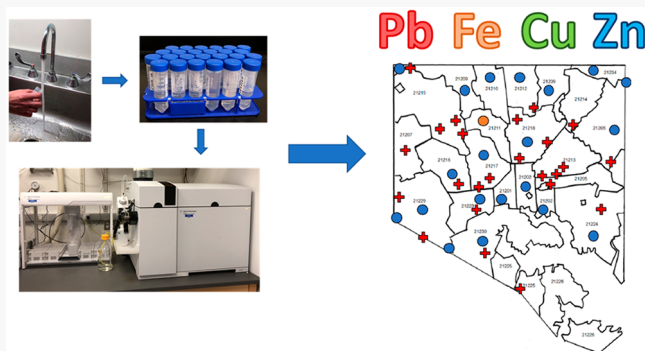
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Supporting Information

ABSTRACT: Municipal drinking water, regulated by the Environmental Protection Agency via the Safe Drinking Water act, has long been assumed to be contaminant-free. However, crises related to drinking water have emerged, most notably the “Flint Water Crisis” in Flint, MI, where high levels of lead (Pb) were detected in the area’s water. Much of the water-sampling data collected in Flint was obtained by “Citizen Scientists” working closely with a team of researchers at Virginia Tech, who used the analytical technique of Inductively Coupled Plasma Mass Spectrometry (ICP-MS) to quantify metal ions present in the water. Inspired by these efforts, we developed adaptable public water testing outreach efforts, led by students in Baltimore city (Middle School, High School, and College), to test the city’s drinking water. These “student-scientists” read news and scientific articles to understand the public health impact of lead in drinking water and the analytical approaches scientists use to detect metal ions in water. The students then developed a written “water collection protocol” and sought participation from colleagues (other students, faculty, and staff) who collected their home drinking water to be tested. The student scientists prepared and analyzed samples for lead (Pb) as well as copper (Cu), iron (Fe), and zinc (Zn) metal ions commonly found in drinking water, to be tested via ICP-MS. Data were then plotted onto a map of Baltimore City, with the metal levels indicated for each Zip code. This outreach event connects science to real-life news events while teaching analytical methodology and can be tailored to students at various stages of their education.

KEYWORDS: Mass Spectrometry, Public Outreach, Hands-On Learning, Elementary/Middle School Science, High School/Introductory Chemistry, First-Year Undergrad/General, Second-Year Undergrad, Upper-Division Undergrad, Analytical Chemistry, Environmental Chemistry, Inorganic Chemistry



INTRODUCTION

In the United States, drinking water is regulated by the Environmental Protection Agency (EPA) under the auspice of the Safe Drinking Water Act of 1974, and the general public considers it safe for consumption.^{1,2} In 2004, the safety of municipal drinking water in Flint, Michigan, came into question after high levels of lead were detected.^{3,4} In the ensuing “Flint Water Crisis,” citizen scientists organized by a team of researchers at Virginia Tech were key contributors to the discovery of high levels of lead in the drinking water.^{3–6} These citizen scientists collected water from their homes in Flint, which was then tested for lead content at Virginia Tech. The work of these citizen scientists contributed to policy changes at the federal, state, and city levels that included changing the source and treatment of drinking water in Flint to ensure its safety. Creating engaged, scientifically literate citizens is a broad goal for science education; inspired by the work of citizen scientists, we sought to pilot an outreach program to engage students at a variety of levels on key skills in citizen science, such as sample collection, and on developing a

knowledge base of analytical chemistry, the kind of work that helps students understand why water would be declared safe or unsafe.

The methodology utilized to quantify the metal ions present in tap water collected by the Flint citizen scientists was inductively coupled plasma mass spectrometry (ICP-MS). ICP-MS instruments enable quantitative analysis of most elements in the periodic table and are sensitive to the parts per trillion (ppt) level. In contrast, a more conventional chemical analysis of water, such as using a colorimetric reagent, will detect contaminants in the parts per billion (ppb) range, an order of magnitude less sensitive than ICP-MS. Indeed, a 1994 experiment on detecting chromium contamination in water

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Table 1. Student Activities and Learning Outcomes

cohort	students	broad learning objectives	cohort-specific learning outcomes, demonstrated by participants
Spring into Maryland Science	Undergraduate students	Role of Citizen Scientist	How to prepare a series of calibration samples for ICP-MS
		Analytical Sample Preparation (ICP-MS)	How to construct a calibration curve for ICP-MS
six-week internship	Senior high school students, undergraduate biology or chemistry majors	(as above but with)	(as above but with)
		Develop a Laboratory Activity Geared toward Middle School Students That Includes Citizen Scientist, Analytical Chemistry and Social Justice Components,	PubMed and Google Scholar literature search
		Develop and Carry Out Activity to Obtain Water Samples from Departmental Colleagues	Water sampling protocol development
			Sample inventory management
			Assist with sample preparation for ICP-MS
			Development of Laboratory Activity
UMB CURE Scholars Program	Middle school students	Role of Citizen Scientist	How to prepare a series of calibration samples for ICP-MS
		analytical sample preparation	How to construct a calibration curve for ICP-MS
		analytical sample analysis (ICP-MS)	

noted that the colorimetric reagent approach should only be used on highly polluted water, such that an educator running the experiment would have to pretreat samples with sufficient chromium to get a positive result.⁷ In contrast to those methods more common in classroom laboratories, ICP-MS is routinely deployed in real-world applications that involve human health, as it is considered a reliable method for sensitive quantification. Baltimore City, the site of our activity, has a history of lead poisoning, which had gained prominence in the national media in 2015 with the death of Freddie Gray, who had elevated lead levels.^{8,9} In Baltimore, elevated lead levels are usually attributed to lead paint, and the data suggest that overall lead poisoning in the state of Maryland has been declining.¹⁰ Nonetheless, lead contamination remains an important public health issue in Baltimore, as is the investigation of environmental sources of lead. The water supply in Baltimore has not typically been implicated as a source of lead toxicity, although in older communities, water is often delivered via lead service lines, which can serve as a source of lead contamination.¹¹ Overall, the broader question of environmental toxins and how they are detected is important to a larger conversation on science's role in broader questions of health.

ICP-MS is more sensitive by an order of magnitude than reagent tests for water contamination and requires a large investment. However, the basic science of ICP-MS is accessible to chemistry students at different levels; basically, ICP-MS depends upon detecting charged particles. Samples are aerosolized into an argon plasma stream, which dries the droplet and dissociates the elements of the sample into charged ions. A mass filtering device, known as a mass spectrometer, then filters the ions, allowing ions with only a fixed mass-to-charge ratio to pass to the detector. Software analyzes the signal against standards to determine the final concentration of each element that is detected.^{12–17}

ICP-MS instruments are regularly becoming part of chemistry and biology departments' analytical service centers in universities, making them an increasingly accessible

analytical teaching tool. We have access to an ICP-MS at the University of Maryland School of Pharmacy, and, inspired by the efforts of citizen scientists to test drinking water in Flint, MI,^{5,18} we deployed ICP-MS and "student scientists" to test drinking water in Baltimore City. Our goal was to translate the science from the headlines from Flint to hands-on practical laboratory experiences for students—to prepare them to engage with the discourse of science whether they choose to later specialize in the sciences or not. Our approach was informed by Alan Irwin's sense of the citizen-scientist.¹⁹ Irwin noted the social importance of environmental issues and the barriers between scientific discourse and the public; Irwin's construct of citizen-scientist invokes political self-hood in its pointed use of "citizen," as opposed to something more neutral, such as "lay-person", to suggest a directed, social focus in the action of citizen-scientists. Thus, water, a basic need for life that is regulated by government, is an important area for investigation, as the role of science education scales beyond schools to the community at large.²⁰ This sense that scientific education might be critically important beyond schooling indicates the importance of being able to adjust scientific education to different backgrounds and contexts outside of traditional educational projects.

■ MATERIALS AND METHODS

Water Sample Collection

Water samples (~25 mL) were collected in 50 mL conical tubes from participants' kitchen faucets first thing in the morning to ensure that no water had been run from the faucet for at least 6 h prior to collection. We specified a 6 h stagnation time to be consistent with EPA guidelines on sampling tap water for lead (see https://www.epa.gov/sites/production/files/2016-02/documents/epa_lcr_sampling_memo_dated_february_29_2016_508.pdf). Participants were also instructed to label the collection tubes with their initials and Zip code.

Sample Preparation and ICP-MS Analysis

The collected water sample (2 mL) was transferred to a 15 mL metal-free conical tube (VWR) and acidified by adding concentrated nitric acid (Trace-Metal grade, Fisher Scientific, 200 μ L) in a fume hood. The concentration of metals in the participants' acidified samples was determined by direct infusion into a 7700x ICP-MS (Agilent Technologies). Metal concentrations in the samples were derived from a calibration curve generated by a series of dilutions of atomic absorption standard (Fluka Analytical, Honeywell International Inc.) prepared in 6% nitric acid. Data analysis was performed using Agilent's Mass Hunter software (4.4 version).

HAZARDS

All instrumentation used (ICP-MS and autosampler) during the analysis of the participants' drinking water was commercially available and run using manufactory guidelines. The argon dewar utilized for the operation of the ICP-MS was set to company specifications and housed in a different location from where the activity was being conducted. To minimize the participants' exposure to nitric acid, the participants measured their water samples and then supplied the sample to the outreach event leader, who then acidified the sample in a chemical fume hood. The event leader was outfitted with appropriate personal safety equipment when handling concentrated acid. The acidified samples were then placed by the event leader in the autosampler, which has an integrated environmental enclosure to ensure the participants did not encounter any acidified solutions.

ACTIVITY

A 2015 editorial in *Nature* notes the increasing contributions that nonscientists can make to research, especially in data gathering.²¹ However, the accuracy of data gathered by citizen scientists can be an issue; in fact, a citizen-scientist initiative to monitor agricultural runoff in water resources reported erroneous measures, based on colorimetric water tests.²² Our activity is meant to provide a conceptual background along with sampling, with analysis conducted by scientific researchers using a state-of-the-art analytical tool (ICP-MS). This activity is designed to be adapted for a wide variety of participants and will teach participants why sampling and data collection must be precise (Table 1).

For our activity, our first student scientists were participants in an annual outreach event called Spring into Maryland Science (SIMSI), hosted at the University of Maryland Baltimore. These student scientists were female undergraduate students from Notre Dame of Maryland University, a women's college in Baltimore City, with five to six participants per year (see the Supporting Information for student demographics). SIMSI is an annual event in which Notre Dame women participate in mini-laboratory experiences to get a first-hand view of what research science involves. The mini-laboratory experiences consist of 2 h "rotations" in three different research laboratories in the Pharmaceutical Sciences Department at the University of Maryland School of Pharmacy. In these experiences, the Notre Dame women are paired with graduate students who have identified a short experiment for the Notre Dame women to run, which is presented in the context of the graduate student's overall thesis project. The lead testing activity was integrated into the SIMSI students' day—following a morning of laboratory experiences the SIMSI

students spent the afternoon analyzing their water samples for metals via ICP-MS. In preparation for the visit and water analysis, students read articles linked from the Flint Water Study Web site (<http://flintwaterstudy.org>), including the directions on how to sample their water (<http://flintwaterstudy.org/information-for-flint-residents/video-water-sampling-kits-for-lead/>). The students were provided with metal-free conical tubes to collect their tap water and were given an instruction sheet (Supporting Information, *SIMSI Student Water Testing Directions*), adapted from the Flint study, that described the water collection technique.

On the day of the SIMSI event, students brought the water samples to campus and during the afternoon worked with the Michel laboratory to acidify the samples and analyze via ICP-MS (for experimental details please see the *ICP-MS Water Testing Worksheet* in Supporting Information). The SIMSI students learned how to make calibration standards for each metal of interest (four metals were tested to keep within the time frame of the activity, and typically one student of the group prepared the calibration samples) and how to determine the lower limit of quantification (LLOQ) for each sample. The latter is achieved by comparing the counts of the metal of interest in the calibration sample at a given concentration versus the counts of the metal of interest in the blank after running the calibration curve and then using a cutoff value of greater than or equal to 5 times the blank to determine the LLOQ. Because the ICP-MS software uses the calibration curve to determine the concentrations of the metal of interest, students were also given "mock" data (which is included in the worksheet used for middle and high school students (see the Supporting Information)), so that they could understand how the calibration curve is used to determine metal concentrations. They were also shown the calibration curve generated by the ICP-MS software. The data obtained from the ICP-MS sample typically contains values with multiple digits (15 or higher); therefore, this also provided the opportunity to discuss significant figures. The SIMSI students were asked to consider which step in the process was the limiting step for significant figures, identified pipetting as the limiting step, and determined that three significant figures would be the correct number of figures to express their data.

Once the students had determined the metal content of their individual water samples, they mapped the metal concentrations onto a master calendar that color coded the concentrations (a replica of which shown in Figure 2). The students then participated in a round-table discussion with the Michel lab on the Flint water crisis. The group discussed how metals can be toxic and the method of measuring metals by ICP-MS, and the role of the EPA in determining toxic levels. The students had been asked to read the EPA Web site on drinking water levels (<https://www.epa.gov/ground-water-and-drinking-water/national-primary-drinking-water-regulations>) prior to SIMSI and had also been encouraged to search for news articles on the EPA limits to promote discussion as to how these limits are derived and the competing interests that factor into these decisions. Students discussed whether they thought that the approaches used by the EPA to set the limitations on metals in drinking water are ideal and what they had learned about doing searches regarding how the limits were determined. We note that, while this manuscript was under revision, the EPA proposed a new update to the "lead and copper rule" for drinking water with a 10 ppb "trigger level", and we plan to include this new

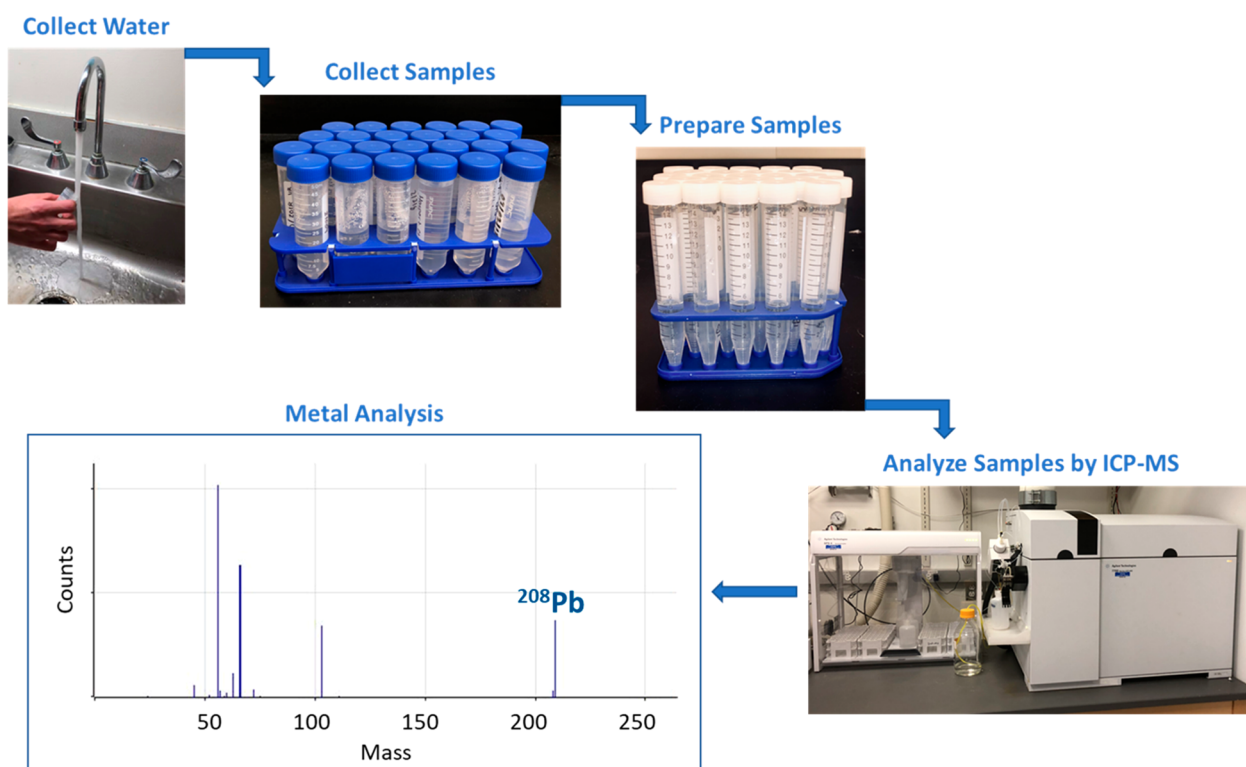


Figure 1. Water Collection, sample preparation, and metal analysis.

update and discussion in future water-testing activities.^{23,24} The discussion also focused on the topic of lead and poor communities. Discussion centered on both the Flint, MI residents and the problem of lead poisoning (from lead paint) in Baltimore City, which disproportionately affects poor residents, and also expanded to the Freddie Gray case, as Freddie Gray had experienced lead poisoning as a child.⁹ The SIMSI activity was then adapted for middle school students. The middle school activity was led by high school and undergraduate students who were participating in a summer internship program at UMB in the Michel laboratory and thus involved students at varied stages of education.

The middle school activity was developed as follows. Two summer interns: a student who had just completed her junior year of high school and a student who had just completed her freshman year of college, with the guidance of the Primary Investigator (PI) (Michel) and Michel lab trainees, modified the SIMSI activity to be appropriate for the educational level of middle school students, and the activity was performed by middle school students who are members of the UMB CURE Scholars program.²⁵ In synthesizing and constructing their own protocols, student-scientists take an active approach to considering the rationale for what they will do, rather than passively follow a prespecified procedure, as the agricultural contamination project, referenced above, had used. The laboratory activity took half a day and included prelaboratory activities that were completed in advance of the laboratory activity. All activities are described in a worksheet that the students received (see the *ICP-MS Water Testing Worksheet* in [Supporting Information](#)). As shown in the worksheet, the prelaboratory activities are set up to (1) inform the students about the Flint Water crisis, with a focus on how the Flint water became contaminated with lead and the difficulties associated with measuring and remediating lead, (2) teach

concepts of solubility and metal toxicity, and (3) provide background on ICP-MS. In addition, as in the SIMSI activity, the middle school students were provided with metal-free conical tubes and instructions as to how to collect water samples, prior to their laboratory experience. The students brought their worksheets and their water to UMB for the half-day laboratory activity.

The activity was led by the summer interns, with the PI (Michel) and her trainees present to offer guidance. Prior to the event, the summer interns worked one-on-one with the PI and her trainees to learn how to run the activity. The interns put together a PowerPoint presentation that described the activity and included details about the analytical approach and safety considerations. The presentation was critiqued by the PI and her trainees and practiced by the interns. The interns then performed the activity on their own, so that they understood the nuances and could troubleshoot. The interns were then ready to run the event. To start, the interns went over the prelaboratory activities using their PowerPoint presentation; the interns then led an activity focused on generating a calibration curve, helped the students prepare their samples for ICP-MS, and assisted in obtaining the ICP-MS data and working up the data. The data were then all plotted onto a map of Baltimore City. The activity ended with a discussion of the findings in view of EPA regulations and metals, lead and poor communities, and lead in Baltimore City (sample discussion topics are provided in the *ICP-MS Water Testing Worksheet* in [Supporting Information](#)). Although none of the water samples contained elevated lead, elevated levels of lead are reported in some children in Baltimore City. This is typically attributed to lead paint, and to illustrate this further, we added red crosses to our map to indicate Zip codes in Baltimore where blood lead levels of greater than 10 $\mu\text{g}/\text{dL}$ from 2014 have been reported.²⁶ Some of these Zip codes matched Zip codes we

had obtained ICP-MS data for water, though that water did not show elevated lead. This allowed for discussion of how scientists must obtain specific data to link the effect (e.g., higher lead in blood) to the cause (i.e., lead paint vs water).

The summer intern students also collected and assayed water for departmental colleagues to obtain additional data for the map of Baltimore City. This was achieved by contacting graduate students, postdoctoral fellows, faculty, and staff, as well as friends and family to collect their home drinking water following the written protocol. The typical sample workflow is shown in Figure 1. The student interns managed sample inventory and helped with sample preparation and sample analysis by ICP-MS. Once the samples were analyzed for their metal content, the interns worked up the data and conveyed the data back to the volunteers. The metal concentrations of the volunteers' water samples were then plotted on a poster-sized map of Baltimore city with colored dots. This allowed the whole department to see the results of the water study and bring awareness to metal concentrations in the local drinking water. The data are by no means a systematic study of drinking water in Baltimore as a function of Zip code, as the samples were dictated by participants, and in some instances multiple measures were made in one Zip code and in others only one measure; however, analyzing the relationship between metal content and Zip code is a potential next step for this project.

RESULTS

All of the variations of water analysis activities allowed for customization depending on the targeted audience. In total 136 samples were analyzed over four years. The results of these samples for Baltimore City are presented in Table 2 and then mapped by Zip code in Figure 2. Data on the water supply infrastructure of Baltimore was not available to include on the map; however, we were able to obtain information on blood lead levels correlated to Zip codes on our map.

All averages found for the Baltimore City drinking water were below the EPA's action level.

CONCLUSIONS

Drinking water, which is essential to life, can also be subject to contamination by elevated levels of metals that can be toxic, as was evident in the Flint water crisis.⁴ Although these metals can have a severe impact on public health, the reliable testing of water might seem inaccessible to the general public, especially if that testing uses the state-of-the-art tools of analytical chemistry, such as ICP-MS. Our activity, which we scaled to learners in middle school, high school, and college, showed students that their environment and health could be objects for their own scientific inquiry.

Handouts were prepared and supplied to the participants. For the younger participant groups, our senior participants (high school and college interns) wrote a water collection worksheet (see the *ICP-MS Water Testing Worksheet* in the [Supporting Information](#)), guided by the PI and her trainees, that included a prelaboratory assignment with reading on the Flint water crisis and an overview on solubility and toxicity, an experimental workflow that included concepts of sample preparation, calibration curve generation, and data analysis, and postlaboratory discussion section that linked the data obtained by the students to the real life issues related to metal contamination in drinking water.

Table 2. Results from Water Analysis

Zip code	number of samples	[Pb] ^a	[Cu] ^a	[Fe] ^a	[Zn] ^a
17331	2	0.10	40.0	3.61	437
20817	1	0.04	26.5	1.28	23.8
20851	1	0.03	176	3.20	16.2
20904	1	0.06	31.9	2.24	99.7
21009	2	0.03	63.1	1.89	151
21042	2	1.72	214	1.03	192
21043	4	0.02	52.6	2.08	97.5
21061	2	ND	10.3	0.66	76.9
21073	1	ND	12.0	5.31	4.34
21075	2	ND	85.8	3.51	37.3
21085	1	ND	176	2.79	26.8
21093	3	4.49	204	10.2	238
21131	1	1.25	39.4	ND	200
21201	19	0.62	274	9.93	289
21202	3	0.30	8.06	43.2	23.1
21204	2	0.79	177	2.40	57.8
21206	2	0.38	116	4.06	111
21208	2	ND	19.2	5.76	0.00
21209	2	0.87	44.6	16.5	227
21210	12	2.56	392	40.5	278
21211	1	8.16	37.1	7.53	34.1
21212	2	1.36	111	50.1	149
21216	4	0.63	58.7	23.5	140
21217	1	0.31	17.7	8.31	74.2
21218	17	1.06	124	3.84	219
21223	2	0.23	13.3	20.2	220
21224	2	0.51	3.19	10.1	69.2
21227	3	0.22	10.2	4.70	83.0
21228	4	ND	29.0	17.0	35.7
21229	6	0.33	16.9	29.2	190
21230	8	1.26	33.2	9.65	236
21231	1	0.03	0.26	0.22	15.4
21234	1	ND	110	6.67	2.47
21236	1	0.13	116	4.14	11.5
21239	2	0.33	33.6	2.11	221
21244	2	ND	65.2	0.77	10.8
21286	1	2.68	34.4	15.9	955
21401	1	ND	19.5	17.1	8.94
21702	1	0.54	52.4	15.5	974

^aMetal concentrations (in ppb) reported are the average of all samples from the Zip code. ND indicates not determined.

As we performed the activity with students at widely different educational levels, we adapted the material to balance scientific rigor with each student's background. A key scientific concept that we focused on was the "properties of metal ions." Here, all groups were introduced to the periodic table and the concepts of metal ions, which can be beneficial or toxic depending on concentrations, with illustrations from the news (e.g., the Flint water crisis, lead in paint in older homes); however, for students with chemistry and biology backgrounds, the roles of metals in binding to proteins and promoting key biological processes were also discussed.

We also included the concept of solubility and linked this concept to ICP-MS and data analysis (see the *ICP-MS Water Testing Worksheet* in the [Supporting Information](#)). The basic concepts of mass spectrometry and ICP-MS were taught to all levels of students. Students were able to observe the ICP-MS in action, see the plasma generated, and analyze the data output. A key concept for ICP-MS is calibration. Student

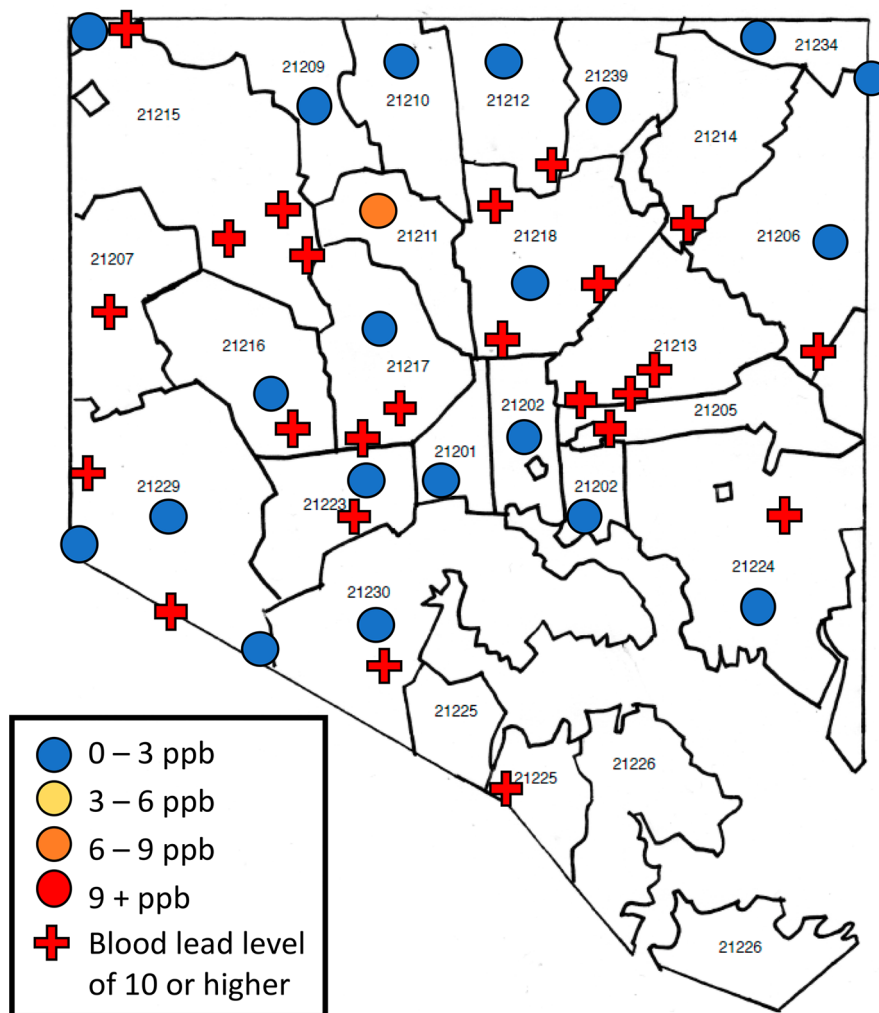


Figure 2. Lead analysis results by Zip code. Blood lead level reflects cases of a blood lead level of greater than 10 $\mu\text{g}/\text{dL}$ from 2014, as initially reported in the *Baltimore Sun*.

scientists learned how to plot a calibration curve for each metal tested and fit their data on the curve to identify the metal content. In addition, the sensitivity of the instrument was described via instruction on LLOQ determination. Other public activities with ICP-MS have sometimes shied away from calibration because of the additional time needed and the complexity.¹ Because our activity was at its shortest a half-day in length and was scaled upward to a six-week lab internship, we could also adjust the level of involvement of participants; our student interns also analyzed the data and reported it back to collaborators as well as performed other research experiments under the School of Pharmacy faculty mentors (not related to this activity).

In addition to these scientific concepts, we contextualized the overall project in the social reality of Baltimore, where students of poor socioeconomic standing have historically been exposed to lead, thought to occur from lead paint exposure.^{8–10,26} In the case of the Flint, Michigan, water crisis, students were interested to learn that, to some community members, the government did not act quickly enough to confirm lead levels in the water and that citizen scientists using ICP-MS were important in confirming that the lead levels were dangerously high, drawing national attention to the crisis.

A limitation of our activity was the laboratory setting. While we provided deep engagement, space constraints limited the number of participants at any given time. We overcame this by having the students come in teams of five to seven participants to have their samples analyzed. The rest of the groups were working on preparing their samples or working on the postanalysis worksheet. One way to increase involvement would be to live stream the ICP-MS analysis and/or provide online content to simulate the ICP-MS measure. We were also limited in our interpretation by the lack of data on the water supply of Baltimore. Also, Baltimore is a city divided into many neighborhoods, with highly unequal socioeconomic areas sometimes in close proximity. Thus, while the Zip code will neither present our data in the context of clear spatial boundaries of socioeconomic zones in Baltimore nor track along the water infrastructure, our intention is to enable future work along those lines by reporting water contamination levels in smaller, more detailed geographic zones than just at the city level. Perhaps other researchers can uncover causal relationships that explain the varying water quality we report.

Overall, ~30 students participated in this project to collect and analyze residential drinking water from student volunteers in the Baltimore, Maryland, region. Students were highly interested in exploring an issue of importance, safe water, with

specialized technology. Students were eager to take home the maps that were created to share with their friends and family.

■ ASSOCIATED CONTENT

SI Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.9b00553>.

ICP-MS Parameters and Notre Dame Student Demographics (PDF)

SIMSI Student Water Testing Directions (PDF)

ICP-MS Water Testing Worksheet (PDF)

ICP-MS Water Testing Worksheet Answer Key (PDF)

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Notes

The authors declare no competing financial interest.

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