CHEM 110 / 154 Online Experiment: Arsenic Toxic Levels

Introduction

Long-term exposure to arsenic at even low-levels is toxic to humans. Arsenic has been linked to a number of cancers. These include cancer of the bladder, lungs, skin, kidney, nasal passages, liver, and prostate. Common non-cancerous symptoms of chronic low-level exposure to arsenic include thickening and discoloration of the skin, headache, nausea, vomiting, diarrhea, abdominal pain, numbness in hands and feet, seizures, partial paralysis, blindness, and mental status changes.

Arsenic is naturally present at very low levels in almost all sources of groundwater, primarily due to water flowing over geological deposits rich in arsenic-containing minerals. The World Health Organization and Environmental Protection Agency have both set the Maximum Contaminant Level (MCL) for arsenic at **0.01 mg / L**. This is deemed to be the maximum level of a contaminant in drinking water at which no known or anticipated adverse effect on the health of persons would occur, and which allows an adequate margin of safety. Unfortunately, the groundwater present in some parts of the world, such as Bangladesh, is well above the MCL. A study conducted in 2017 showed that upwards of 57 million people in Bangladesh obtain their water from shallow wells testing at 0.05 mg As / L or higher. As a result, as many as 43 thousand deaths each year have been linked to arsenic-poisoning in Bangladesh. Deeper wells (> 150 m) and filtration-facilities that can supply an entire community are being built in the most heavily poisoned regions. In the most heavily poisoned regions.

Arsenic can also be present in groundwater due to human activity. One common source used to be the copper-mining industry, where arsenic and other toxic elements found in copper-containing ores were accidently released into the environment. The proper use of tailing-ponds has mostly eliminated this problem, as long as the structural integrity of the pond is not compromised. A local example of accidental release is the failure in August 2014 of the tailing-pond dam at the Mount Polley copper, silver, and gold mine, located in the Cariboo region of central British Columbia. Within a span of a few days, the entire 4 square-kilometre tailing-pond had emptied into local creeks, lakes, and the Cariboo river. Elevated levels of arsenic, selenium, and other metals were detected at various test sites, but fortunately, local water sources have since recovered.ⁱⁱⁱ

Experiment Overview

There has been an accidental release of arsenic from a mine. Samples of river water have been collected at four testing-sites located downstream from the mine. Your tasks are:

- (1) Find a method to quantitatively analyze for arsenic in water.
- (2) Determine the arsenic level, in units of mg / L, at the four sites. Graph this data.
- (3) Assuming that at least some of the samples will analyze above the MCL of 0.01 mg As / L, determine what the minimum safe-distance is from the mine.
- (4) Predict the arsenic levels at two properties located at known distances from the mine.

Experimental Method

Arsenic commonly dissolves in water as the arsenate ion, AsO₄³-. Arsenic is in the 5+ oxidation-state when in this form, and is also known as As(V).

Aqueous silver nitrate, AgNO₃, reacts with aqueous AsO₄³⁻ to form solid silver arsenate, Ag₃AsO₄(s), as per equation 1. The nitrate spectator ion, NO₃-, has been omitted.

$$AsO_4^{3-}(aq) + 3 Ag^{+}(aq) \rightarrow Ag_3AsO_4(s)$$
 Eqn 1.

If an excess of silver nitrate is added to a water sample suspected of containing arsenate, then hopefully all of the arsenic will precipitate out of the sample as solid silver arsenate. If so, then gravimetric analysis could be used to determine the amount of arsenic present in the sample.

In Part One of this online experiment, you will test to see what percent of arsenate in a sample of known concentration is precipitated out of solution by silver nitrate. If at least 98% of arsenic in the known sample is converted to Ag₃AsO₄, then you can be confident in using gravimetric analysis with silver nitrate to analyze water samples of unknown arsenic concentration.

In Part Two of this experiment you will determine the amount of arsenic present in water-samples obtained from four test sites. This assumes that you will find the proposed testing-method to be reliable. One site is located directly at the mine, but the other three sites will be at distances downstream from the mine that are unique for each student performing this experiment. For example, 2.5 km, 7 km, and 14 km downstream of the mine. Students will need to use MS-Excel or Google Sheets to create a graph plotting arsenic level versus distance from the mine. A sample graph is shown below, along with the trendline and equation of the line-of-best-fit. The equation will be used for calculations discussed later on.

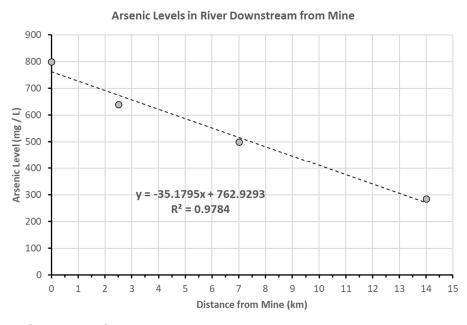


Figure 1. Plot of Distance from Mine versus Arsenic Level. (false data; does not match the lab)

Experimental Procedure

This is detailed on the website.

- 1. Watch the 6-minute video demonstrating the Virtual Lab software for this experiment
- 2. Access the Virtual Lab website^{iv} and perform the experiment to obtain the mass of Ag₃AsO₄(s) precipitated in three samples:
 - 0.01 M Na₃AsO₄(aq)
- Sample #1 (A)
- Sample #2 (C)
- 3. Obtain the mass of two more samples (B and D) from simple calculations.
- 4. Calculate the arsenic levels for each sample.
- 5. Use MS-Excel or Google Sheets to prepare a graph of Distance versus Arsenic Level.

Video guidance (15 - 20 minutes long) is provided for graphing and creating a trendline. Choose from either the MS-Excel or Google Sheets presentations.

Part One Procedure

- 1. Open the Virtual Lab website: http://chemcollective.org/activities/vlab/75
- 2. From the Stockroom Solutions tab, obtain the Erlenmeyer flasks of 1 M AgNO₃ (silver nitrate) and 0.01 M Na₃AsO₄ (sodium arsenate). Each flask contains 100 mL of solution. These are the precipitating agent and standard analyte, respectively.
- 3. Tip the flask of AgNO₃ over the mouth of the flask of Na₃AsO₄ and pour exactly 1.00 mL of the silver solution into the arsenate solution. *Use "Precise" mode, not Sig Fig or Realistic.*
- 4. Look at the Information panel, located to the left of the Workbench. Record the mass of Ag₃AsO₄(s) present onto the second column of the data table on your report sheet. *If the Information panel is not visible, click on the flask containing arsenate*.
- 5. Continue adding the precipitating agent (silver nitrate) to the analyte (sodium arsenate) in 1.00 mL increments until the mass of Ag₃AsO₄(s) reaches a maximum. That is, the mass remains constant upon addition of more silver nitrate. Record the mass of precipitate present after each addition of 1 mL of silver nitrate.
- 6. When finished, right-click on the flask of arsenate and select "Remove".

Part Two Procedure

- 1. In a manner similar to Part One, bring the Sample 1 flask onto the virtual workbench and add to it 1 M AgNO₃ in 1 mL increments. Record your masses in the third column of the report sheet. This is sample "**A**" for graphing purposes.
- Repeat with the Sample 2 flask and record masses in the last column. This is sample "C" for graphing purposes. Samples B and D are not located in the Virtual Lab software.

Treatment of Results

CALCULATIONS: Part One

- 1. Calculate the number of **moles of Ag₃AsO₄ precipitated** in the flask of Na₃AsO₄. Use the maximum precipitate mass obtained. Molar mass of silver arsenate is 462.52 g/mol.
- 2. Calculate the number of **moles of arsenate (AsO₄³-) initially present** in the flask of 0.01 M Na₃AsO₄. Recall that you started with 100.00 mL of solution.
- 3. Calculate the **percent of AsO**₄³⁻ **that was precipitated** in the Na₃AsO₄ flask as solid Ag₃AsO₄. As one mole of arsenate is present in each of these compounds, simply divide moles AsO₄³⁻ initially present by moles Ag₃AsO₄ obtained and then multiply by 100.

CALCULATIONS: Part Two

Masses of Ag₃AsO₄ for Samples B and D

Samples B and D have already been analyzed for you and their results are given to you on the website for this experiment as a percent of the values for A and C, respectively. Sample B was collected downstream of A, while D was collected downstream of C.

Consult your version of the online Raw Data and calculate the mass of Ag₃AsO₄ precipitate for Samples B and D. An example using false numbers is given below:

- Precipitate masses of 0.493881 g and 0.308675 g are measured for Samples A and C, respectively, using the VL software.
- The website lists Sample B as 80% of Sample A and Sample D as 57% of Sample C.
- Therefore: Sample B = $0.80 \times 0.493881 \text{ g} = 0.395105 \text{ g}$ Sample D = $0.57 \times 0.308675 \text{ g} = 0.175945 \text{ g}$

You now have the mass of Ag_3AsO_4 precipitate for all four water-samples. If your calculations are done correctly, the mass of precipitate should continually decrease from samples A - D.

Arsenic Level in Samples A - D.

For each sample, convert mass Ag₃AsO₄ precipitate to moles Ag₃AsO₄. Then convert to mass of arsenic (atomic mass 74.92 g/mol; 1:1 stoichiometry). Multiply by 1000 to obtain mass As in units of mg. This is the amount of arsenic present in each 100 mL sample. Multiply by 10 to convert to mg / L. Report to 0 decimal places. For example:

$$\frac{0.493881 \text{ g Ag}_3\text{AsO}_4}{462.52 \text{ g/mol Ag}_3\text{AsO}_4} \text{ x } \frac{1 \text{ mol As}}{1 \text{ mol Ag}_3\text{AsO}_4} \text{ x } \frac{74.92 \text{ g As}}{\text{mol As}} \text{ x } \frac{1000 \text{ mg}}{1 \text{ g}} \text{ x } 10 = 800 \text{ mg As / L}$$

GRAPH

Use MS-Excel or Google Sheets to prepare a plot of Distance from Mine versus Arsenic Level, similar to Figure 1. The distances of your samples A – D are obtained from your version of the online Raw Data and should have already been copied onto page 2 of the lab report sheet.

As demonstrated in the graphing videos, add a trendline to your graph and record the equation for the line-of-best-fit and the statistical R^2 value onto your report sheet. If all data-points fit the line perfectly, then $R^2 = 1$. The raw data provided to students in this online experiment typically yields R^2 values in the range of 0.97 - 0.99.

- Report R² to 4 decimal places. This will probably require first making the entire equation for the line-of-best-fit 4 decimal places as well.
- When reporting the equation for the line, report the slope to only 2 decimal places and the y-intercept to 0 decimal places. *This will simplify subsequent calculations*.

Print a full-page hardcopy of your graph for submission along with your lab report.

CALCULATIONS FROM THE GRAPH

The equation for the line-of-best-fit (y = mx + b) can be used to perform additional calculations, where: y = Arsenic Level and x = Distance from Mine.

If distance (x) is known, then arsenic level (y) can be calculated from:

$$y = mx + b$$

For example, Figure 1 shows an equation of: y = -35.18 x + 763. A sample taken 10 km downstream from the mine would be predicted to have an arsenic level of 411 mg / L.

If arsenic level (y) is known, then distance (x) can be calculated from:

$$x = \frac{(y - b)}{m}$$

For example, using the equation of Figure 1, the MCL of 0.01 mg As / L would be predicted to be attained 21.7 km downstream from the mine.

Please refer to your version of the Lab Report Sheet for any requested calculations.

¹ Toxicology information obtained from https://www.epa.gov/dwreginfo/chemical-contaminant-rules.

Bangladesh study from https://undark.org/2017/08/16/bangladesh-arsenic-poisoning-drinking-water/.

iii Mount Polley spill from https://en.wikipedia.org/wiki/Mount Polley tailings spill.

iv Virtual Lab module programmed by Dr. Jordi Cuadros, Instituto Químico de Sarriá and Dr. Dave Yaron, Carnegie Mellon University, 2005. Lab Manual and Report Sheet written by Dr. Matt Le Page, CapU, 2020.