

Tap Water Analysis by Atomic Absorption and Ion Chromatography

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Introduction

The use of tap water in our every day lives is a natural routine due to its ease of access. With the flip of a faucet handle we readily have clean water for cooking, cleaning, washing, and drinking. Since tap water plays a key role in our daily lives, knowing the ion concentrations it contains is of great importance for our health and safety. The United States Environmental Protection Agency (U.S EPA) and the California State Water Resources Control Board (State Board) created set standards for public drinking water systems to follow, and ensuring requirements are met with the Safe Drinking Water Act (SDWA). Water is broken into two categories based on origin, surface and ground. Surface water comes from lakes and rivers whereas ground water comes from aquifers and wells. In the 2016 Water Quality Report Issued by the City of Sacramento, 79% of our water is surface water and the remaining 21% is ground water. As water passes through land formations on the surface or underneath, it will encounter chloride, sulfate, magnesium, calcium, and nitrate. Concentrations of each will fluctuate due to weather changes and or pollution. The varying concentrations of salts attribute to the “hardness” of water on a scale of 0-180+ ppm with the equivalence to CaCO_3 . The water hardness ranking system is as follows: 0-60 ppm for “soft”, 61-120 ppm for “moderately hard”, 121-180 ppm for “hard”, and 181+ ppm for “very hard”. The determination of hardness for this study will be from a tap water sample taken in Sacramento, Ca (38.5642 degrees N, -121.3807 degrees W). These contaminants are expected to be in our waters do to natural mineral deposits being dissolved and aren't of main concern. The use of fertilizer can increase the concentrations of nitrate and can cause health issues to arise when concentrations are over 10 ppb according to 2016 Water Quality Report. According to Pesticide Safety Education Program (PSEP), infants are susceptible to Methemoglobinemia when nitrate concentrations are

increased in drinking water. This occurs in infants because they lack the enzyme that converts methemoglobinemia back to hemoglobin, which is responsible for carrying oxygen in the blood.

Methods

I. Atomic Absorption Spectroscopy (AAS)

A liquid sample, as seen in figure 1, containing dissolved metals gets sucked up and sprayed into the flame by the nebulizer to evaporate the liquid and break down the solid complexes into atoms. The hollow cathode lamps are selected for the element of interest. The lamp provides the analytical light line (photons) with the same energy as the analyte atoms being absorbed in the flame. The

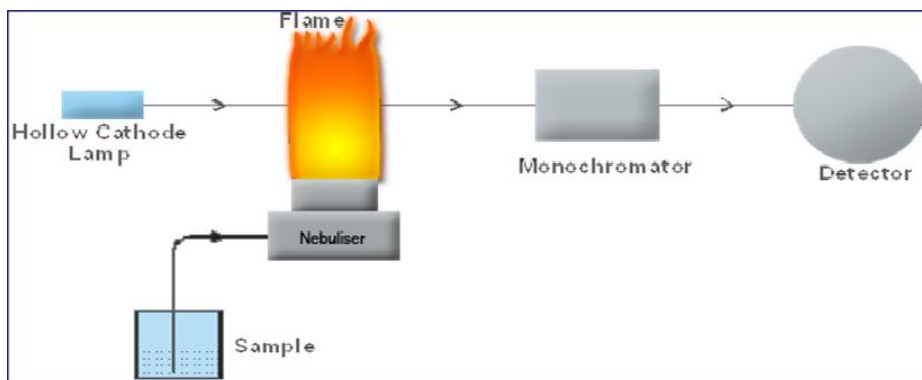


Figure 1. Atomic absorption spectrometer diagram

The monochromator helps isolate the analytical lines' photons passing through the flames and the photons are then converted into electron/signals by the detector. The detector is connected to a computer that displays the absorbance related to each solution ran. The known concentrations and absorptions were then used to construct a Beer's Law Plot to get the linear line equation needed to calculate the diluted and original solution concentrations of the standard solutions and tap water. The calcium standard was made from a calcium carbonate solution with a calcium concentrate of 604.3 ppm. Five standard solutions were prepared from that by making a table with calculated standard concentrations found by using the $M_1V_1=M_2V_2$ equation. Five concentrations were then

selected by the professor in ranges 1-10 ppm. The solutions were made with volumetric pipettes (1-5 ml) and volumetric flasks (250-1000 ml) in accordance to the table determined by the selected concentrations. DI water was used to dilute all the solutions. The magnesium standard was prepared in the same manner with the differences of first diluting a semi-stock of magnesium with DI in a 1:100 ratio before preparing the five standard solutions and selecting five standard concentrations in ranges of 0.1-1.0 ppm. Three diluted tap water solutions were made in ratios of 1:50, 1:20, and 1:4 for tap to DI water.

II. Ion Chromatography

A small sample is injected into the system and passes through the column. Inside the column the anions are being separated between the mobile and stationary phases based on ion-ion interaction. The more attracted to the column, the more it will stay in stationary phase and have a longer retention time. The eluent used was aqueous Na_2CO_3 and NaHCO_3 to keep the anions from leaving the stationary phase and promote ion exchange to avoid a net charge. The anions will pass through the suppressor as seen in figure 2. The suppressor is where Na_2CO_3 and NaHCO_3 are converted into H_2CO_3 , a weak acid that produces less conductivity for a better signal and detection of smaller ions in the detector.

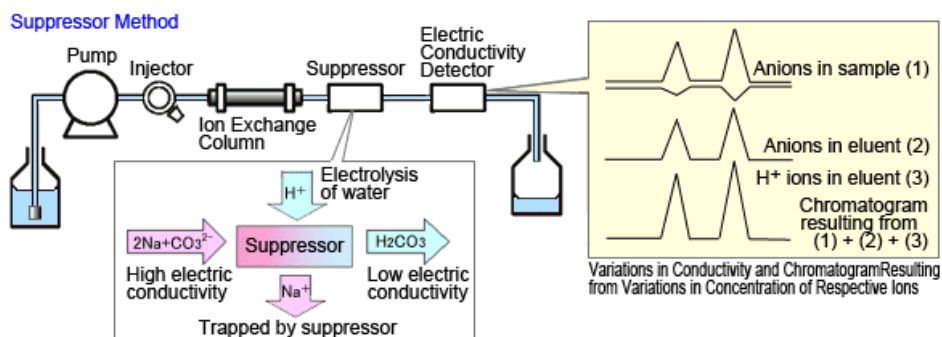


Figure 2. Ion chromatography diagram

Two solution standards were made at 50% and 150% of the estimated concentrations of the unknown. The concentration of the unknown was first calculated and plugged into the $M_1V_1=M_2V_2$ equation to make a table of concentrations based on the volumes of the pipettes and flasks needed, like the table in the AAS. Two concentrations were then picked that were closest to the estimated unknown concentrations and the standards were made with stock concentration of the identified unknown in accordance to the constructed table. The two standards were ran alongside the unknown and home tap water. The chromatographs were then used to construct a linear regression plot of peak area vs. concentration including the linear line equation and R^2 value. The peak of the unknown was plugged into the linear line equation to find the diluted concentration, which was then divided by the dilution factor to find the original concentration.

Results

A calibration curve was constructed for Ca^{++} and Mg^{++} with each exhibiting a strong linear relationship between absorbance and concentration. Calcium produced an R^2 value of 0.9993 (Figure 3) with an original concentration of 5.4 ppm and 95% confidence interval of +/- 0.4 ppm (Table 1). The standard deviation of calcium is 11.0% (Table 1). Magnesium produced an R^2 value of 0.9991 (Figure 4) with an original concentration of 11.2 ppm and 95% confidence

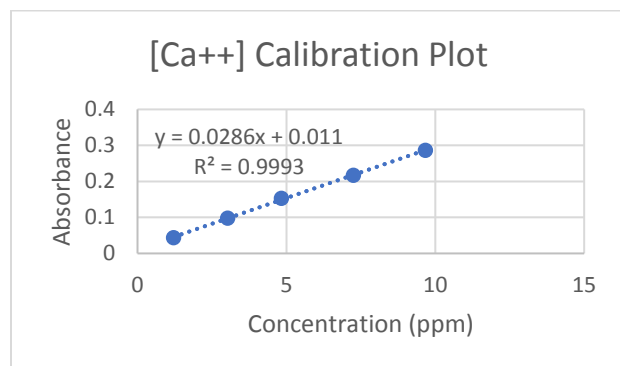


Figure 3. Ca^{++} ion standard calibration curve

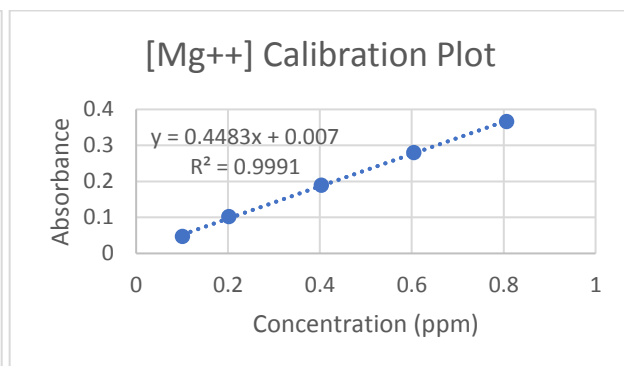


Figure 4. Mg^{++} ion standard calibration curve

Diluted Ca ⁺⁺ Concentration (ppm)		Diluted Mg ⁺⁺ Concentration (ppm)	
Absorbance	0.166	Absorbance	0.257
Concentration	5.4	Concentration	0.56
95% +/-	0.4	95% +/-	0.04
S _x	0.1103	S _x	0.1265
Original Ca ⁺⁺ Concentration (ppm)		Original Mg ⁺⁺ Concentration (ppm)	
Dilution Factor	1	Dilution Factor	0.05
Concentration	5.4	Concentration	11.2
95% +/-	0.4	95% +/-	7.7

Table 1. Concentrations of Ca⁺⁺ and Mg⁺⁺ for diluted and original tap water

interval of +/- 7.7 ppm (Table 1). The standard deviation of magnesium is 12.7% (Table 1). The concentrations of chloride, nitrate, and sulfate (Table 2) were found by using the instructor's standards along with the peak areas of the tap water plugged into the $M_1V_1=M_2V_2$ equation. The sample tap water hardness is 59.6 ppm and was found by adding the conversions of calcium carbonate from calcium and magnesium.

Compound	Retention Time (min.)	Standard Concentration (ppm)	Peak Area (μS*min)	Estimated Concentration (ppm)
Cl ⁻	1.93	20	0.934	6.69
NO ₃ ⁻	3.70	40	1.164	16.06
SO ₄ ²⁻	6.26	40	1.124	11.59

Table 2. Concentrations of ions from tap water

Discussion

The hardness of the sample tap water is 59.6 ppm, which falls right under the limit of 60 ppm for “soft” water hardness. The absorbances of calcium and magnesium in the tap water sample are within their standard limits of their calibration curve plots. Their concentrations are also within their maximum contamination levels (MCL) with magnesium under 50 ppm and calcium is NA due to it naturally occurring underground. The precision of calcium and magnesium are reliable with an accuracy of 83% for magnesium and 70% for calcium. The use of the one-point calibration

line works well for an estimated unknown concentration but can't be relied on for precision. This is due to the assumption that the standards were made correctly and used for one standard concentration point. The one-point calibration line is intended to be used as an estimated concentration needed to make other standards and find a more accurate concentration from a multipoint calibration curve. The estimated concentrations of chloride and sulfate are well below their MCLs of 500 ppm. Nitrate on the other hand is over its MCL of 10 ppm. A possible factor for the high nitrate concentration is the use of fertilizers in the area where the tap water was sourced from. These concentrations are only estimations made from their peak areas and there could be errors due to contamination during dilution, systematic error when making the standards, and or analytical error when injecting the sample. With consideration to these factors, the results for accuracy and precision of the estimated concentrations are reliable.

References

- D. Bhanot (2013) *Introduction to AAS Component Parts*. Retrieved April 15, 2018, from <http://lab-training.com>
- B. Baker, P. Curb, R. Dixon, & J. Toofan (2018) *Chemistry 31: Inorganic Quantitative Analysis Laboratory Manual*. California State University, Sacramento. Spring 2018.
- City of Sacramento Department of Utilities (2016). *2016 Water Quality Report: Consumers Confidence Report*. Retrieved April 15, 2018, from <https://www.cityofsacramento.org>
- K. Porter, M. Trautmann, M. McCasland, & R. Wagenet (2012). *Nitrate: Health Effects in Drinking Water*. Retrieved April 15, 2018, from <http://psep.cce.cornell.edu>
- Shimadzu Excellence in Science (2018) *Inorganic Anion Detection*. Retrieved April 15, 2018, from, <https://www.shimadzu.com>