# Analysis of Heavy Metal in Hair Samples using ICP-OES

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## Abstract

Exposure to heavy metals such as mercury, lead, and arsenic can negatively affect the structure and function of cells, tissues, and organs. Such heavy metals can be excreted from the hair as metabolic products during its growth process thus hair analysis is thought to be a reliable indicator for environmental accumulation of heavy metals. Our research aims to develop a methodology to quantify heavy metal concentrations found in biological samples using Inductively Coupled Plasma Spectroscopy. Current research involves preparing hair samples for analysis and creating calibration curves for heavy metals through a process that is accurate, precise, and easily replicated.

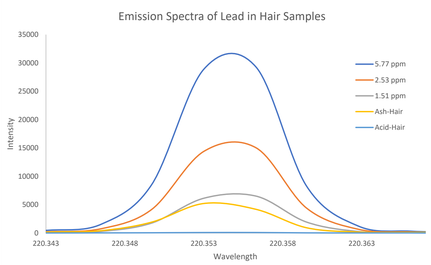
## Methodology

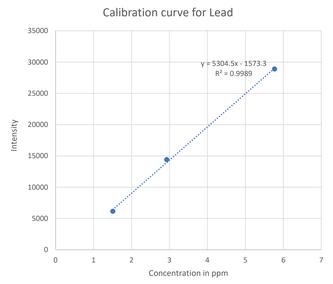
Inductively-coupled plasma optical emission spectrometry (ICP-OES) was the analytical machinery used to detect element concentrations. ICP-OES uses a high energy plasma, mostly composed of argon, which forms electrons and other charged species within the plasma matrix. The sample analyte is aerosolized using a nebulizer for detection through interaction with the plasma. Each element absorbs and emits light at a unique wavelength allowing the machine to measure the intensity of the desired element using a calibrated detector. By using known concentrations of an element and measuring the intensity calibration curves can be created using linear regression to quantify the unknown concentration of that specific element.

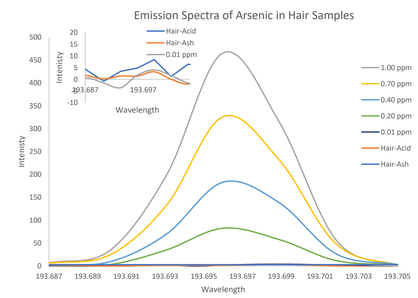
Using American Chemical Society grade standards of arsenic (As) and lead (Pb); dilutions were made in 2% $HNO_3$. A gram of hair samples was weighed in a crucible then dry ashed in an oven set at 510˚C for 3 hours to ash. The ashes were cooled then dissolved in 2% $HNO_3$ in a 50.00 mL volumetric flask, labeled hair-ash. Another sample was prepared by placing the sample in a test tube of 7 mL of 70% HNO3. This was placed inside of a water bath until fully dissolved. 2 mL of 35% H2O2 was added and placed into a sonicator then diluted in 200 mL of DI water. This sample was labeled as Sample Hair-Digest. Both samples were filtered through PTFE 0.45 µm before the final product was placed into the auto sample zone. The 2% $HNO_3$ in deionized water was used as the blank for each standard and sample.

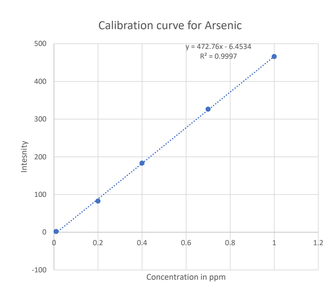
## Results

The graphs below represent the intensities of standard's concentration, hair samples, and a of Pb, and As respectively. Each graph was baselined at a single wavelength, which is unique for each element and was set by the machine. Then each spectrum was adjusted by subtracting the intensities attributed to the blank.




- The linear regression of the intensity versus concentration of the standards was taken and put into the slope-intercept form.
- Table 1 shows the spectral line, regression equations used for determining exact concentration as well as the correlation coefficient

Table-1

| | Spectral line | Regression equations | Correlation Coefficient |
|---|---|---|---|
| **Pb** | 220.353 | y=5304.5x-1573.3 | .9989 |
| **As** | 193.696 | y=472.76x-6.4534 | .9997 |

Table-2

| | |
|---|---|
| Plasma | 10.00 |
| Nebulizer | 0.55 |
| Auxiliary | 0.20 |

- The following concentration of lead and arsenic were calculated using the linear regression equations. Calculation results are shown in Table-3

Table-3

| | Hair-Ash | Hair-Digest |
|---|---|---|
| **Pb (ppm)** | 1.280 | 0.314 |
| **As (ppm)** | 0.0166 | 0.0238 |

- The measured intensities for arsenic were very low within the 1-5 range so it cannot be said with certainty how much arsenic is present.

## Discussion

- Previous studies show the average amount of lead in hair to be 3.53 ppm . The tested hair samples show values below that limit deeming them acceptable by Mayo Clinic.

| Element Tested | Hair-Digest (ppm) | Hair-Ash (ppm) | Safe |
|---|---|---|---|
| Pb | 1.280 | 0.314 | Yes |
| As | 0.0166 | 0.0238 | Inconclusive |

- It is important to note the varying concentration of lead and arsenic between the two different sample preparation methods. The higher concertation in the ash method could be due to the abrasive nature the sample was prepared in leading to degradation of some of the lead. However, the opposite was seen with arsenic having a higher concentration in the hair-digest method.
- Arsenic was seen at very low intensities making the results not entirely reliable. The detection limit for arsenic would need to be determined for us to say with confidence there is less than 0.0238 ppm of arsenic.
- Future samples will be run using different elements such as cadmium and mercury and the arsenic will be re-evaluated using more sensitive settings

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## Bibliography

1.Michalak, I., Wołowiec, P., & Chojnacka, K. (2014). Determination of exposure to lead of subjects from southwestern Poland by human hair analysis. *Environmental monitoring and assessment*, *186*(4), 2259–2267. https://doi.org/10.1007/s10661-013-3534-3

2.Gaines, P. (n.d.). Ashing Procedures. Retrieved from https://www.inorganicventures.com/trace-analysis-guide/ashing-procedures

3.Inductively coupled plasma atomic emission spectroscopy. (2019, December 20). Retrieved from https://en.wikipedia.org/wiki/Inductively_coupled_plasma_atomic_emission_spectroscopy

4.Hair Elements. (n.d.). Retrieved from https://www.doctorsdata.com/hair-elements/

5.https://www.mayocliniclabs.com/test-catalog/Clinical+and+Interpretive/8651