Analysis of Lead (Pb^{2+} ion) in Drinking Water Collected from Various Parts of Newark, Edison (Including the MCC campus).

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Abstract:

The purpose of this research project was to determine the concentration of Lead (Pb^{2+}) in the drinking water of New Jersey residents. Lead is a toxic metal that can be harmful to human health even at low exposure levels therefor we needed to perform analytical techniques that can measure Lead (Pb^{2+}) in the drinking water at low concentrations. The techniques include analysis of lead through Atomic Absorption (AA), Electrochemistry, and Inductively coupled plasma mass spectrometry (ICP-MS). The task was to learn how to use each instrument to their full capacity by learning the components of it, how to use their respective software, and all other aspects that it takes to run that instrument. All these instruments are proven to be able to detect lead concentrations however each instrument had a different level of sensitivity.

Introduction:

Lead is a toxic metal that is harmful to human health at low concentrations. One can be exposed to lead through inhalation of lead particles generated by burning materials containing lead, ingestion of lead-contaminated dust, water, and food. [1]Once lead enters the body, it is distributed to organs such as the brain, kidneys, liver and bones. [1]In children it can cause behavior and learning problems, lower IQ, hyperactivity, slowed growth, hearing problems, anemia. [1]In pregnant women it can cause reduced growth of the fetus and premature birth. [1]In adults it can cause cardiovascular effects, increased blood pressure and incidence of hypertension, decreased kidney function, reproductive problems (in both men and women). There is a water crisis in Newark, New Jersey which has made news headlines as the residents of Newark have concentrations of lead that exceed the EPA limit of 15ppb. [2]Newark first became aware of elevated lead levels in 2017 after the city changed the water's acidity, which may have made it more corrosive and caused lead from the pipes to enter the water supply. Analyzing the concentrations of lead in the water samples of not only Newark residents but others around New Jersey can help identify if there is lead contamination throughout our state.

Experimentation:

Equipment/Materials: (Electrochemistry)

- CV-50W Voltammetric Analyzer
- Ag/AgCl (Saturated KCl) reference electrode
- Pt Wire (Working electrode)
- Pt Counter Electrode
- KCl ASC Reagent
- DI Water
- Lead Nitrate ASC Reagent

Equipment/Materials: (Atomic Absorption)

- Buck Scientific Atomic Absorption/Emission Spectrometer 210VGP
- Lead hollow cathode lamp
- 1000ppm Lead Stock standard
- Nitric Acid (trace metal grade)
- DI Water

Equipment/Materials: (ICP-MS)

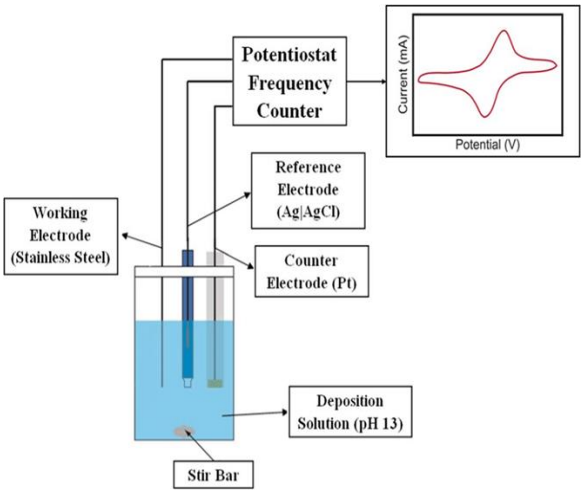
- Agilent Technologies 7800 ICP-MS
- Agilent Technologies 8900 ICP-MS Triple Quad
- 1000ppm Lead Stock standard solution
- Sc 1000ppm Stock Standard solution
- Nitric Acid (trace metal grade)
- DI Water

Part 1: Collecting the water samples

Sample ID	Location
196159	MCC Main Hall
196160	Edison, NJ
196161	Newark, NJ
196162	Westfield, NJ
196163	New Brunswick, NJ

Collected drinking water samples from 5 different locations. The locations are Middlesex County College, Edison, Newark, Westfield, and New Brunswick. These locations were chosen at random to see if lead contamination was occurring in other parts of New Jersey besides Newark. Collecting the water samples was a difficult task as I had to travel to different areas and convince strangers to let me test their drinking water for lead contamination. Various people refused to even open the door to a stranger and others simply said no upon my request. All water samples collected were filtered water as that is the water that they drink from. Assigned the water samples NJL ID's which is the samples code at New Jersey Laboratories. It begins with the year followed by a number in the sequential order.

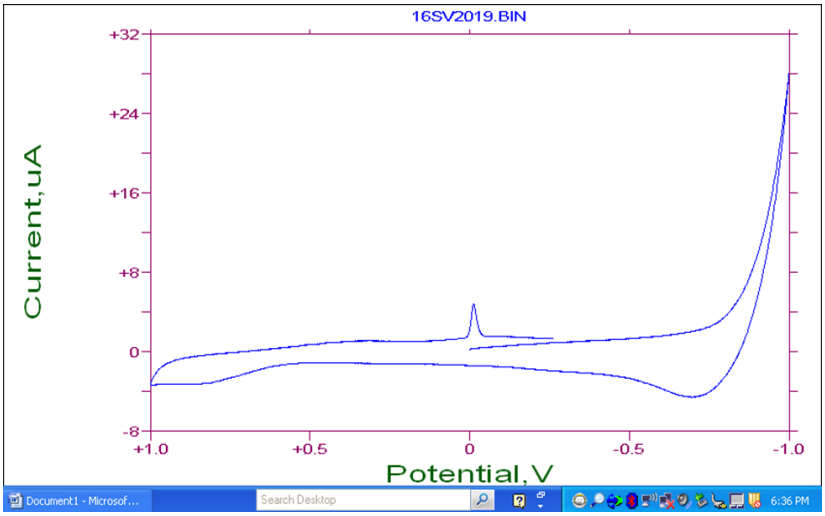
Part 2: Cyclic Voltammetry



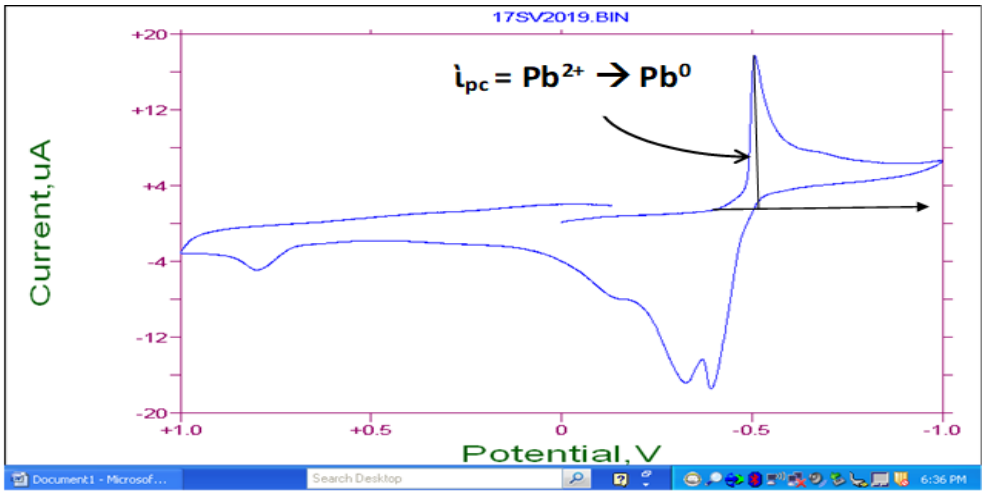
Cyclic Voltammetry (CV): is an electrochemical tool which measures the current (i_P) under a potential gradient applied to a specific electrode (Pt or GC). CV is performed by scanning the potential between +1.0 V to -1.0V and eventually measuring the resulting current. The Working Electrode is the electrode in an electrochemical system on which the reaction of interest is occurring. The working electrode is often used in conjunction with an auxiliary electrode, and a reference electrode in a three-electrode system. The Counter Electrode is an electrode used in a three-electrode electrochemical cell for voltammetry analysis in which an electric current is expected to flow. The Reference Electrode is an electrode which has a stable and well-known electrode potential.

The electrochemical analysis of Pb was conducted by Riyanto et el. *Determination of Lead in Wastewater Using Cyclic Voltammetry By Platinum Wire Electrode.EKSAKTA Vol. 14, No. 2, pg. 22-33. (Indonesian Journal).* The published journal used a different cyclic voltammeter than the one available to us a Middlesex County College. The goal of this analysis is to see if our instrument with its parameters and electrodes can detect Pb^{2+} and what is the maximum concentration the instrument is capable of reading. The electrochemical analysis of Pb was performed in a 0.1M KCl solution at room temperature. The cyclic voltammetry analysis was performed in a three-electrode system using Pt wire as the working electrode, an Ag/AgCl (saturated KCl) as the reference electrode, and Pt plate as the counter electrode.

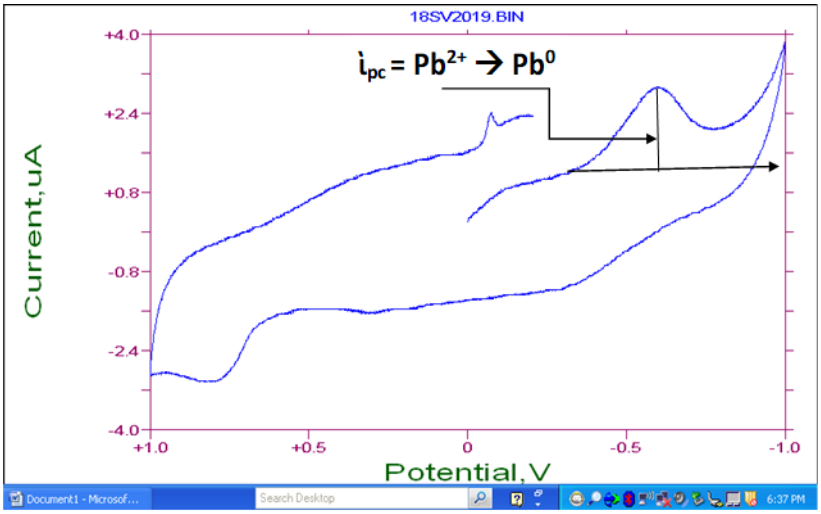
Tried to get a calibration curve by the same method as the journal. Ran the baseline of the 0.1M KCl solution.



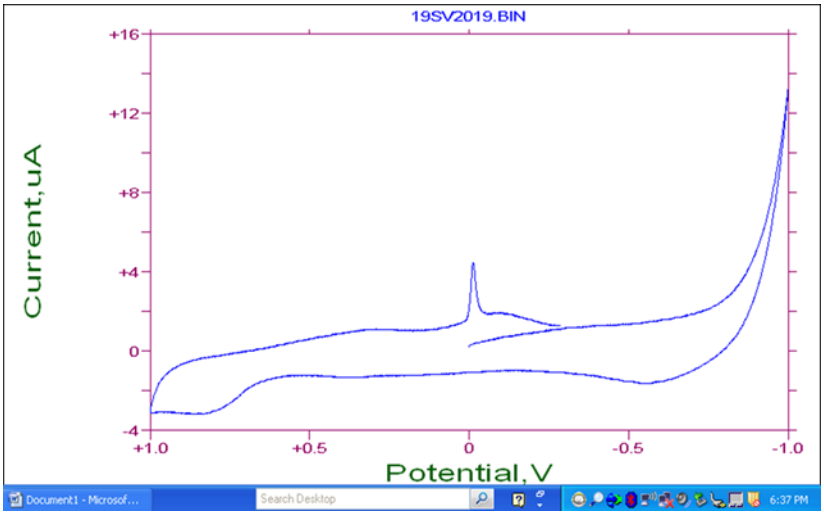
Then injected 10µL of $Pb(NO_3)_2$ 1000mg/L and ran to get the voltammogram. The instrument could not detect nothing and the resulting voltammogram was the same as the baseline. As we thought that maybe our instrument was not capable of detecting lead, Prof. Ghosh insisted that we measured the $Pb(NO_3)_2$ similarly to the way other voltammetry analysis are read in this instrument.



17SV2019: 3.3mg of Lead nitrate in 10mL of 0.1M KCl Soln. = 0.001M Lead Nitrate soln ~ 250ppm Lead Conc.



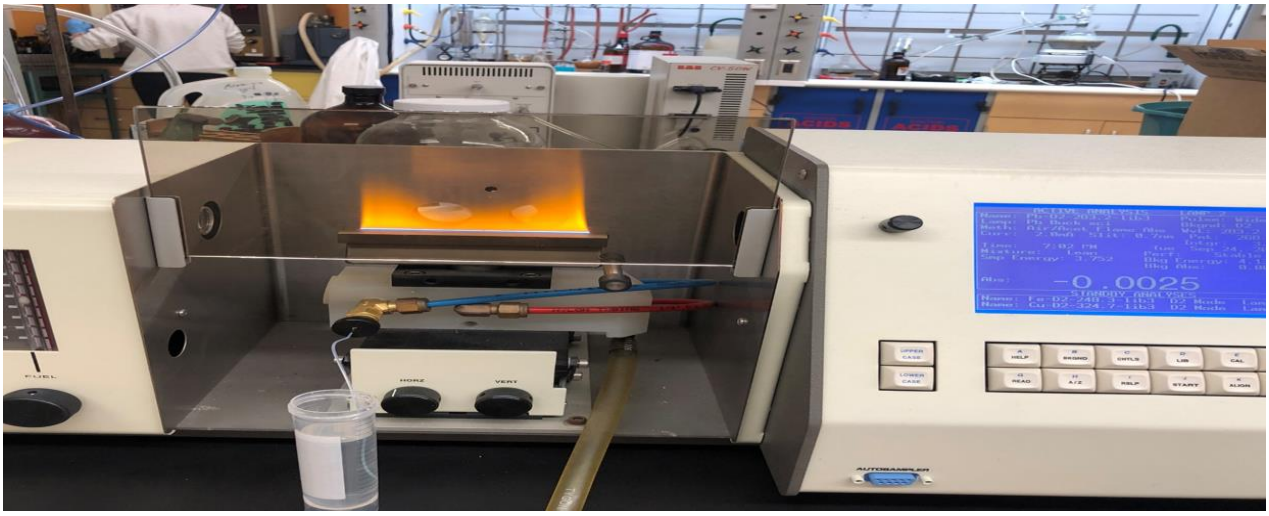
18SV2019: 1mL above soln + 9mL 0.1KCl soln. = 0.0001M Lead Nitrate soln ~ 25ppm Lead Conc.



19SV2019: 1mL above soln + 9mL 0.1KCl soln. = 0.00001M Lead Nitrate soln ~ 2.5ppm Lead Conc.

Our resulting voltammograms show that our instrument can detect Pb^{2+}. However, the instrument can only measure up to 25ppm concentration.

Part 3: Atomic Absorption



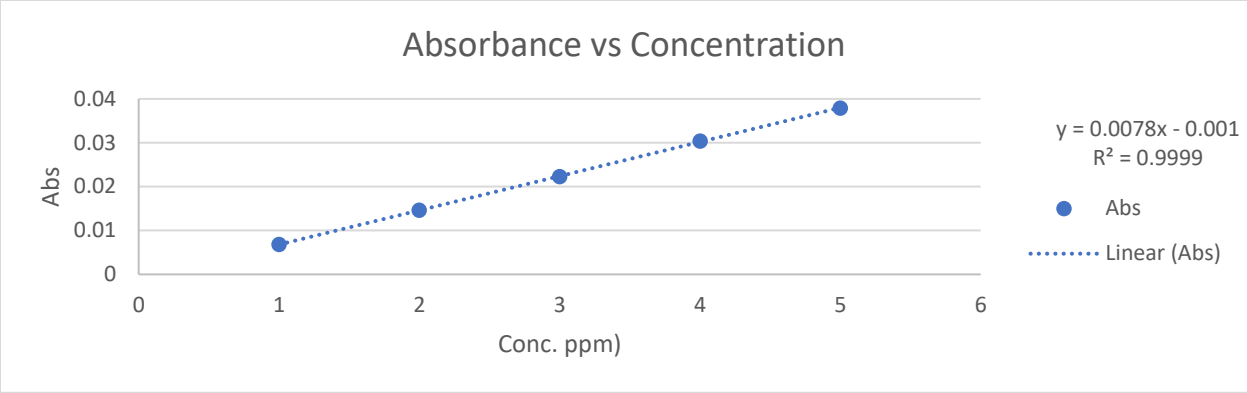
Atomic absorption spectrometry (AAS) is a technique in which free gaseous atoms absorb electromagnetic radiation (light) at a specific wavelength to produce a measurable signal (Absorbance). Needed to create a calibration curve of standards of known concentration of lead. Prepared the following standards using a 1000ppm stock standard lead solution. All dilutions were prepared in 5% HNO_3.

mL of 1000ppm stock solution	**Final Volume**	**Final Conc.** **(ppm = mg/L)**
0.1mL	100mL	1ppm
0.2mL	100mL	2ppm
0.3mL	100mL	3ppm
0.4mL	100mL	4ppm
0.5mL	100mL	5ppm

The Results are as follows:

Std	Abs 1	Abs 2	Abs 3	Avg Abs
1ppm	0.0062	0.0073	0.0070	0.0068
2ppm	0.0144	0.0150	0.0144	0.0146
3ppm	0.0218	0.0228	0.0223	0.0223
4ppm	0.0300	0.0310	0.0301	0.0304
5ppm	0.0374	0.0382	0.0382	0.0379

Plotted the results in an Absorbance Vs. Concentration graph.



As we can see by our R^2 value the data collected for the standards are accurate. Now measured the water samples and the results are as follows.

Sample ID	Abs
196159, MCC	-0.0025
19160, Edison, NJ	-0.0030
196161, Newark, NJ	-0.0018
196162, Westfield, NJ	-0.0021
196163, New Brunswick, NJ	-0.0024

The samples absorbance cannot be measured as the negative number indicates that the instrument cannot detect the conc. of lead ions present in the water. Our conclusion is that the amount of lead in the samples are below1ppm!

Part 4: ICP-MS

Inductively coupled plasma mass spectrometry (ICP–MS) is an instrumental analytical technique based on the use of a high temperature ionization source (ICP) coupled to a mass spectrometer. It's a type of mass spectrometry that uses an Inductively coupled plasma to atomizes a sample and create atomic and small polyatomic ions, which are then detected. It is known and used for its ability to detect metals and several non-metals in liquid samples at very low concentrations from ppm to ppt. The ICPMS consists of a sample introduction system which is composed of a nebulizer and spray chamber and provides the means of getting samples into the instrument. An ICP torch and RF coil which generates the argon plasma, which serves as the ion source of the ICP-MS. The interface links the atmospheric pressure ICP ion source to the high vacuum mass spectrometer. A Vacuum system – provides high vacuum for ion optics, quadrupole, and detector. The Ion optics guide the desired ions into the quadrupole while assuring that neutral species and photons are discarded from the ion beam. The Collision/reaction cell precedes the mass spectrometer and is used to remove interferences that can degrade the detection limits achieved. The Mass spectrometer – acts as a mass filter to sort ions by their mass-to-charge ratio (m/z) and the Detector counts individual ions exiting the quadrupole. All aspects of the instrument are controlled by the system controller and data handling to obtain final concentration results.

For ICP-MS we also need to create a calibration curve of standards of known concentration of lead. Prepared the following standards using a 1000ppm stock standard lead solution. All dilutions were prepared in 5% HNO_3. Prepared a 1ppm Solution as follows:

1000ppm ($\mathbf{1000}\boldsymbol{ppm} \times \mathbf{0.05}\boldsymbol{mL}$)/$\mathbf{50}\boldsymbol{mL} = \mathbf{1}\boldsymbol{ppm}$

mL of 1ppm stock solution	**Final Volume**	**Final Conc.** **(ppm = mg/L)**
0.0125mL	50mL	0.25ppb
0.05mL	50mL	1ppb
0.1mL	50mL	2ppb
0.25mL	50mL	5ppb

0.5mL	50mL	10ppb

Once all the standards are prepared created a sequence in the ICP-MS to run and analyze the samples. The instrument Mass Hunter software takes the counts of individual ions exiting the quadrupole and from that generates data handling to obtain final concentration results. The data is transferred to an PPB ICP-MS Auto Calculation excel sheet.

Results:

Element	Lead	ICP-MS #	2	Date of Instrument Analysis	30Sep2019
Book #	539	Page (s)	155	OOS # (if applicable)	N/A
Isotope #	206	Tuning Mode	H2 Gas	DR# (if applicable)	N/A
Units	PPB				

Calibration:	Concentration of Std	Ratio	Corrected Ratio	Correlation Coefficient	0.9996
0	0	0.001083673	0.00000000	Correlation	NLT 0.9975
1	0.25	0.005616297	0.00453262	Pass/Fail	PASS
2	1	0.016076128	0.01499246		
3	2	0.029910079	0.02882641		
4	5	0.065923841	0.06484017		
5	10	0.135099983	0.13401631		

*µg found = 0.001*DF*conc from graph

µg/serving = µg found*serving size(g)/smp. Wt (g)

Calibration Verification

Theoretical Concentration	Ratio	Corrected Ratio	Actual Found	% Recovery Specification: 85% - 115%	Pass/Fail
5.0	0.067369046	0.06628537	4.94	99	PASS

Drift Calculation

Standard Ratio	(Corrected) Drift Standard Ratio	%Recovery	Final Result (%)	Specification	Pass/Fail
0.13401631	0.13848573	103	3	NMT 20%	PASS

Sample ID	Serving size (g)	Specification/ Target	Smp Wt (g)	Ratio	Corrected Ratio	conc from graph	Dilution Factor	*µg found	µg/serving	mg/serving
196159.0000	1.0000	Report Only	1.00000	0.001260189	0.00017652	-0.05734104	1	-0.00005734	-0.00005734	-0.00000006
196160.0000	1.0000	Report Only	1.00000	0.002215246	0.00113157	0.01480385	1	0.00001480	0.0000148	0.0000000
196161.0000	1.0000	Report Only	1.00000	0.043282318	0.04219864	3.11700387	1	0.00311700	0.0031170	0.0000031
196162.0000	1.0000	Report Only	1.00000	0.001508735	0.00042506	-0.03856586	1	-0.00003857	-0.0000386	0.0000000
196163.0000	1.0000	Report Only	1.00000	0.001100053	0.00001638	-0.06943770	1	-0.00006944	-0.0000694	-0.0000001
					-0.00108367	-0.15253549		0.00000000	#DIV/0!	#DIV/0!
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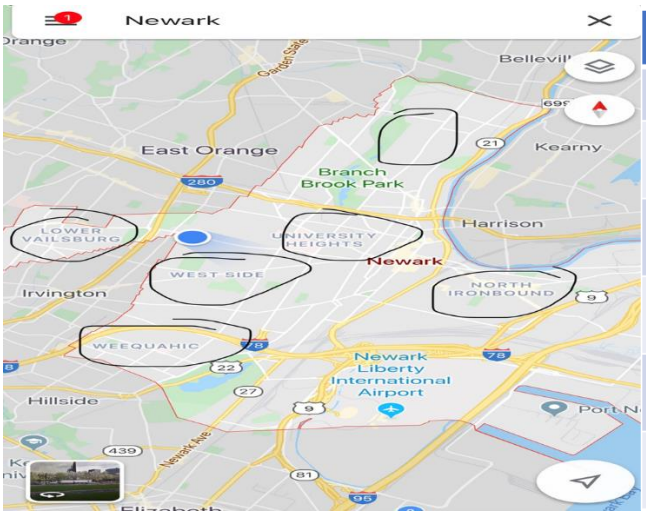
					-0.00108367	-0.15253549		0.00000000	#DIV/0!	#DIV/0!
Drift Std		10ppb		0.139569406	0.13848573	10.39051514		0.00000000	#DIV/0!	#DIV/0!

196159 (MCC), 196162 (Westfield), and 196163 (New Brunswick) the lead conc. is not detected which ensures us that the concentration of lead is below 0.25ppb. 196160 (Edison) has 0.0148ppb of Pb and 196161(Newark) has 3.117ppb of Pb in their water samples. The amount of lead in all the water samples did not exceed the EPA limit of 15ppb of Pb. However, noticed that

the water sample from Newark, NJ did have small traces of lead. Even small amounts of lead can severely affect the human body therefor more water samples from different areas of Newark will be collected.

Part 5: Collecting more water samples from Newark and analyzing conc in ppt



Sample ID	Location
197207	Lower Vailsburg, Newark NJ
197208	West Side, Newark NJ
197209	Weequahic, Newark NJ
197210	North Ironbound, Newark NJ
197211	University Heights, Newark NJ
197212	Forest Hill, Newark NJ

To further analyze the lead concentrations in Newark, NJ the water samples are going to be tested using a newer ICP-MS instrument model that can measure the concentration of lead in parts per trillion. For this ICP-MS we also need to create a calibration curve of standards of known concentration of lead. Prepared the following standards using a 1000ppm stock standard lead solution. All dilutions were prepared in 5% HNO_3. Prepared a 1ppm and10ppb Solution as follows:

1000ppm $(\mathbf{1000}\boldsymbol{ppm} \times \mathbf{0.05}\boldsymbol{mL})/\mathbf{50}\boldsymbol{mL} = (\mathbf{1}\boldsymbol{ppm} \times \mathbf{0.5}\boldsymbol{mL})/\mathbf{50}\boldsymbol{mL}$ = 10ppb soln.

mL Of 10ppb stock solution	mL Of 1ppm stock solution	Final Volume	Final Conc. (ppb = µ g/L)
0.05mL	N/A	50mL	0.01ppb
0.5mL	N/A	50mL	0.1ppb
N/A	0.025mL	50mL	0.5ppb
N/A	0.05mL	50mL	1ppb
N/A	0.1mL	50mL	2ppb

Once all the standards are prepared created a sequence in the ICP-MS to run and analyze the samples. The instrument Mass Hunter software takes the counts of individual ions exiting the quadrupole and from that generates data handling to obtain final concentration results. The data is transferred to an PPB ICP-MS Auto Calculation excel sheet.

Results:

Element	Lead	ICP-MS #	2	Date of Instrument Analysis	30Sep2019
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Isotope #	206	Tuning Mode	H2 Gas	DR# (if applicable)	N/A
Units	PPB				

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1	0.25	0.005616297	0.00453262	Pass/Fail	PASS
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4	5	0.065923841	0.06484017		
5	10	0.135099983	0.13401631		

*µg found = 0.001*DF*conc from graph

µg/serving = µg found*serving size(g)/smp. Wt (g)

Calibration Verification

Theoretical Concentration	Ratio	Corrected Ratio	Actual Found	% Recovery Specification: 85% - 115%	Pass/Fail
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Drift Calculation

Standard Ratio	(Corrected) Drift Standard Ratio	%Recovery	Final Result (%)	Specification	Pass/Fail
0.13401631	0.13848573	103	3	NMT 20%	PASS

Sample ID	Serving size (g)	Specification/ Target	Smp Wt (g)	Ratio	Corrected Ratio	conc from graph	Dilution Factor	*µg found	µg/serving	mg/serving
196159.0000	1.0000	Report Only	1.00000	0.001260189	0.00017652	-0.05734104	1	-0.00005734	-0.00005734	-0.00000006
196160.0000	1.0000	Report Only	1.00000	0.002215246	0.00113157	0.01480385	1	0.00001480	0.0000148	0.0000000
196161.0000	1.0000	Report Only	1.00000	0.043282318	0.04219864	3.11700387	1	0.00311700	0.0031170	0.0000031
196162.0000	1.0000	Report Only	1.00000	0.001508735	0.00042506	-0.03856586	1	-0.00003857	-0.0000386	0.0000000
196163.0000	1.0000	Report Only	1.00000	0.001100053	0.00001638	-0.06943770	1	-0.00006944	-0.0000694	-0.0000001
					-0.00108367	-0.15253549		0.00000000	#DIV/0!	#DIV/0!
					-0.00108367	-0.15253549		0.00000000	#DIV/0!	#DIV/0!
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					-0.00108367	-0.15253549		0.00000000	#DIV/0!	#DIV/0!
Drift Std		10ppb		0.139569406	0.13848573	10.39051514		0.00000000	#DIV/0!	#DIV/0!

- 197205 Lead not detected <0.01ppb (Susan – A) (Dr. Susan Altman, Director of CELT)
- 197206: Lead not detected <0.01ppb (Susan – B)
- 197207 (Lower Vailsburg): 0.32ppb of Pb
- 197208 (West Side): 0.067ppb of Pb
- 197209 (Weequahic): 3.88ppb of Pb
- 197210 (North Ironbound): 0.123ppb of Pb
- 197211(University Heights): 0.157ppb of Pb
- 197212 (Forest Hill): 0.264ppb of Pb

The amount of lead in all the water samples did not exceed the EPA limit of 15ppb of Pb. With the new linearity smaller concentrations of Pb was found in the water samples of Newark residents. Even this small amount is hazardous to the human health.

Conclusion:

The amount of lead in our drinking water can have severe health implications on our body. Even at low levels as parts per billion and parts per trillion is enough to affect us. All the water samples tested below the EPA limit of 15ppb. However, the Newark water samples did show small concentrations of lead.

Acknowledgements:

Dr. Phalguni Ghosh and Dr. Steven Rowley

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John Engell, David Rosen, Gita Apparaju

New Jersey laboratories CEO Sandra B. Lee

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