

Quantitative Determination of Heavy Metals in Ready to Eat Food Products

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Abstract:

The heavy metals (such as Cd, Pb, Zn, Cu etc.) at very low concentration from aqueous solution can be analyzed using AAS, ICP-MS, spectrophotometry and Voltammetry. Each method has its advantages and limitations. Voltammetry is one of the advanced techniques with a very high detection limit. The concentration of metal up to micro-molar or lower than that can be detected using Voltammetric technique. It also can detect more than one metal in the solution provided, if they have noticeable difference in their decomposition potential. The presence of heavy metals, in various ready-to-eat food products (like noodle, pasta, soup etc.) available in local market is investigated in this work. The results indicate that these metals (such as Cd, Pb, Zn, Cu etc.) are detected in nearly all samples; however, their concentrations remain within the acceptable limits. Two different methods were used to prepare the experimental solutions of the said ready to eat food samples in this work, one is digestive juice condition and other is by ashing method. In most of the samples of noodle, pasta and soup, the amount of metals detected by using both methods are in good agreement with each other (which can be considered as validation of results). Overall it indicates that the ready to eat food products are not harmful, with respect to heavy metals, as they are not detected beyond the permissible limit.

Keywords: Voltammetry, Heavy Metal, Ashing, Digestive Conditions.

1. Introduction:

Heavy metals are elements with an atomic weight over 63.5 and a specific gravity higher than 5.0 that are generally harmful to human health and the environment. The major elements included in this class are: Lead (Pb), Cadmium (Cd), Cobalt (Co), Chromium (Cr), Copper (Cu), Iron (Fe), Arsenic (As), Nickel (Ni), Zinc (Zn) and Mercury (Hg)[1]. Out of these Heavy metals, Pb, Cd, Cu and Zn are potential environmental contaminants with the capacity of causing human health problems, which enter through the food. With growing international trade, food safety has emerged as an important global human health issue. The food items containing these hazardous metals in digestible form and present above the permissible limits, pose serious danger to human health[2]. They are given special attention throughout the world due to their toxic effects even at very low concentrations. Several cases of human disease, disorders, malfunction and malformation of organs, are probably due to metal toxicity.

Studies on these metals, are important from a public point of view, where attention has been drawn to the necessity of measuring the accumulation of heavy metals, particularly those metals, which pose serious health hazards to human (e.g. Cd and Pb). In today's industrial society, there is no escaping exposure to toxic chemicals and metals[3].

In globalization, industrial development and very fast lifestyle, the food consumption habits are also changing from more nutritious and balance diet to easy-to-cook / ready to eat food. The child's attention attracting advertising, colourful packaging and the most importantly time saving recipes (few minute type) all have also contributed to the growing demand for ready to eat food products. Basically, the increasing population and urbanization, the severe shortage of time on part of bachelors, spinsters and the working mothers must be the additional factor to the increasing consumption of ready to eat food. The change in feeding habits and way of life, have combined to make the ready to eat food (like noodles, pasta, soup mix etc.) very popular[4, 5]. Monitoring the concentrations of various metals in food is critical because these contaminants have deleterious effects on humans. Many illnesses and diseases such as hypertension, cancer, depression and mental disorders have been associated with increased concentrations of heavy metals such, as cadmium, lead, copper, chromium, nickel, manganese and zinc in human organs.

The literature high lights, the Uttar Pradesh Food Safety and Drug Administration prosecuted Nestle India as samples of their Maggi noodles were found to contain high levels of lead beyond the permissible level. In Bangladesh, some brands of commercial noodles were found to contain varying concentrations of different heavy metals. Very little study has been reported on heavy metal and health risk assessment on pastas consumed in Nigeria. Hence, this study aimed at assessing some heavy metal (Cd, Pb, and Zn) levels in locally produced and imported pastas (noodles, spaghetti and macaroni) [6].

2.METHODOLOGY

2.1 Collection of samples:

The commercially and readily available brand samples of ready to eat food products like Noodles (3 brands), Soups (2 brands), Oats (2 brands) and Pasta (2 Brands) with criteria of not being older than 3 months with respect to manufacturing date, were collected (purchased) from the reliable mega market stores.

2.2 Preparation of solutions:

2.2.1 Digestive condition:-

A known quantity of sample (5g) was soaked for about 30 mins in warm (70-78°C) in 0.1N HCl solution and then boiled for 15 minute. Then the obtained solution was filtered through Whatman Filter paper, then 0.745 g KCl (AR grade without further purification) was added to the standard flask (to get 0.1M KCl to act as supporting electrolyte for voltammetry) and finally diluted to 100 cm³ with distilled water.

2.2.2. Ashing and solution of white ash:-

A known quantity of sample (10g) was ashed in the furnace at 800-900°C till complete white ash was obtained (about 5 to 6 hrs). Then the obtained white ash was transferred to the Borosil beaker and by adding 10 cm³ concentrated HCl was heated on sand bath to evaporate the HCl completely, the same step is repeated one more time and finally the obtained residue is extracted in 20 - 30 cm³ distilled water by boiling for 10 to 12 minutes. The obtained solution is then filtered through Whatman Filter paper, then AR grade KCl (as mentioned above in 2.2.1) was added to the standard flask and finally diluted to 100 cm³ with distilled water.

2.2.3. Standard 50 ppm stock solution:

The standard 50 ppm stock solutions with respect to Cd, Pb, Zn and Cu was prepared by dissolving 0.1015g of CdCl₂.5H₂O; 0.0915g of Pb(CH₃COO)₂.3H₂O; 0.1042g of ZnCl₂; 0.126 g CuSO₄.5H₂O and

7.45 g KCl (0.1M KCl to act as supporting electrolyte for voltammetry) in 1000 cm³ using distilled water. This one stock solution has 50 ppm with respect to Cd, Pb, Zn and Cu. This stock solution was used for standard addition method of Voltammetric measurements by both method digestive conditions and ashing method.

3. EXPERIMENTAL DETAILS

The Metrohm Potentiostat voltammetry station 884 Professional VA is used for this project. All the Voltammetric measurements are carried out by standard addition method as the amount of metals expected in the sample solutions of ready to eat food products is expected to be in PPB level, where calibration curve may not suit the concentration range.

In the Voltammetric cell initially 20 cm³ experimental sample solution was taken and to this 0.5, 1.0, 1.5, 2.0 and 2.5 cm³ of 50 ppm stock was added. The sample DC voltammetric curves were recorded for all 6 solutions that are the sample and 5 additions of the standard. The instrumental parameters are given in Table 1.

Table 1: Voltammetric instrumental parameters and settings used for recording all Voltammogram

Parameter	Settings and values
Mode	Sample DC
Working electrode	Static Mercury drop
Reference electrode	Ag/AgCl
Method	Standard addition
Nitrogen Purge Time	3 min
Drop Size	Medium
Drop time	2 Sec
Initial Potential	-1.2V
Final potential	0.1V
Current range	Auto selected to 500nA or 1mA
Ensemble average	Three runs

4. OBSERVATIONS:

The voltammograms recorded as per the experimental set up given in Table 1 with sample prepared by using digestive conditions (method 2.2.1) are shown in Figure 1.1 to Figure 1.9

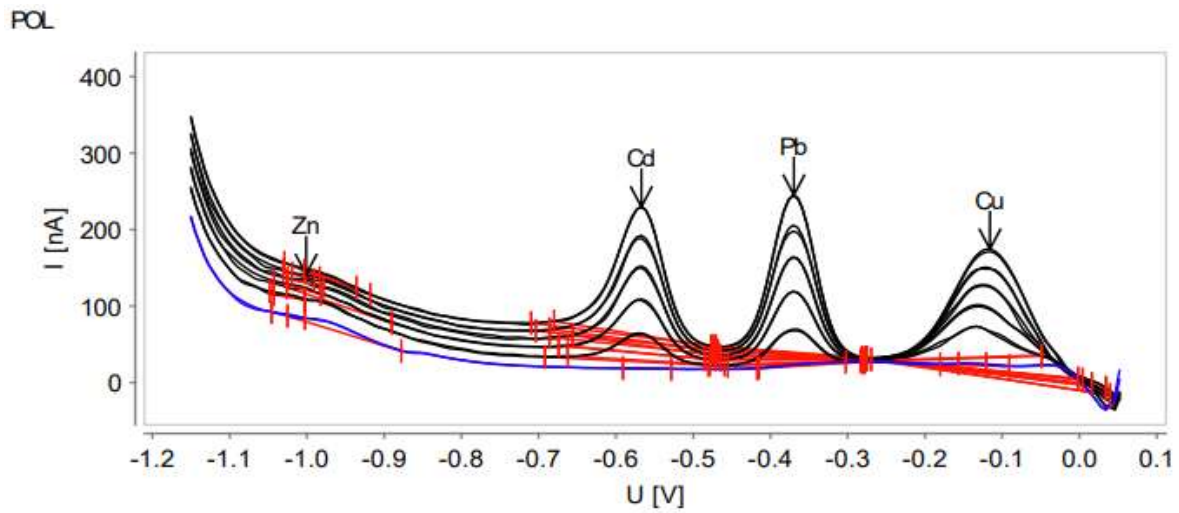


Figure 1.1: Sample DC voltammogram for Noodles sample A solution and 5 standard additions

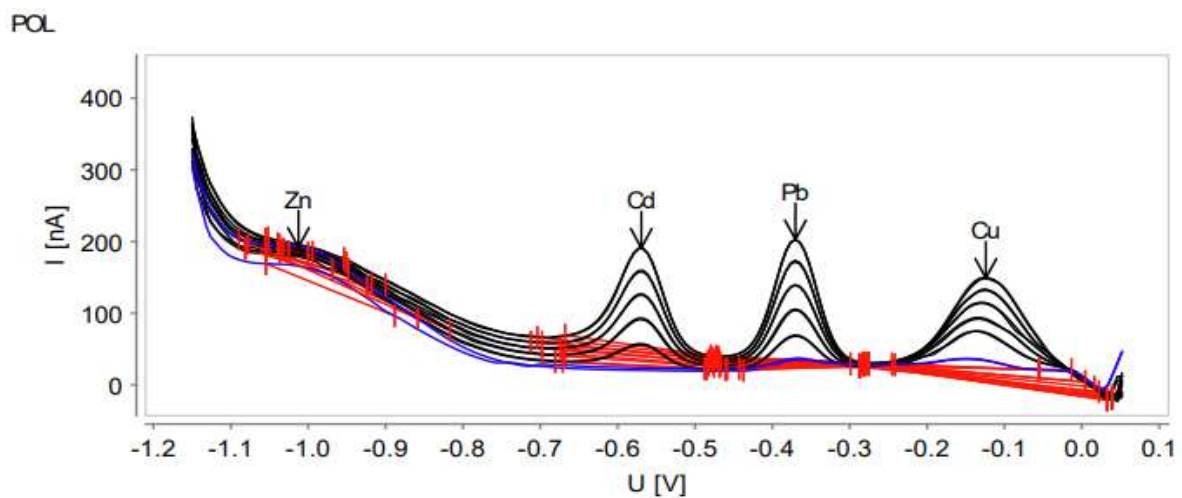


Figure 1.2: Sample DC voltammogram for Noodles sample B solution and 5 standard additions

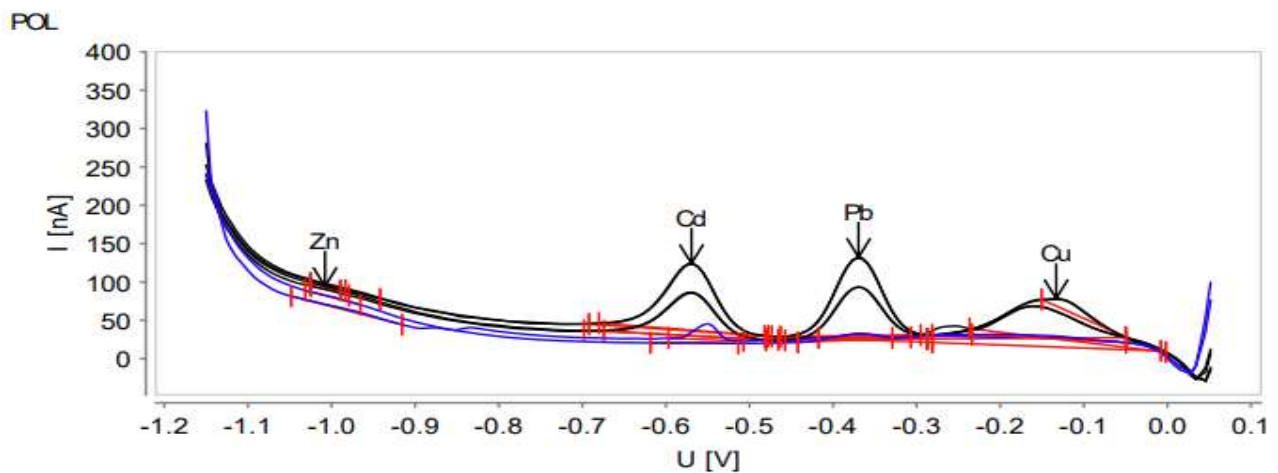


Figure 1.3: Sample DC voltammogram for Noodles sample C solution and 5 standard additions

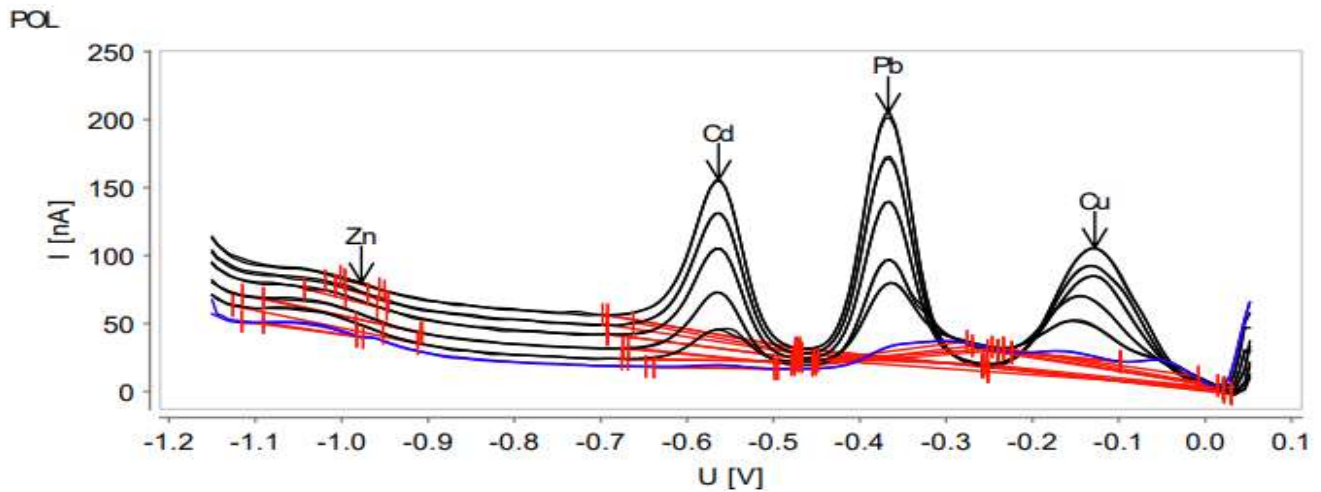


Figure 1.4: Sample DC voltammogram for Pasta sample A solution and 5 standard additions

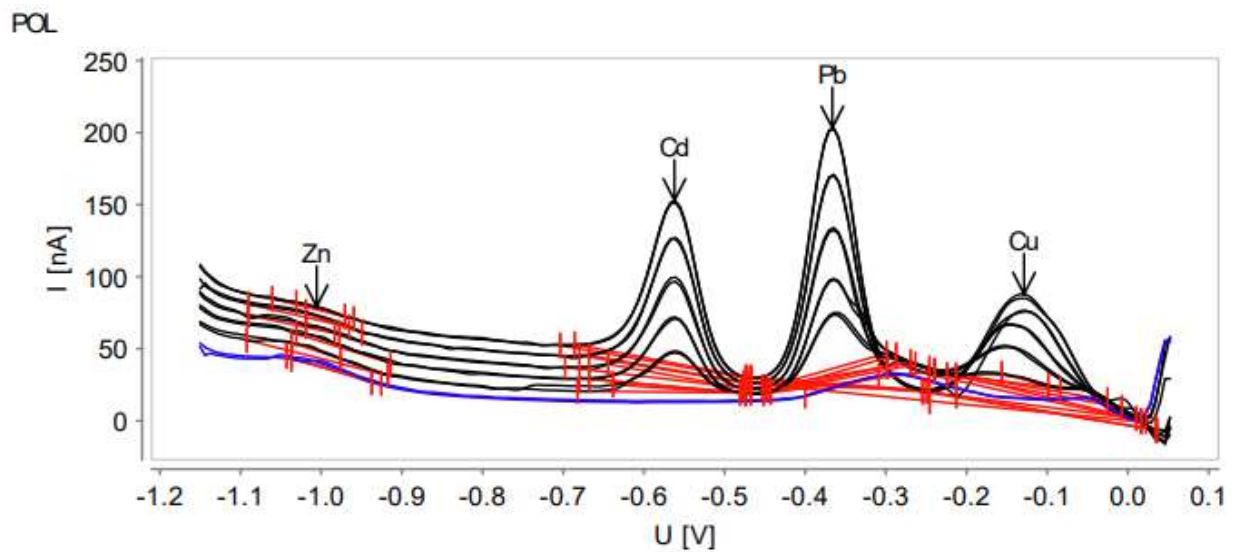


Figure 1.5: Sample DC voltammogram for Pasta sample B solution and 5 standard additions

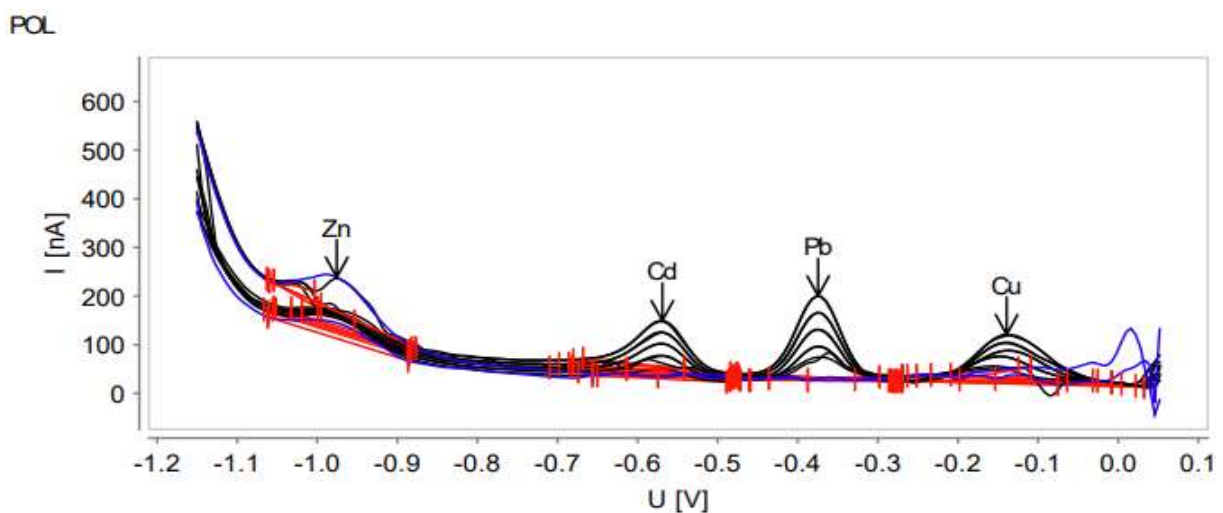


Figure 1.6: Sample DC voltammogram for Oats sample A solution and 5 standard additions

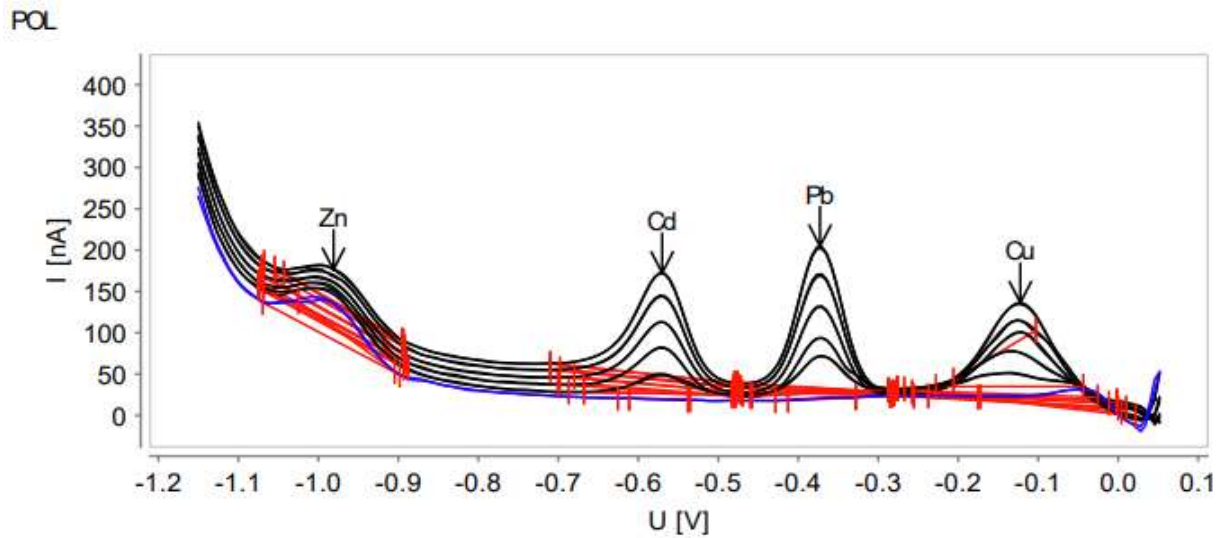


Figure 1.7: Sample DC voltammogram for Oats sample B solution and 5 standard additions

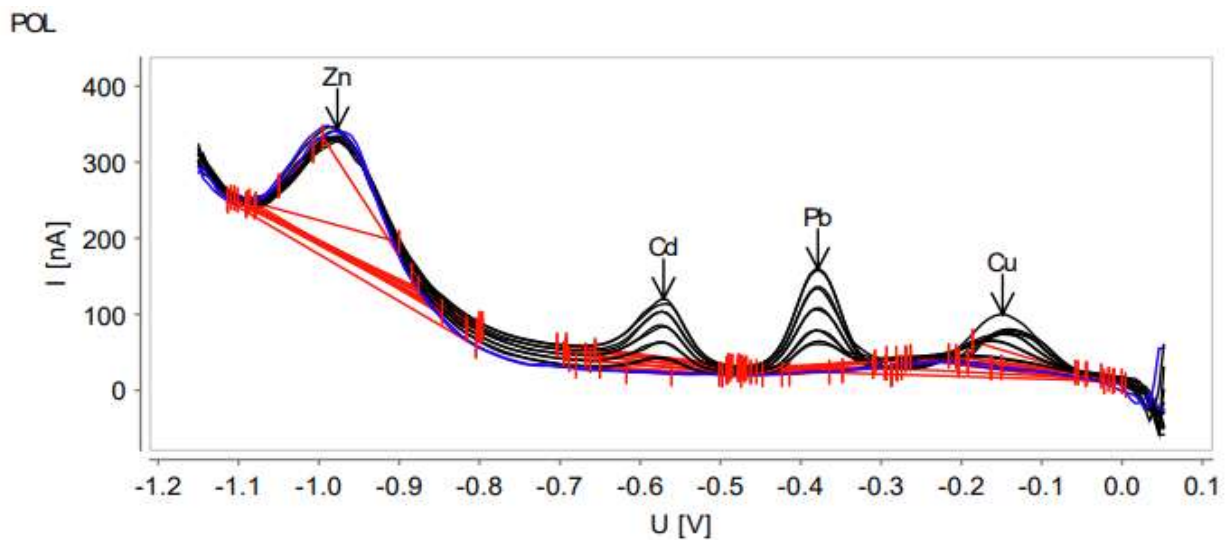


Figure 1.8: Sample DC voltammogram for Soup sample A solution and 5 standard additions

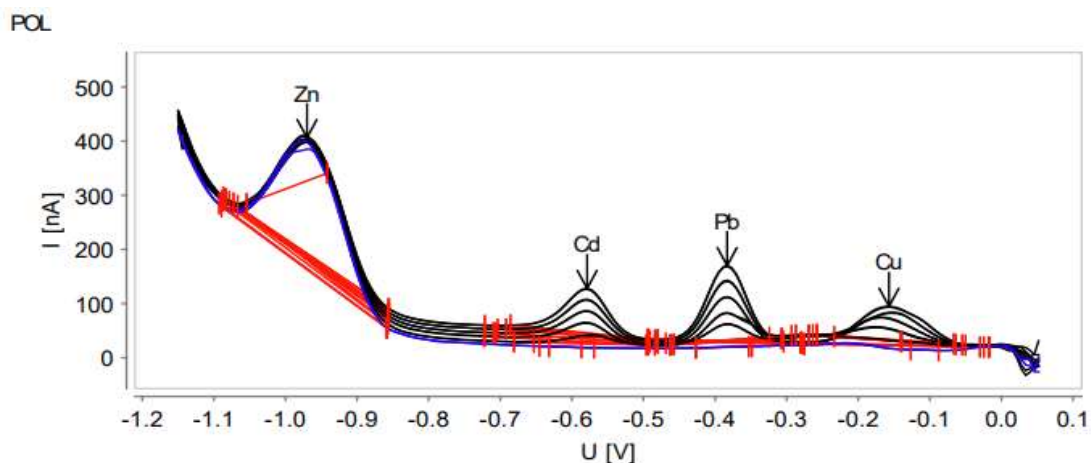


Figure 1.9: Sample DC voltammogram for Soup sample B solution and 5 standard additions

Observations of ashing samples:

The ashing method was used on some of the samples as these samples were prepared to validate the method of preparing the digestive condition solutions method to determine the presence of hazardous metals like Cd, Pb, Zn and Cu. The voltammograms recorded as per the experimental set up given in Table 1, with sample prepared by using ashing (method 2.2.2) are shown in Figure 2.1 to Figure 2.9

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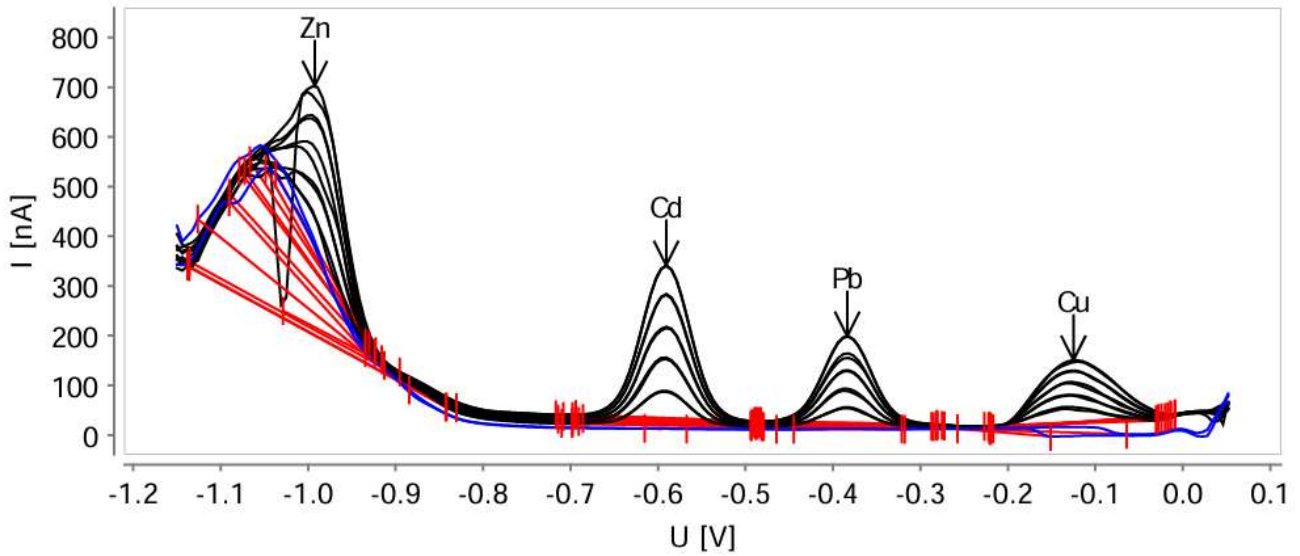


Figure 2.1: Sample DC voltammogram for Noodle ash sample A solution and 5 standards

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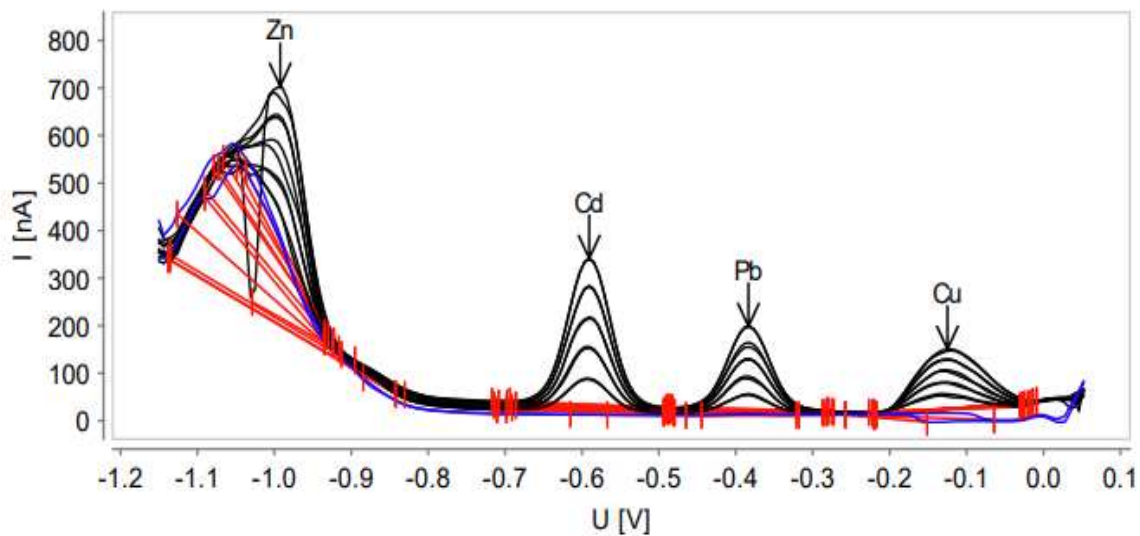


Figure 2.2: Sample DC voltammogram for Noodle ash sample B solution and 5 standards

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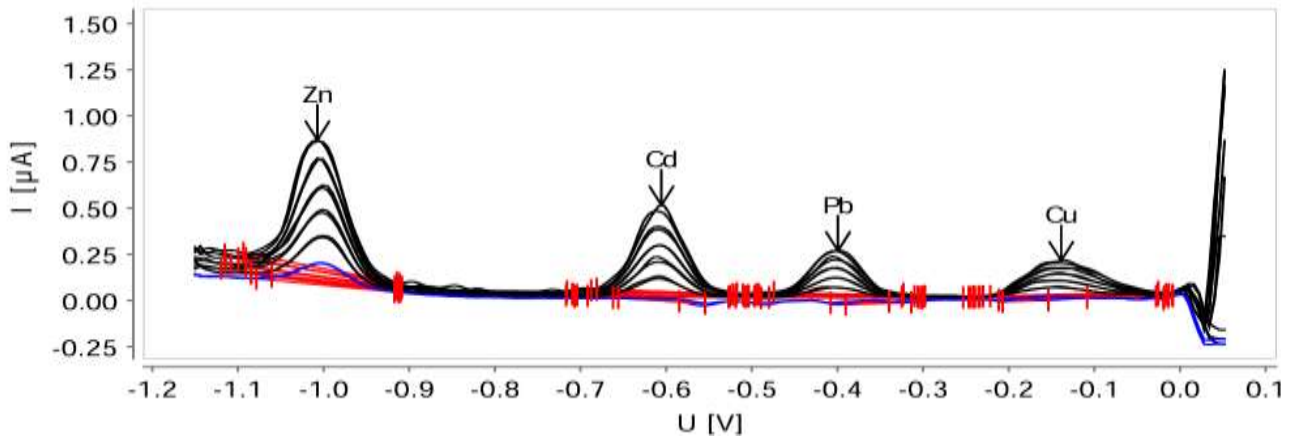


Figure 2.3: Sample DC voltammogram for Noodle ash sample C solution and 5 standards

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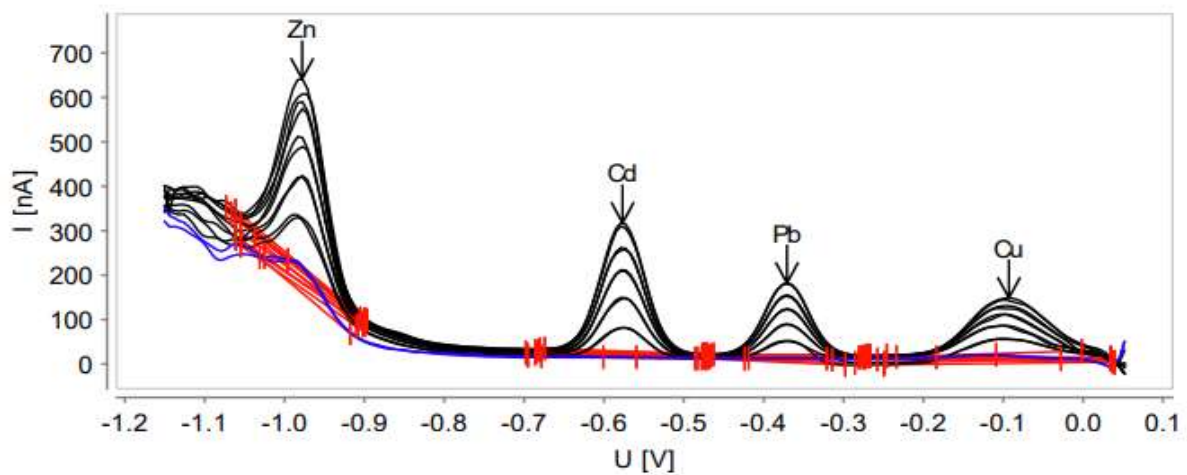


Figure 2.4: Sample DC voltammogram for Pasta ash sample A solution and 5 standards

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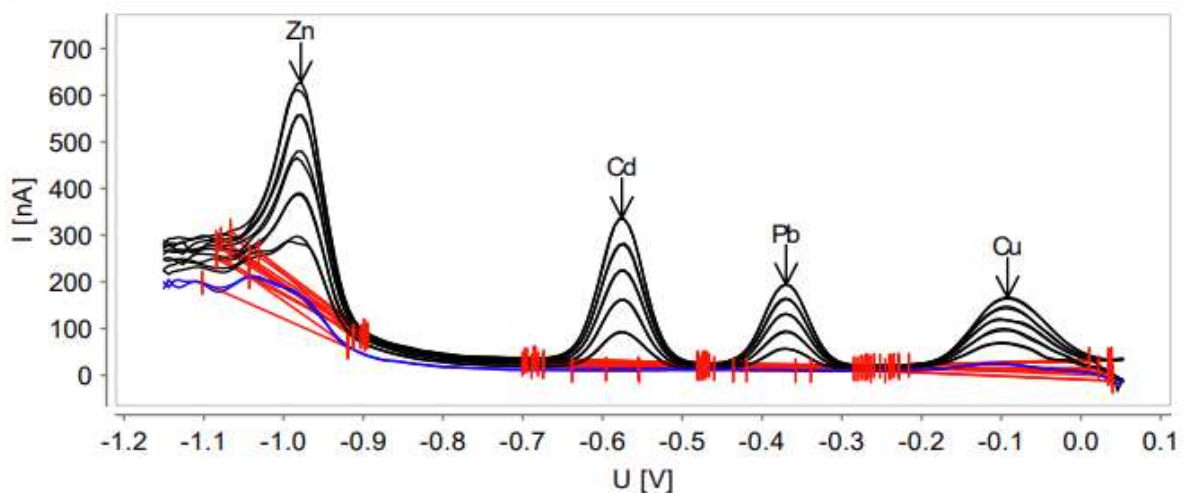


Figure 2.5: Sample DC voltammogram for Pasta ash sample B solution and 5 standards

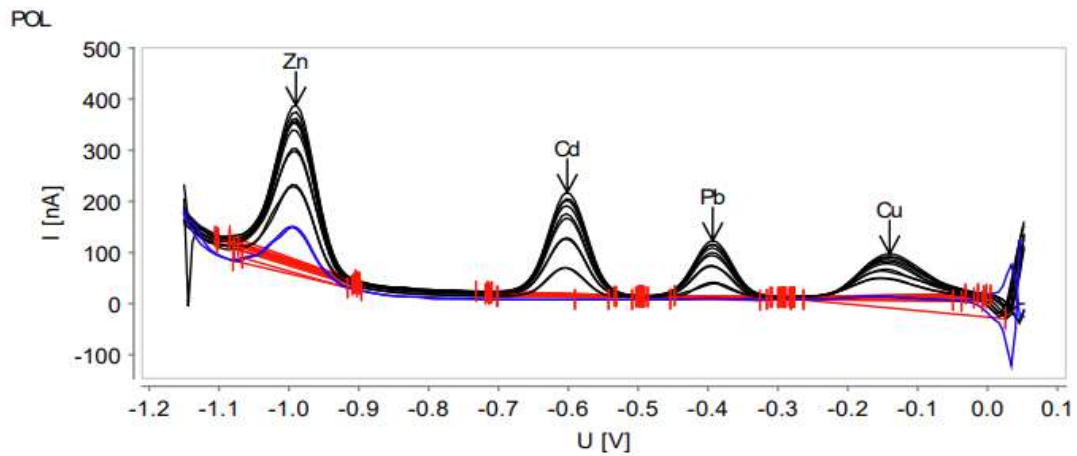


Figure 2.6: Sample DC voltammogram for Oats ash sample A solution and 5 standards

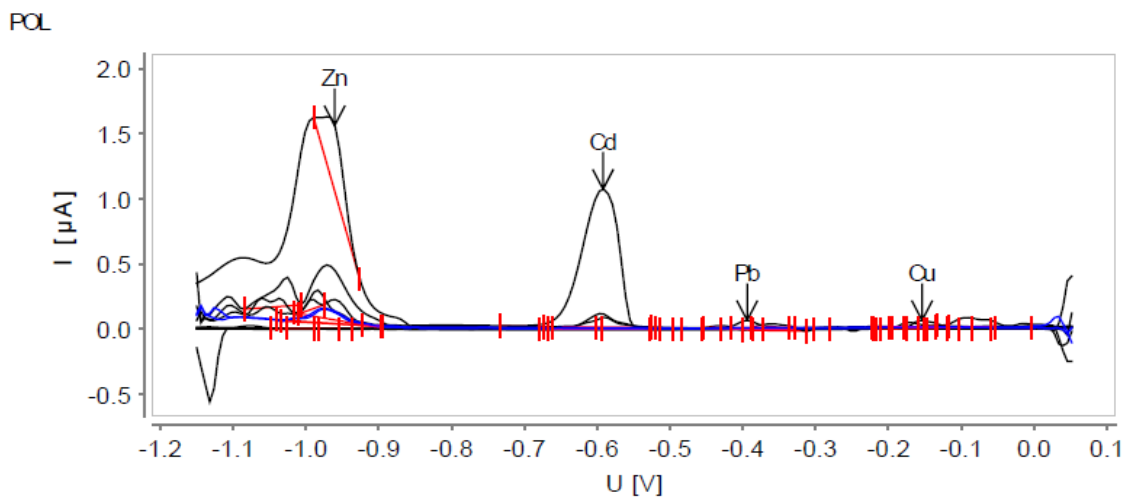


Figure 2.7: Sample DC voltammogram for Oats ash sample B solution and 5 standards

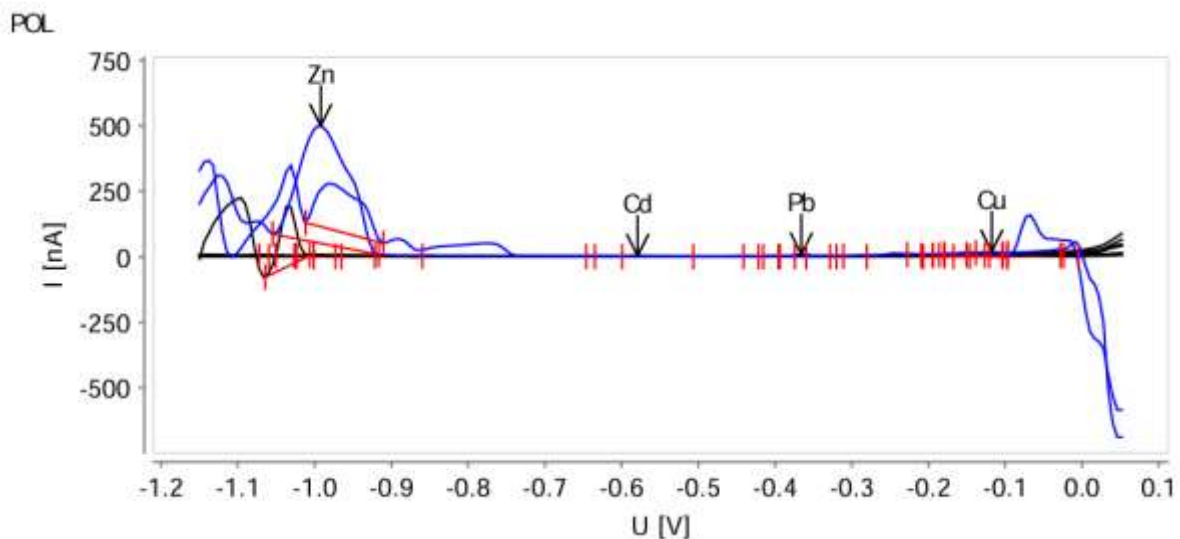


Figure 2.8: Sample DC voltammogram for Soup ash sample A solution and 5 standards

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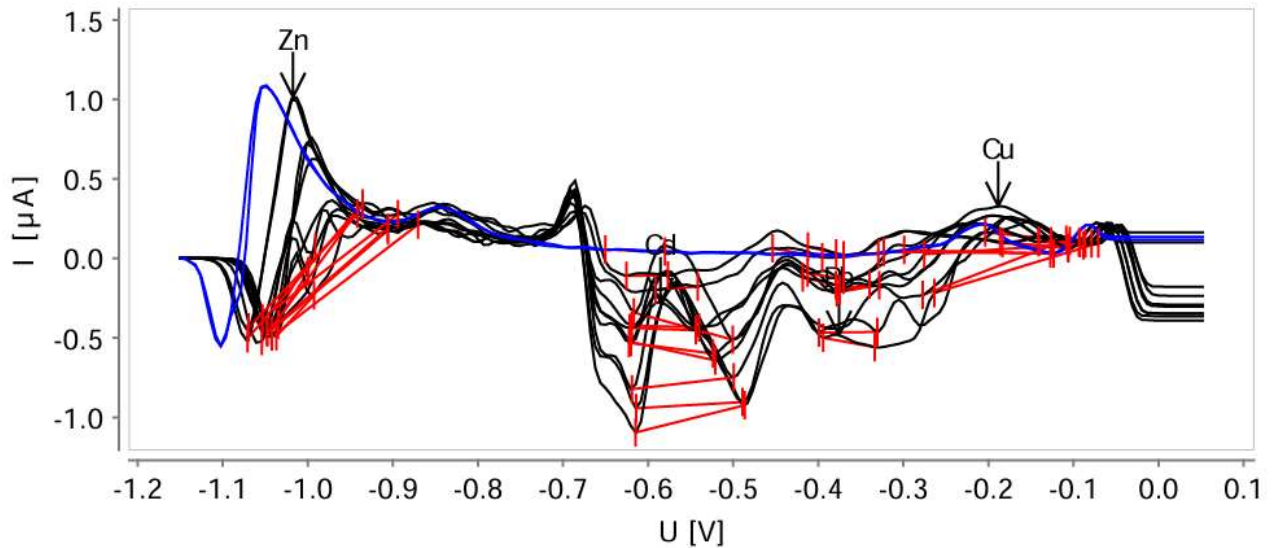


Figure 2.9: Sample DC voltammogram for Soup ash sample B solution and 5 standards

5. RESULTS:

The results of Voltammetric measurements carried out as per the standard addition method shown in the Figure 1.1 to Figure 1.9 and Figure 2.1 to Figure 2.9 are analyzed by plotting the graphs of peak current against the concentration of standard added. The table of volume of standard added and the resulting concentrations in the total solution in the voltammetric cell are given in Table 2.

Table 2: The volume of 50 ppm standard metal ion solution to the 20 cm³ experimental solution and the resultant concentration of metals in the total cell solution.

Solution no.	1	2	3	4	5
Vol. of 50 ppm solution added to 20 cm ³ cell solution	0.5	1.0	1.5	2.0	2.5
Conc. in experiment cell w.r.t std added	1.22	2.38	3.49	4.55	5.56

Representative Standard addition graphs:

Few of these graphs are given below as reference in Figure 3.1 to Figure 3.18, and the results of all measurements are given in Table 3 and Table 4.

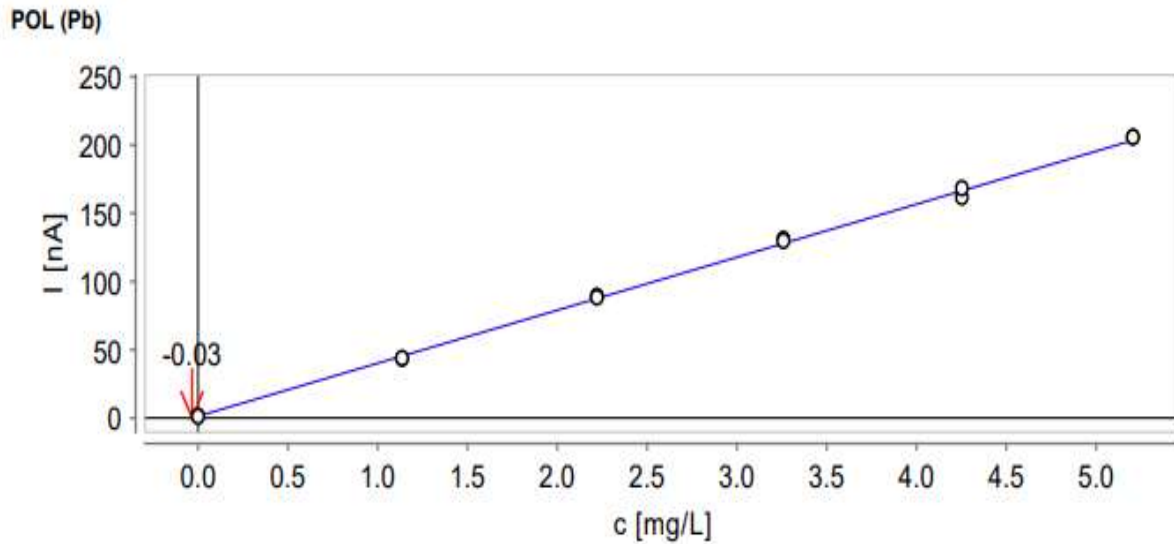


Figure 3.1: Standard addition plot of peak current against concentration of standard added for amount of Lead (Pb) in the Noodle Sample A by digestive method.

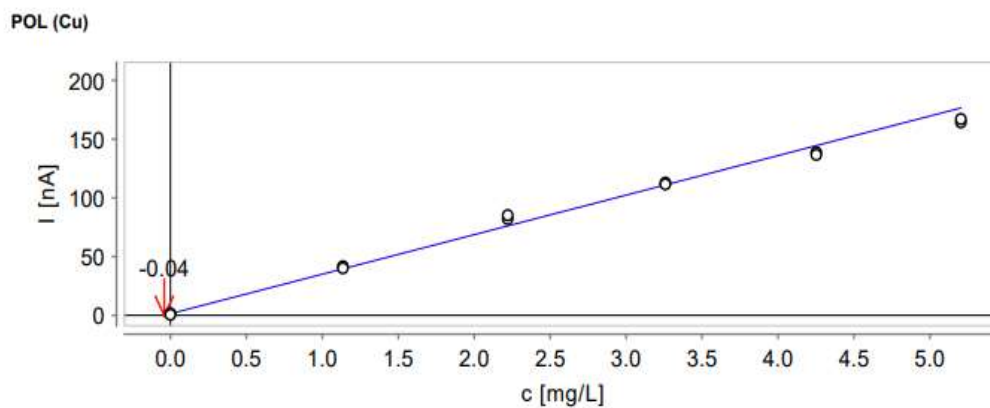


Figure 3.2: Standard addition plot of peak current against concentration of standard added for amount of Copper (Cu) in the Noodle Sample A by digestive method

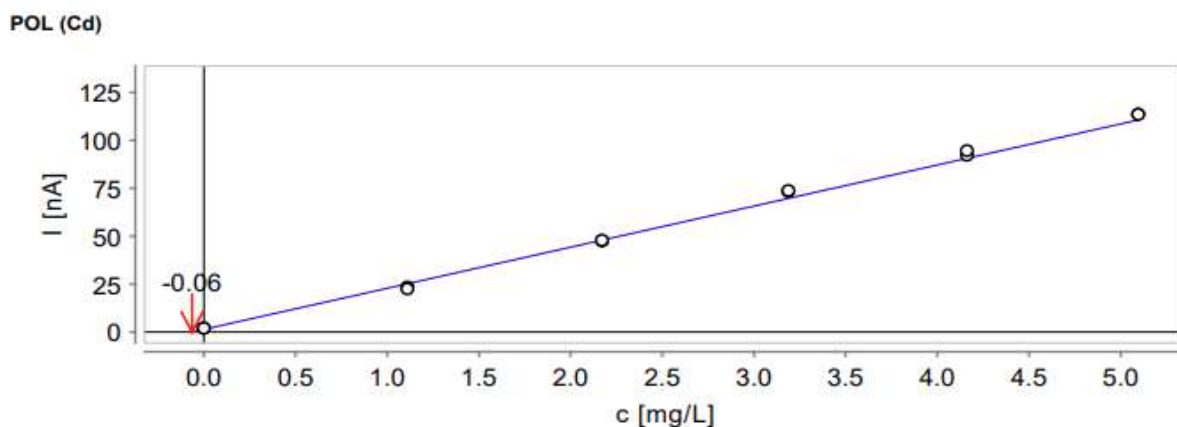


Figure 3.3: Standard addition plot of peak current against concentration of standard added for amount of Cadmium (Cd) in the Pasta Sample A by digestive method.

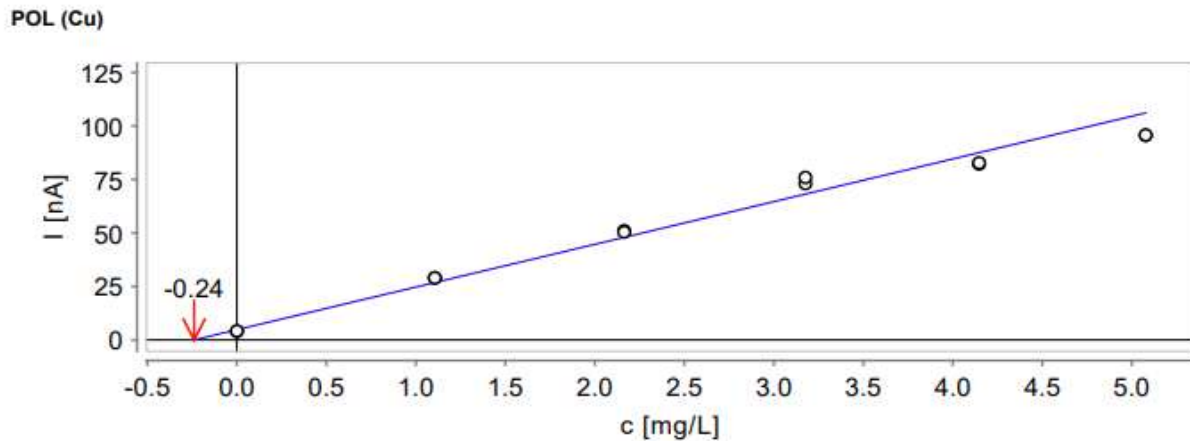


Figure 3.4: Standard addition plot of peak current against concentration of standard added for amount of Copper (Cu) in the Pasta Sample A by digestive method.

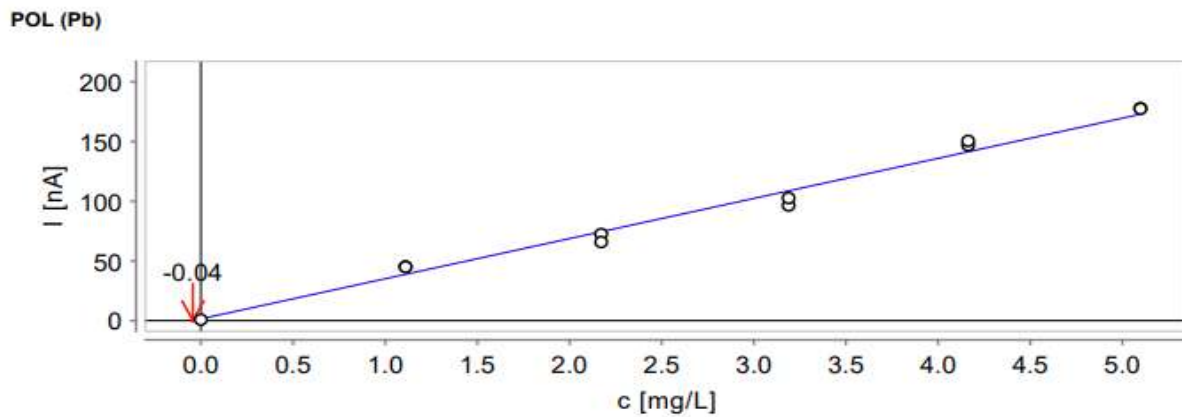


Figure 3.5: Standard addition plot of peak current against concentration of standard added for amount of Lead (Pb) in the Pasta Sample B by digestive method.

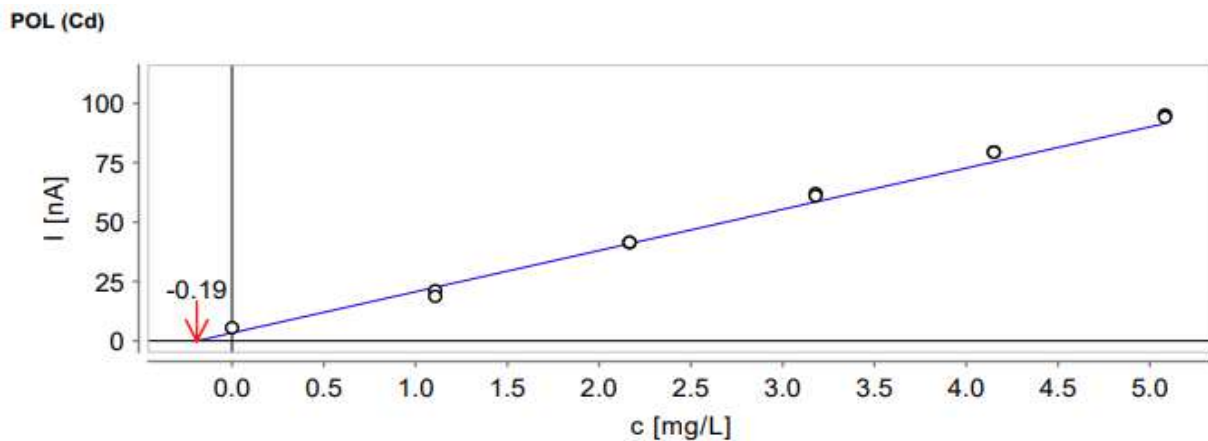


Figure 3.6: Standard addition plot of peak current against concentration of standard added for amount of Cadmium (Cd) in the Oats Sample A by digestive method.

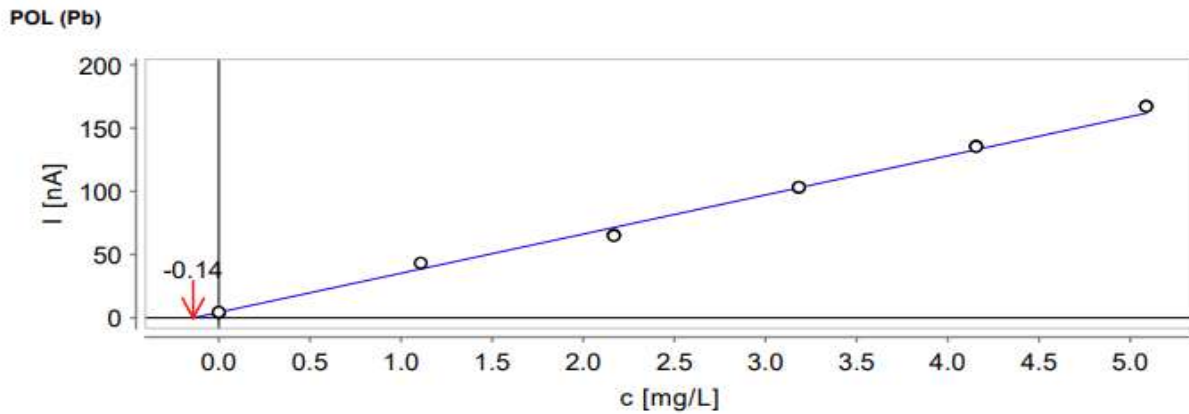


Figure 3.7: Standard addition plot of peak current against concentration of standard added for amount of Lead (Pb) in the Oats Sample A by digestive method.

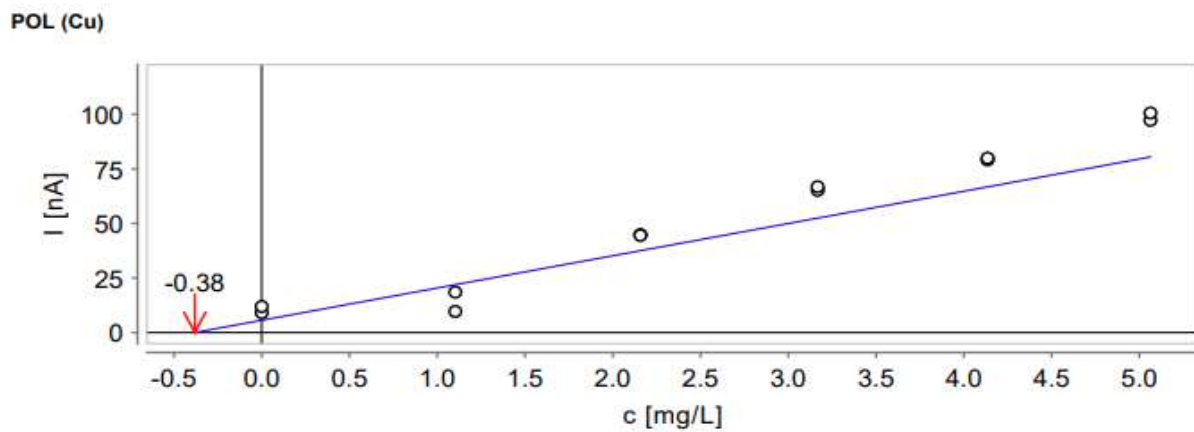


Figure 3.8: Standard addition plot of peak current against concentration of standard added for amount of Copper (Cu) in the Oats Sample A by digestive method.

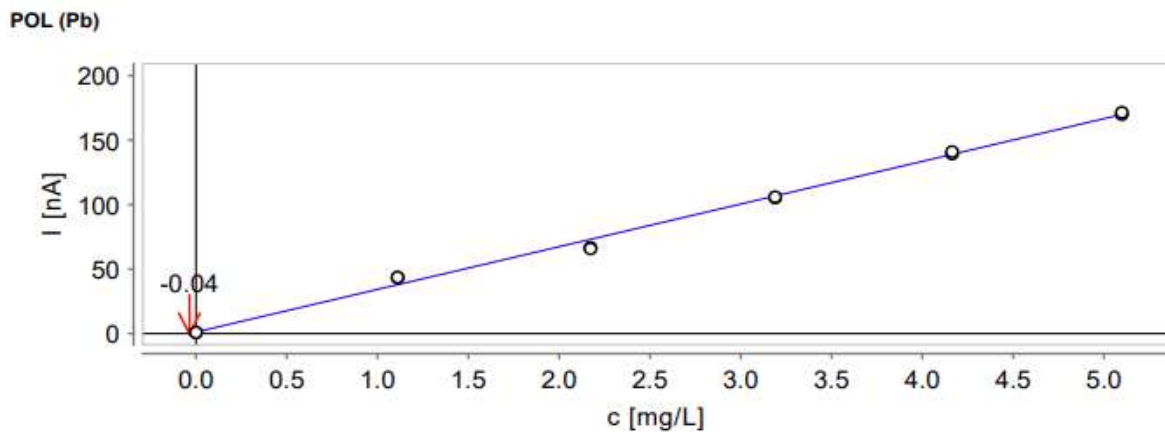


Figure 3.9: Standard addition plot of peak current against concentration of standard added for amount of Lead (Pb) in the Oats Sample B by digestive method.

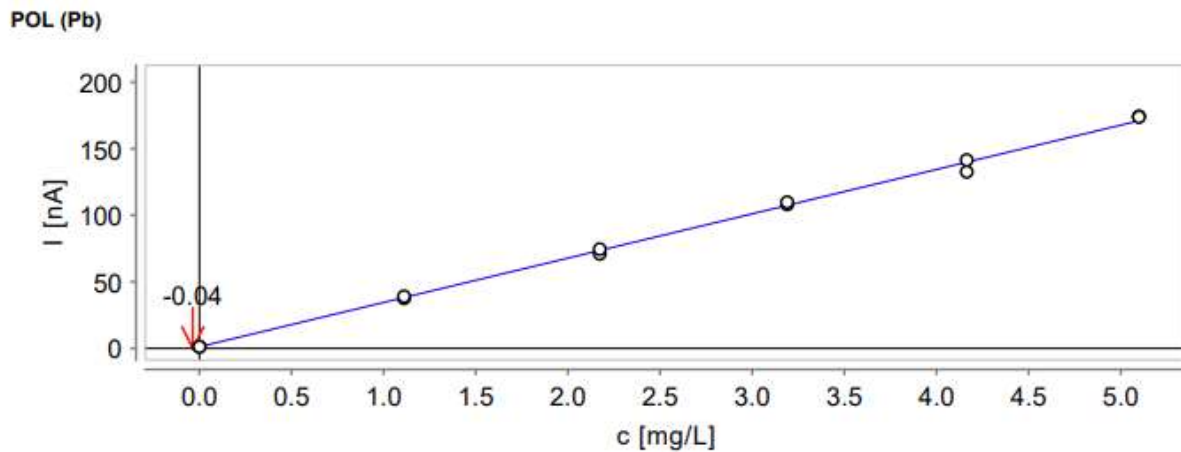


Figure 3.10: Standard addition plot of peak current against concentration of standard added for amount of Lead (Pb) in the Noodles Sample A by ashing method.

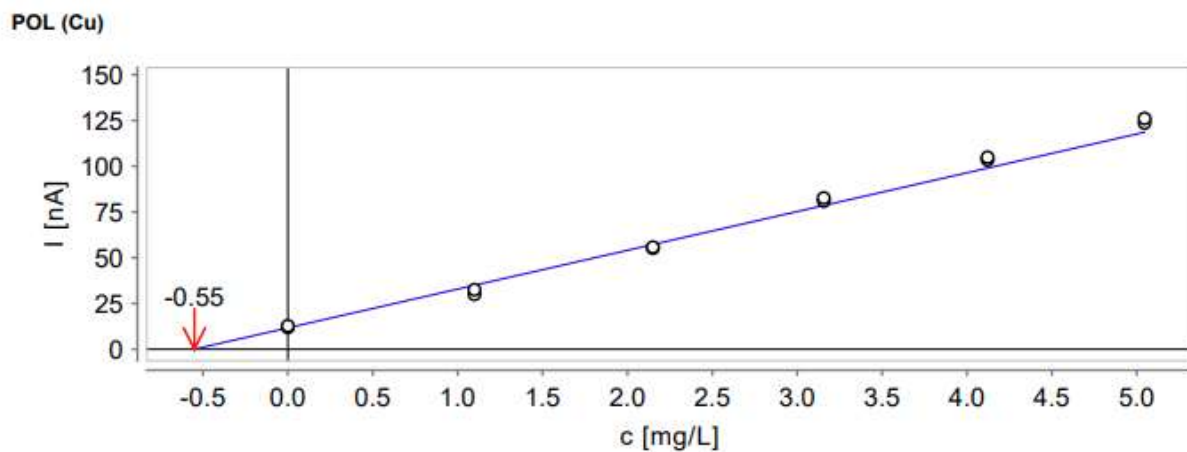


Figure 3.11: Standard addition plot of peak current against concentration of standard added for amount of Copper (Cu) in the Noodles Sample A by ashing method.

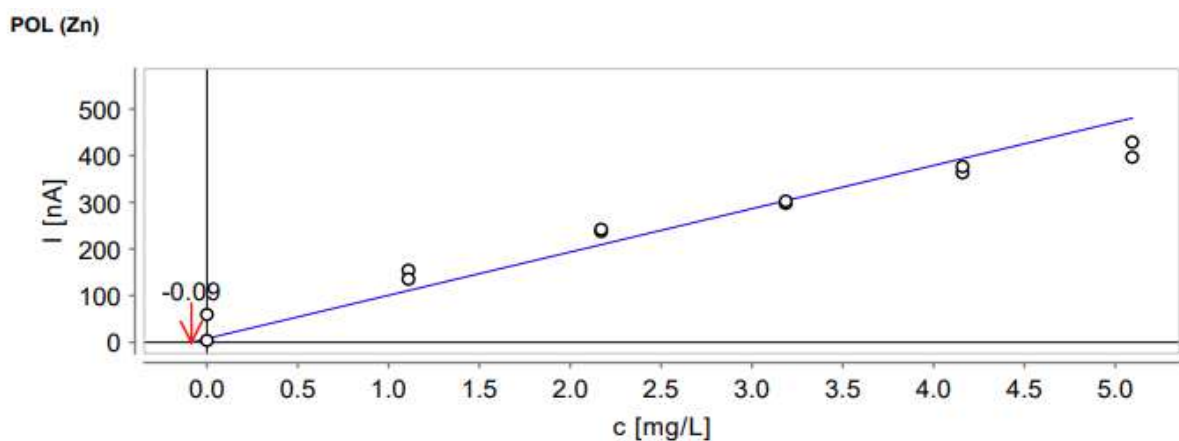


Figure 3.12: Standard addition plot of peak current against concentration of standard added for amount of Zinc (Zn) in the Pasta Sample A by ashing method.

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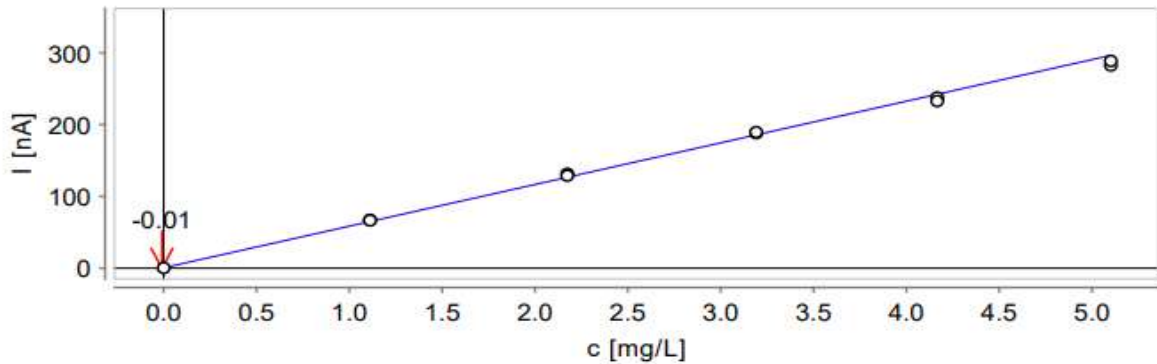


Figure 3.13: Standard addition plot of peak current against concentration of standard added for amount of Cadmium (Cd) in the Pasta Sample A by ashing method.

POL (Cu)

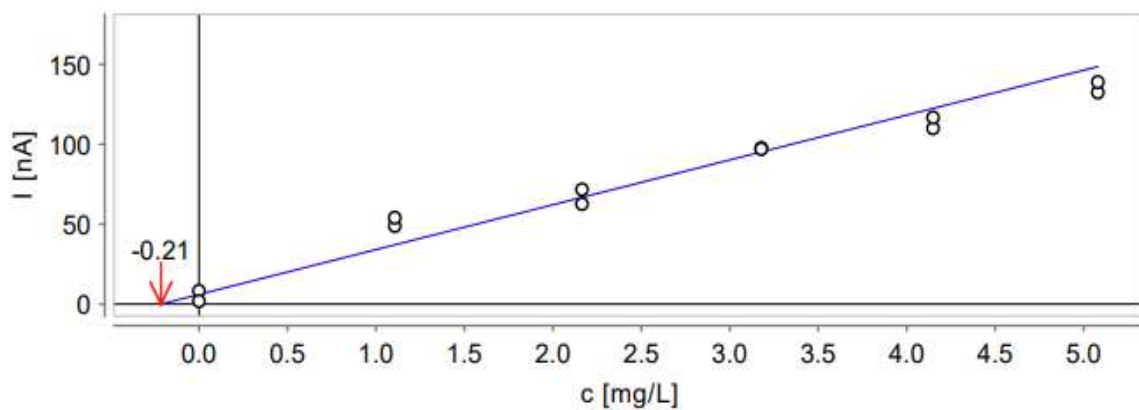


Figure 3.14: Standard addition plot of peak current against concentration of standard added for amount of Copper (Cu) in the Pasta Sample A by ashing method.

POL (Pb)

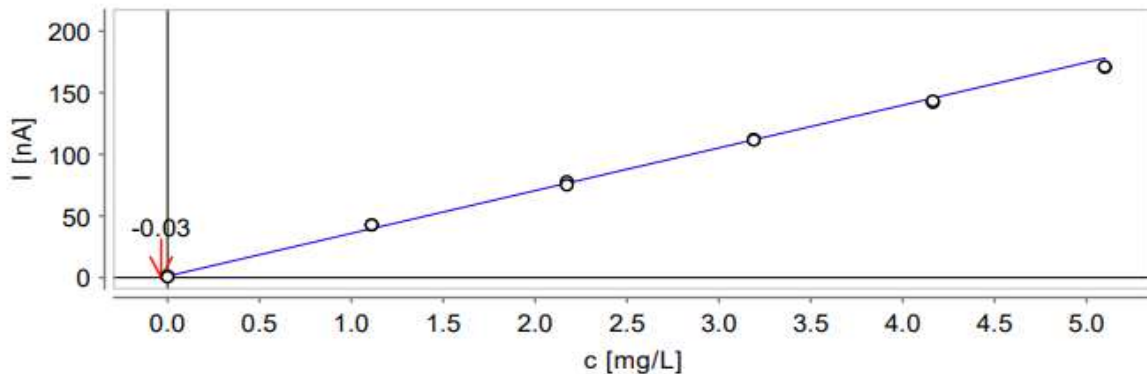


Figure 3.15: Standard addition plot of peak current against concentration of standard added for amount of Lead (Pb) in the Pasta Sample B by ashing method.

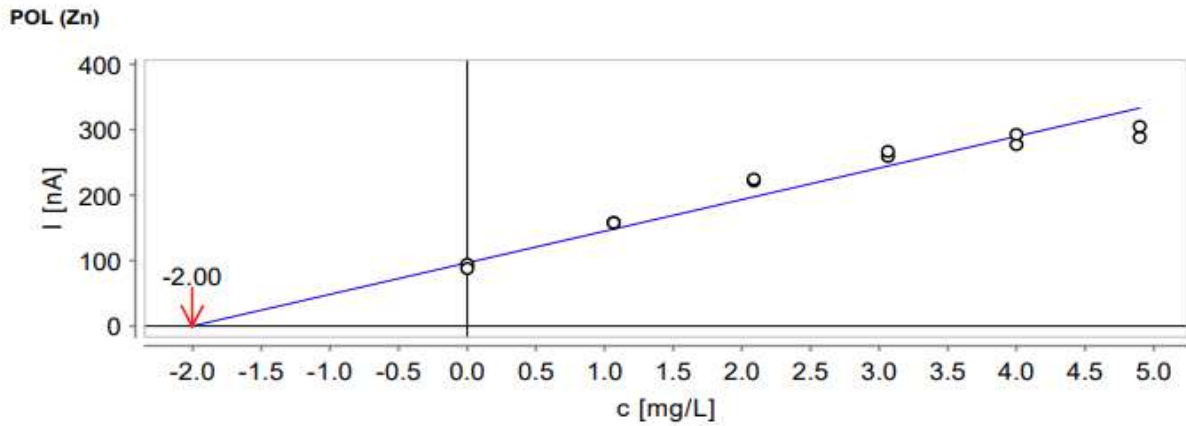


Figure 3.16: Standard addition plot of peak current against concentration of standard added for amount of Zinc (Zn) in the Oats Sample A by ashing method.

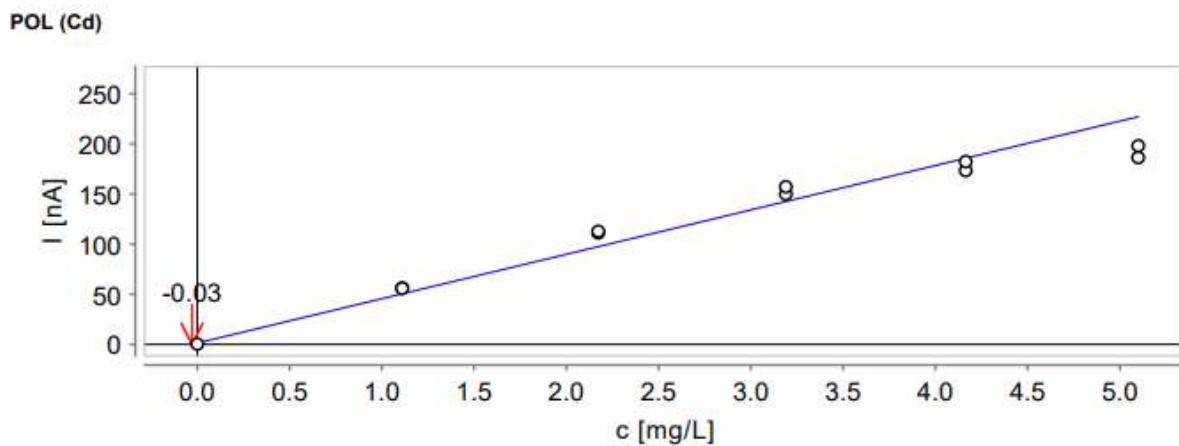


Figure 3.17: Standard addition plot of peak current against concentration of standard added for amount of Cadmium (Cd) in the Oats Sample A by ashing method.

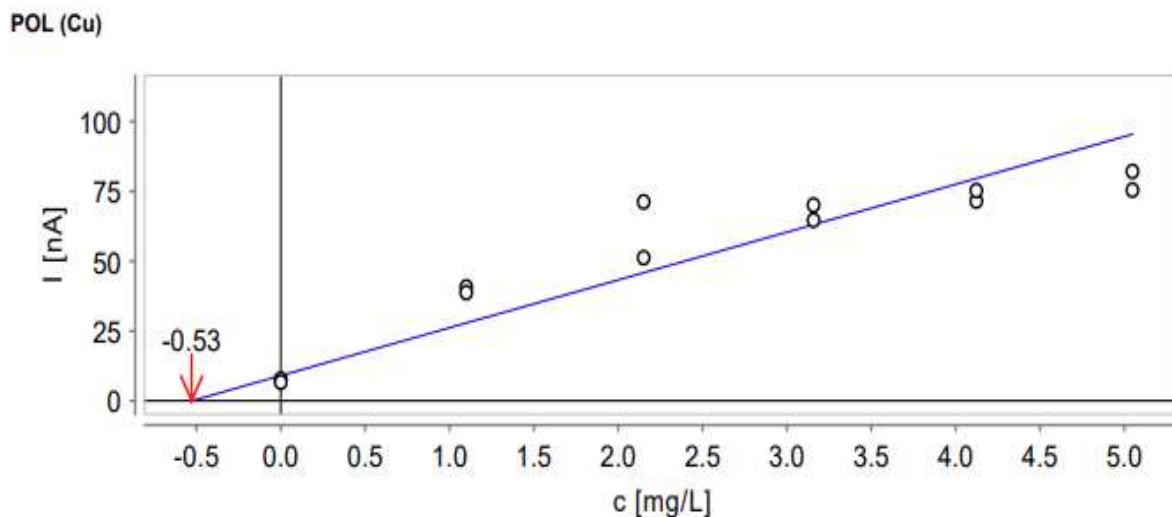


Figure 3.18: Standard addition plot of peak current against concentration of standard added for amount of Copper (Cu) in the Oats Sample A by ashing method.

Table 3: Amount of Heavy metals present in the ready to eat food sample detected by digestive conditions method.

Sample	Metal [PPM that is mg / kg of the sample]			
	Zn	Cd	Pb	Cu
Noodles A	BDL	BDL	0.6	0.8
Noodles B	BDL	BDL	7.2	8.8
Noodles C	BDL	4.0	2.8	BDL
Pasta A	BDL	1.2	BDL	4.8
Pasta B	BDL	BDL	0.8	BDL
Oats A	BDL	3.8	2.8	7.6
Oats B	BDL	BDL	0.8	BDL
Soup A	BDL	0.4	1.2	BDL
Soup B	BDL	BDL	0.4	BDL

[BDL: Found below detection limit of the instrument]

Table 4: Amount of Heavy metals present in the food sample detected by ashing method

Sample	Metal [PPM that is mg / kg of the sample]			
	Zn	Cd	Pb	Cu
Noodles A	BDL	0.1	0.4	5.5
Noodles B	9.2	0.1	0.2	3.4
Noodles C	9.3	1.3	2.8	4.2
Pasta A	0.9	0.1	0.6	2.1
Pasta B	5.6	0.1	0.3	11.5
Oats A	20	0.3	1.1	5.3
Oats B	BDL	BDL	BDL	BDL
Soup A	BDL	BDL	BDL	BDL
Soup B	BDL	BDL	6.6	BDL

[BDL: Found below detection limit of the instrument]

6. CONCLUSION:

The results obtained in this project indicate the presence of heavy / hazardous metals in almost all the ready to eat products. The presence of heavy / hazardous metals is well within the limits prescribed in various reviews [8, 9]. The amount of Zinc is below the detection limit in all samples tested by digestive method, but was detected by ashing method in Pasta sample A (0.9 ppm) and surprisingly very high in Oats sample A (20 ppm). Copper is also found to be above 5 ppm in several samples by both the methods.

Oats sample A showed the highest presence of all metals and it is observed by both the methods this indicates that the digestive condition method developed is in good agreement with the very commonly used ashing method.

The results obtained here in this project indicate that the total metal content of all ready to eat food samples are in partially soluble form in the digestive juices. The concentration detected by ashing method is more in almost every case indicating that these metals, though they are present in the ready to eat food products, will not get absorbed in the human system as they are not in soluble form in digestive conditions. Hence the ready to eat food products can be considered safe for consumption even by the kids.

8. ACKNOWLEDGEMENT

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9. https://www.google.com/search?q=metal+limits+in+pasta+packaged+food&scaesv=6449538529ffda2f&rlz=1C1PRFE_enIN821IN821&biw=1366&bih=633&ei=N_AAZ77oK_ac4EPwJnkAU&ved=0ahUKEwj37iL4faIAxV2zjgGHUdMGVIQ4dUDCA8&uact=5&oq=metal+limits+in+pasta+packaged+food&gs_l=lp=Egxnd3Mtd2l6LXNlcnAi21ldGFsIGxpbWl0cyBpbiBwYXN0YSBwYWN-

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