

RESEARCH ARTICLE

Determination of Zinc (II) by Cloud point Extraction with Solvent extraction in Different Samples

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ABSTRACT

Sensitive solvent extraction method with Cloud point Extraction method used for determination of Zinc (II) by organic reagent N-(5,6-dimethoxypyrimidin-4-yl)-4-((4,5-diphenyl-1H-imidazol-2-yl)diazanyl)benzenesulfonamide and studies ion pair association complex for (NDPDBS) and Zn^{2+} in acidic HCl medium shows the wave length of maximum absorbance was $\lambda_{\text{max}}=294\text{nm}$, all studies about optimum condition for giving higher extraction method where, $\text{pH}=9$ as optimum in presence $50\text{ }\mu\text{g Zn}^{2+}$ in 10 mL aqueous solution, 0.5 mL surfactant Triton X- 100, and heating temperature at $90\text{ }^{\circ}\text{C}$ For 15 min , $1\times 10^{-4}\text{ M}$ (NDPDBS), The stoichiometry show up the composition of ion pair association complex extracted was $1:1$, and addition to other studies such as effect of interferences, electrolyte and else, Statistical values were also recorded for $\text{LOD} = 0.13904674\text{ ppm}$, $\text{LOQ} = 0.42135377\text{ ppm}$ and Sandell's sensitivity $(2.33\times 10^{-3}\text{ }\mu\text{g/cm}^2)$.

Keyword: Determination of Zinc (II) by Cloud point extraction method, using organic reagent (NDPDBS)

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1. Introduction

The use of green technologies is a key focus in the development of modern analytical chemistry^[1]. Currently, as environmental awareness increases and many industries emphasize using more environmentally friendly methods. Methods in line with green chemistry principles are gaining popularity in the chemical industry and Cloud point extraction (CPE) fits perfectly into this trend.^[2] This process is considered an environmentally friendly extraction procedure because it uses small amounts of toxic organic solvents^[3,4]. The basic method for extracting the cloud point relies on the fundamental principle of using a non-ionic surfactant that causes micelles to form in aqueous solutions when heated to a specific temperature^[5,6]. Many variables, including the type and concentration of surfactants, as well as the pH of the sample solution, time to reach equilibrium, interfering substances, and electrolytes, affect the quality of extraction of the target compounds^[7]. Zinc is regarded as the most significant tracer for human health, because it acts as a catalyst and controller in several metabolic systems, including DNA synthesis, gene activity, signal transduction, and hormone production. The risk of nutritional inadequacy now affects at least 18% of the global population. However and one of the most common micronutrient deficiencies in infants and pregnant and lactating mothers and adults globally is zinc deficiency^[8]. And Zinc deficiency is mainly attributed to nutritional factors, but the aggravation of the problem is closely related to the presence of toxic metals accumulated in water bodies. And the danger of

these metals lies in the complexity of their specific oxidation and reduction reactions, as well as their chemical stability and high resistance to decomposition, which leads to serious health risks as a result of interference with biological processes ^[9]. Additionally, excessive levels of zinc, can have adverse effects on human health and such as abnormalities in the metabolism of energy and increased oxidative damage, inhibited growth, impaired resistant function, pregnancy complications, loss of weight, and sickness ^[10]. Coordination compounds can be defined as those complexes resulting from the chemical bonding of a central metal ion (or metal atom) with a number of organic and inorganic ions or molecules that possess non-sharing, bondable electron pairs called ligands. The bonding between the organic reagent molecule and the metal ion through two or more sites simultaneously leads to the formation of a stable ring structure known as a chelating complex ^[11-15]. This type of complex (chelate) requires the bonding of the metal ion and the negatively charged organic reagent molecule, such that the bonding results in a neutral complex, which is the chelate complex. This complex contains the coordination bonds between the positive metal ion and the coordination sites in the organic reagent molecule. The organic reagent may contain two or more coordination sites, which leads to the formation of a ring bond that increases the stability of the complex formed and extracted according to the principle of solvent extraction to the organic phase. Most metal ions have coordination sites that are ready for the bonding process with the electron pairs possessed by the organic reagents, thus forming chelate complexes ^[16]. Sulfadoxine has been used to estimate zinc, and a literature review has revealed various techniques for zinc estimation, such as spectrophotometry ^[17,18]. In general, UV-Vis spectrophotometry is the most widely used technique due to its simplicity sensitivity, and cost-effectiveness therefore, developing selective and sensitive methods using visible spectrophotometry is of paramount importance ^[19-24]. Several studies have applied the cloud point extraction (CPE) technique in combination with the spectroscopic method, to separate and determination a number of elements in different environmental models, and results were characterized by accuracy and high sensitivity ^[25]. Spectrophotometric study of Zn (II) and Co (II) extracted according to onium systems from acidic HCl Solution by (2,4-dimethyl pentan-3-one) as onium complex^[26].

2. Experimental

2.1. Reagents

For the preparation of the solutions in this study, distilled water was used in all practical experiments, and the chemicals used were obtained from accredited companies, stock solution of Zn (II) in concentration 1mg/mL was proposed by dissolved g of ZnCl₂ in 100 mL hydrochloric acid distilled water in volumetric flask, other solutions of Zn (II) proposed by dilution with distilled water in suitable volumetric flasks. as well as prepared organic reagent solution in 1×10⁻⁴ M by dissolved g of (NDPDBS) in 100 mL distilled water, and other solutions proposed by dilution with distilled water.

2.2. Instrumentation

For spectrophotometric studies and absorbance measurements used double beam Uv-vis, Biochrom type (80-700-11- Libra s60) from Japan and exact weight used balance A&D (DOOL,CE, HR 200) japan, for heating the solutions used Water bath, (Hamburg - 90), England, and PH-meter (WTW Listed / 8693, Laboratory Equipment, E163694, CE, in Germanu).

2.3. Preparation of organic reagent (DNBDIBS)

The newly synthesized organic reagent was prepared N-(5,6-dimethoxypyrimidin-4-yl)-4-((4,5-diphenyl-1H-imidazol-2-yl)diazenyl)benzenesulfonamide (NDPDBS), and the corresponding synthesis equations are shown below Fig(1).

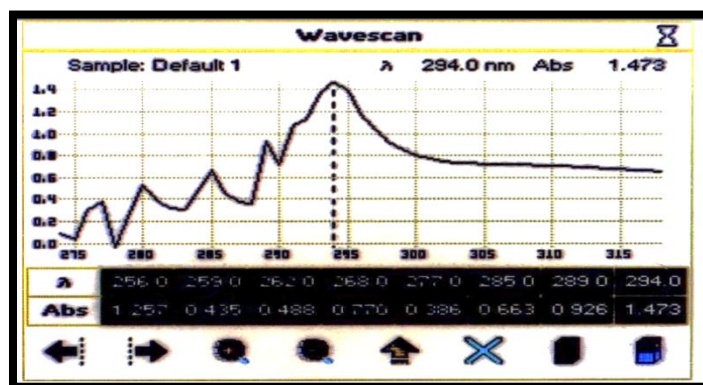


Figure 2. UV-Vis absorption spectrum for ion pair association complex of Zn^{2+} with (NDPDBS)

The spectrum shows up the wavelength for maximum absorbance was $\lambda_{\text{max}} = 294 \text{ nm}$, used this wavelength to measure the absorbance of alcoholic solutions in subsequent experiments.

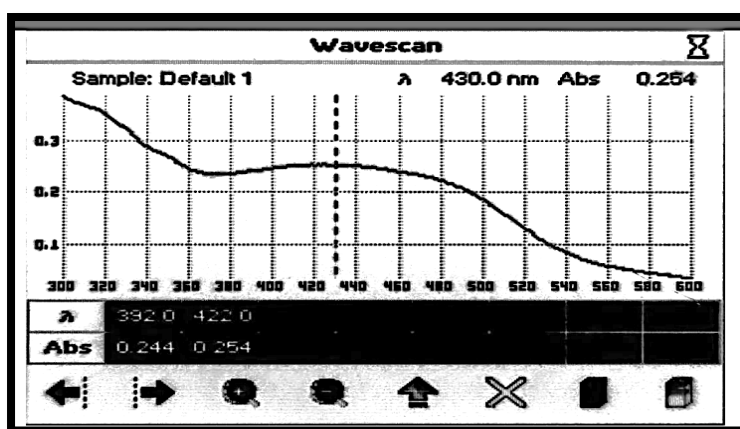


Figure 3. UV-Vis absorption spectrum for (NDPDBS)

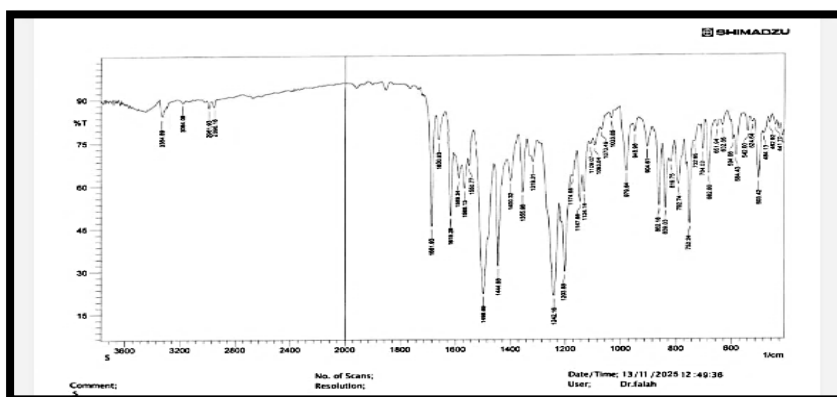


Figure 4. IR spectrum for (NDPDBS) ligand

3.1.1. Effect of PH

Prepared 10 mL aqueous solution contain $50 \mu\text{g Zn}^{2+}$ ion, $1 \times 10^{-4} \text{ M}$ (NDPDBS), at different pH (6-11), and 0.5 mL surfactant Triton-X-100, heat these solutions in electric water bath at 90°C for 15 min., until formation CPL, then separation CPL from aqueous solution and dissolved in 5 mL ethanol, and complete the work as in general method, the results were as in Figs 6, 7.

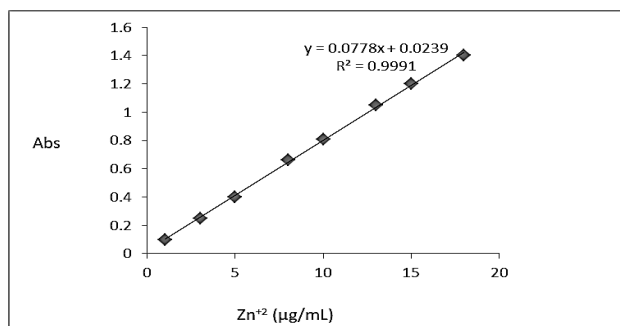


Figure 5. Calibration curve for determination Zn^{2+} ion in aqueous solution by dithizone spectrophotometric method

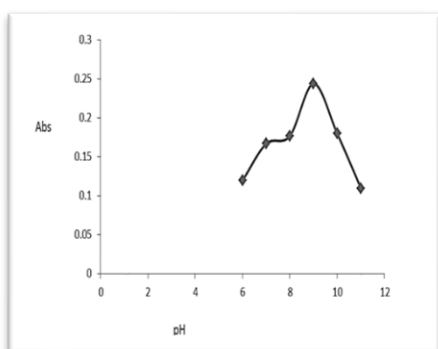


Figure 6. Effect of pH formation and stability of ion pair association complex

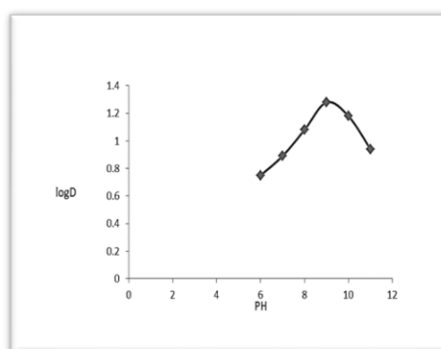


Figure 7. Effect of pH concentration on extraction efficiency and D-values

The results appear pH=9 was the optimum, give higher extraction efficiency and D-value to formation ion association complex and distribution to cloud point layer, pH values lower than the optimum (acidic medium) reduce the extraction efficiency due to the protonation, at pH values higher than the optimum, the distribution ratio (D) decreases as a result of the formation of more stable Zn^{2+} with OH^- species, which are not extractable by the extractant. Consequently, the extraction efficiency also decreases.

3.1.2. Effect of Surfactant Volume

A series of 10mL aqueous solutions was prepared each one contain 50 μg Zn^{2+} , pH=9, and 1×10^{-4} M in (NDPDBS) and different volume of surfactant Triton X-100 (0.1-1)mL, then heat all these solutions in electrostatic water bath at 90°C for 15 min to formation cloud point layer CPL, and complete the work as in general method the results were as in Fig 8,9

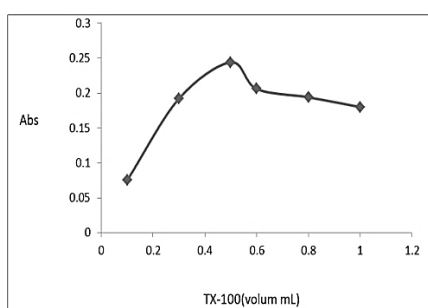


Figure 8. Effect of surfactant volume on Cloud point layer formation

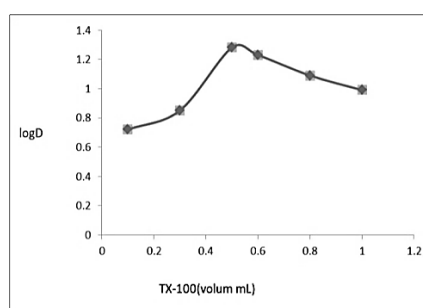


Figure 9. Effect of surfactant volume on extraction efficiency and D-value

The results show up 0.5 mL of surfactant was the optimum volume gave higher extraction efficiency because this volume give best thermodynamic and kinetic equilibrium for aggregation micelles to formation good Cloud point layer, with smaller volume and higher density, which is have large surface area to accommodating large amount of ion association complex extracted.

3.1.3. Temperature Effect

10 mL aqueous solutions was prepared, contain 50 μg Zn^{2+} ion, $\text{pH}=9$, and $1 \times 10^{-4} \text{M}$ in (NDPDBS) and 0.5 mL of surfactant Triton X-100, at different temperature (70-95) $^{\circ}\text{C}$ for 15 min, until formation cloud point layer CPL, and complete the work as in general method, the results were as in Fig 10,11

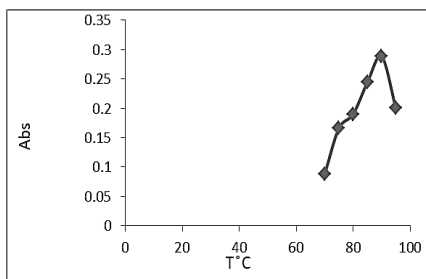


Figure 10. Effect of temperature On cloud point formation

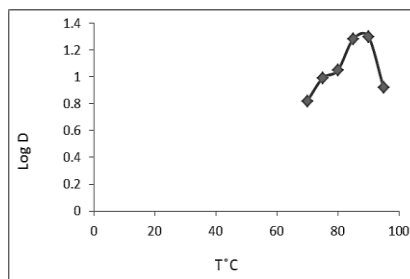


Figure 11. Effect of temperature on extraction efficiency and D-value

The results appear 90 $^{\circ}\text{C}$ was the optimum temperature gave higher extraction efficiency. After calculated extraction constant K_{ex} from D-values by thermodynamic relation below the results clear in Fig(12).

$$K_{\text{ex}} = \frac{D}{[\text{zn}^{+2}]_{\text{eq}}[\text{DNBDIBS}]_{\text{eq}}}$$

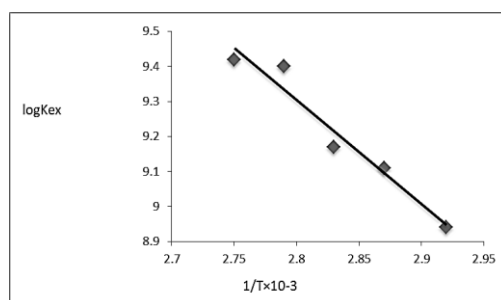


Figure 12. Effect of Temperature on extraction constant

From the slope of the straight line in Fig (12) the thermodynamic were calculated data in table(1).

Table 1. The thermodynamic values (ΔS_{ex} , ΔG_{ex} , ΔH_{ex})

ΔH_{ex}	ΔG_{ex}	ΔS_{ex}
45.953 kJmol $^{-1}$	-65.43 kJmol $^{-1}$	+306.840 kJmol $^{-1}$

3.1.4. Effect of heating time

Prepared many 10 mL aqueous solution contains 50 μg Zn^{2+} ion, $\text{pH}=9$, and $1 \times 10^{-4} \text{M}$ in (NDPDBS) and 0.5 mL of surfactant Triton X-100, at temperature 90 $^{\circ}\text{C}$, for different heating time (5-25) min, and complete as in general method, the results were as in Fig 13,14

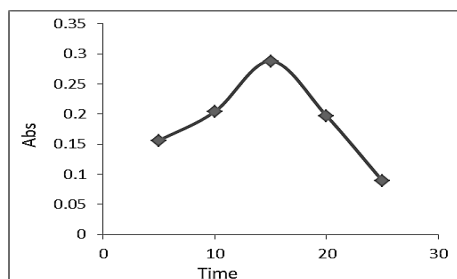


Figure 13. Effect of heating time on quality of cloud point layer

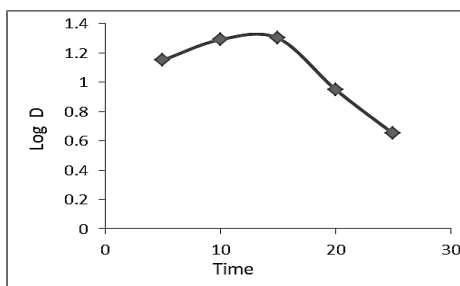


Figure 14. Effect of heating time on extraction efficiency and D-value

The results show up 15 minutes was the optimum time of heating to give higher extraction efficiency. Because heating time represent the kinetic side of extraction method help to good aggregation micelles with complete dehydration to formation CPL with larger surface area to accommodating large amount of complex extracted.

3.1.5. Stoichiometry

In order to determine the more probable structure of ion pair association complex extracted for Zn^{2+} ion with (NDPDBS), used two spectrophotometric methods: Slope analysis metal and Slope ratio method the solutions treated according to general method , the results as in Figs 15,16,17.

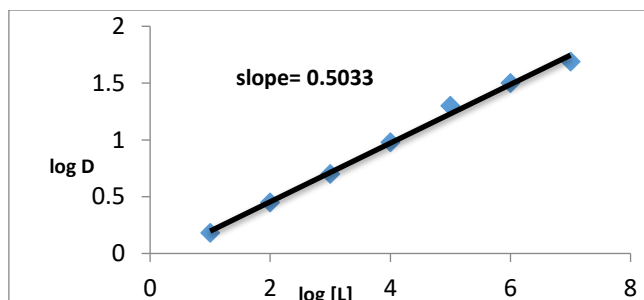


Figure 15. Effect of (NDPDBS) concentration of extraction efficiency and D-values

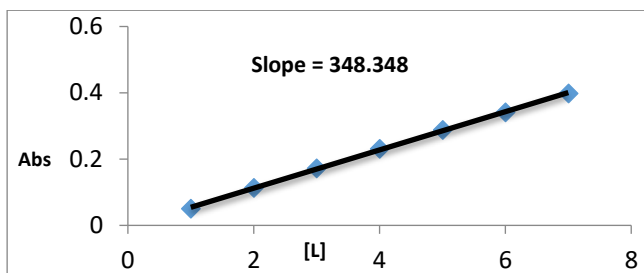


Figure 16. Effect of (NDPDBS) Conc. On formation and stability of ion pair association complete

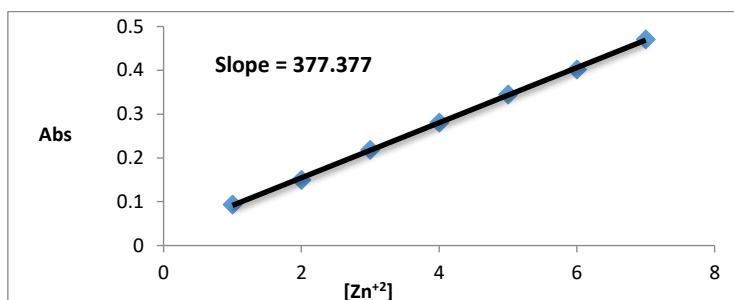


Figure 17. Effect of Zn^{2+} concentrations on formation and stability of ion pair association complete

$$\text{slope ratio} = \frac{348.348}{377.377} = 0.923$$

The slope and slope ratio values lead to the structure of ion pair association complete extracted was 1:1 $[Zn(NDPDBS)]^+Cl^-$

3.1.6. Electrolyte Effect

Through the general method of extraction and at optimum conditions extracted Zn^{2+} from 10mL aqueous solution in exists different electrolyte at different concentrations, the results were as in Table (2).

Table 2. Effect of different conc. for different electrolytes on formations and stability of ion pair association complete extracted on extracted efficiency and D-value

Electrolytic salt	Abs. $\lambda_{\max}=294$	D
MgCl ₂	0.372	46.2
NH ₄ Cl	0.296	26.8
NaCl	0.356	40.7
LiCl	0.395	48
CaCl ₂	0.331	39
KCl	0.325	36
AlCl ₃	0.292	24

The results appear existence electrolyte in aqueous solution with Zn²⁺ ion effected to increase extraction efficiency and D-values, that is mean electrolyte increase chances of formation ion pair association complete, so that distribution it to CPL.

3.1.7. Effect of Interferences

By existence 0.01M of foreign ions in aqueous solution with Zn²⁺ ion, extracted the ion pair association complex formed for Zn²⁺ ion according to general method, the results were as in Table (3).

Table 3. Effect of interference on extraction efficiency 0.01M

Interferences	Abs. $\lambda_{\max}=294$	D
CuCl ₂	0.206	13
CdCl ₂	0.195	9
NiCl ₂	0.243	19.5
CrCl ₃	0.369	22
HgCl ₂	0.184	7

The results shows exists foreign ion in aqueous solution with Zn²⁺ ion effect to decrease extraction efficiency and D-values, so that this decreasing differ with different foreign ion.

3.1.8. Variations Surfactant Kind

According to the used extraction method and at optimum conditions by use 1×10⁻⁴M (NDPDBS) with Different surfactant, extracted Zn²⁺ ion as ion pair association complex and results were as in Table 4.

Table 4. Effect of surfactant kind on extraction efficiency of Zn²⁺ ion

Surfactant Type	Abs. $\lambda_{\max}=294$	D
TritonX-100	0.288	20
Tween 20	0.021	3.55
Tween 40	0.159	7.33
Tween 80	0.203	11.5

The results showed a decrease in the extraction efficiency of the Zn²⁺ ion as ion pair association complex when using Tweens as surfactants. These results indicate the Tweens are not suitable surfactants on extraction of Zn²⁺ ion.

3.2. Spectrophotometric Determination Zinc(II)

Zinc (II) was determined spectrophotometrically in different environmental samples and using an appropriate masking agent, Calibration curve after setting the absorbance versus $\mu\text{g/L}$ for the zinc ion as in Fig. 18.

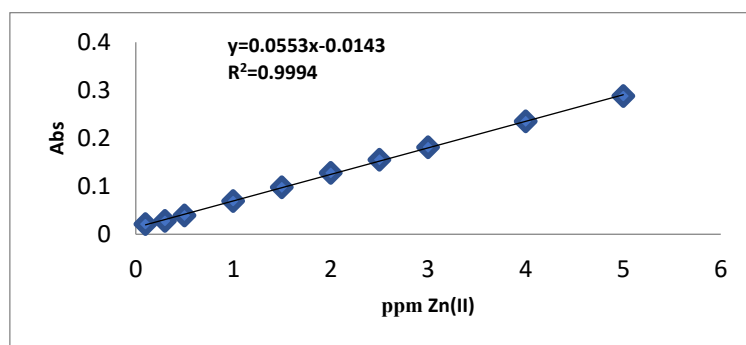


Figure 18. Calibration curve of Zn(II)

Analytical parameters for the calibration curve of the Zn (II) for the applied method are given in Table 5.

Parameters	Zn(II)
λ_{max} (nm)	294
Parameter linearity (ppm)	0.1-5
Molar absorptivity($\text{L.mol}^{-1}.\text{cm}^{-1}$)	3516.95044
Mean	0.149125
Slope	0.05529795
LOD(ppm)	0.13904674 ppm
LOQ(ppm)	0.42135377 ppm
Sandell's sensitivity ($\mu\text{g}/\text{cm}^2$)	2.33×10^{-3}

The samples were prepared according wet digestion method [22] and processed by the applied analytical method and at optimum conditions, then the absorption was measured, through the calibration curve, the zinc ion was determined in those samples. The results compared with FAAS, as standard method, and the results were shown in Table 5.

No.	Sample	FAAS Applied method	Applied method (ppm)
1	Avocado	2.345	2.883
2	sunflower	4.057	4.317
3	Lentils	4.123	4.235
4	Banana	0.210	1.029
5	Ordinary soil	2.032	2.782
6	Clay soil	3.252	3.597
7	Almonds	2.882	3.124
8	Drainage water	0.850	1.071
9	Plain water	1.210	1.364
11	Meat	4.123	4.599

4. Conclusion

1. The efficiency of the extraction method depends on the ability of the organic reagent to form a chelating complex with the metal ion under study.
2. Extraction efficiency varies depending on the ability of the metal ion to form a chelating complex.
3. The study demonstrated that acidity plays an important role in the formation of the complex in the aqueous phase and its extraction within the CPL at pH = 9 .
4. Heating time represented the kinetic side of extraction method lead to the quantity of heating in the solution to decide the velocity of micelles to aggregation.
5. The CPE extraction technique highlights the significant role of the surface volume or concentration used, as the optimal aggregation yields the Critical volume, the surface type on the other hand, is a complementary factor that plays a role in micelles formation, both contributing to controlling the efficiency of the extraction process.

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