
Synergistic Extraction of Zinc (II) with TOA and TPPO

Mumtaz Begum , A. V. L. N. S. H. Hariharan*

*Department Of Chemistry, GITAM Institute of Science,

GITAM(Deemed to be University), Visakhapatnam, A.P – 530045, India.

Abstract:

Zinc (II) has been effectively extracted in acidic media using a combination of tri-octyl amine (TOA) and triphenyl-phosphine oxide (TPPO) with xylene. The stoichiometry calculation of different parameters like contact time, pH, and temperature was observed and noted from the zinc (II) concentration during the extraction of zinc (II). The equilibrium time and thermodynamic factors like ΔH , ΔG , and ΔS were measured. In the stripping process of the extracted metal by nitric, sulfuric, perchloric, and hydrochloric acids, 99% of Zn (II) was recovered by two contact stages using sulfuric acid from the organic phase. The main goal was to examine the impacts of TOA and TPPO in an acidic medium to correlate the interaction among the mixtures. It was easy to validate the presence of TOA and TPPO in the extraction and interaction of zinc (II) in the binary combination by employing FTIR spectra analysis, which also provided information on the chemical interactions produced. TG-DSC and Fourier Transform Infrared (FTIR) spectra show two separate stages during the thermal dissociation of zinc: the ligand complex of TOA and TPPO in xylene.

Keywords: -Zinc (II) -Trioctyl amine (TOA), Triphenylphosphine oxide(TPPO), xylene, mineral acid.

1. Introduction

Due to rising consumption, there has been a noticeable growth in the demand for metals.

A mixture of 2-hydroxy-5-nonylacetophenone oxime (Mextral84H)and bis(2,4,4 -trimethylpentyl)-phosphinic acid used to extract zinc and the values of isotherms showed that the addition of Cyanex 272 to Mextral 84H causes apparent synergistic shifts for zinc at pH 1.0 [1]. Tri-n-octylamine separates zinc-iron from used pickling. This study examines how a unique family of hydrophobic ionic liquids extracts transition heavy metal Zinc and other metal ions from HCl aqueous solution without chelating agents.[2]. Zinc is extracted as the chemical ZnCl2 2TRPO in this process, combinations of trialkyl phosphine oxide (TRPO, CYANEX 923),

Organophosphorusacids,di(2-ethylhexyl)phosphoric acid(D2EHPA),and bis(2,4,4-trimethylpentyl) phosphinic acid is used.[3]. To extract HCl solutions containing Mn(II), Cu(II), Zn(II), Fe(III), Co(II), and Zr(IV), it is attempted to compare the solvent extraction capabilities of TBP and di-n-butyl sulfoxide (DBSO). The effects of the organic group on the process of extraction behaviour have been studied through research into the extraction of Fe(III) by triphenylphosphine oxide (TPPO) and diphenyl sulfoxide (DPSO).[4].Unlike most investigations on ammoniacal sulfate systems, the extracting power of zinc with 4-ethyl-1-phenyl-1, 3-octadione (XI-55 and HA) from ammoniacal chloride medium was examined in both the presence and absence of TOPO. It was chosen because complex sulfide ores and secondary materials can be leached of zinc by concentrated ammonium chloride (Allan, 1995; Olper et al., 1993).[5]. Donald C Batsky observed the precipitation of triphenylphosphine oxide with ZnCl2 in polar solvents. TPPO efficiently precipitates phosphine oxides selectively to remove metal ions from mixtures, as when enriching uranium ores. Additionally, although the creation of pure ZnCl2(TPPO)2 crystals from ethanol solutions has been documented, it has never been used to remove TPPO from reaction mixtures.[6]. One can separate Cl ions from zinc waste lixivium using trioctylamine as an extractant, 2-octanol as a solvent, and sulfonated kerosene as a diluent. With ideal values of 99.47%, 595, and 99.99%, the impacts of trioctylamine concentration on the extraction efficiency, separation factor, and stripping efficiency were calculated.[7]. TBP (tributyl phosphate) and Hostarex A226 (diiso tridecylamine) have been used to analyse the

extraction of Zn(II) from Australian hot-dip galvanizing effluent streams and were shown to be the most effective for removing zinc ions from a chloride matrix. Additionally, two substitutes, Hostarex A324 (tri-iso-octylamine) and Hostarex A327(tri-n-octyl/n-decylamine) were examined in model solutions with zinc concentrations of up to 80 g/L and a range of acid concentrations. [8]. Trace levels of zinc in high-purity solders are separated and determined using spectrophotometry. When anionic zinc chloro-complexes are extracted with tri-n-octylamine in benzene, zinc at as low a level as 0.001% in the solder is successfully separated. Zinc is removed with 0.5M sodium hydroxide, and tartaric acid covers any remaining metal traces using the reagent 4-(2-Pyridylazo)resorcinol. [9]. Zinc (II) is extracted using the industrial extractant Hostarex DK-16 (high molecular weight -diketone), which resembles LIX 54 in structure. The study was conducted on solutions with Zn(II) concentrations between 0.01 and 0.20 g/dm3. When extracting Zn(II) from solutions containing 15 g/dm3 of Zn(II) and 3 mol/dm3 of ammonia, Chen et al. (2011) looked at the synergistic effects of mixes of diketone and trioctylphosphine oxide (TOPO). The extractant, ammonia, and ammonium sulfate concentration all affected zinc extraction, but contact duration had no effect.[10]. Copper (II) and zinc(II) ions can be separated from aqueous solutions via synergistic extraction and transport through polymer inclusion membranes (PIMs). In polymer membranes, trioctylphosphine oxide (TOPO) and trioctylmethylammonium chloride (Aliquat 336) were combined to operate as a selective extractant and an ion carrier. 0.025 M TOPO and 0.06 M Aliquat 336 in kerosene were used in the solvent extraction method to separate Zn (II) ions from Cu(II) ions successfully.

The paper describes the separation and recovery of zinc (II) from salt solution and synthetic zinc samples in acidic media with selective extractants.

2. Objectives:

Extraction of Zinc (II) in acidic media using a combination of tri-octyl amine (TOA) and tri-phenyl-phosphine oxide (TPPO)in xylene.

It was aimed to observe the impacts of TOA and TPPO in an acidic medium to correlate the interaction among the mixtures.

. Calculation of different parameters like contact time, pH, and temperature etc. was aimed during the extraction of zinc (II).

The equilibrium time and thermodynamic factors like ΔH , ΔG , and ΔS will be done.

Attempts will be made to estimate zinc in alloys and synthetic samples.

Methods:

3. Reagents And Equipments:

Trioctylamine (TOA) and Triphenylphosphine oxide (TPPO) were mixed at a known weight of 0.25M, each in xylene having an approximate isomeric composition, o-xylene, m-xylene, and p-xylene 35% each, and the mixture was standardized using EDTA solution. Zinc(II) stock solution before being diluted using the appropriate procedure to achieve the desired molar concentration. Zn (II) stock solution(0.5M) in 250 ml diluted using the appropriate procedure to achieve the desired molar concentration and standardized with standard EDTA solution complexometrically. Zinc can be extracted quantitatively from aqueous solutions of 1*M* in HCl, H₂SO4, and HNO₃. The above chemicals used in the analysis were analytical-grade reagents.

The strength of Zinc (II) in the provided sample was determined using an inductively coupled plasma-optical emission spectrometry type ICP-MS Model Perkin Elmer, Nexlon-1000, a magnetic stirrer with temperature control, and an electrode with a digital pH meter.

4. Experimental Technique:

In a 250 ml separating funnel, a sample of Zn (II) 0.05 M with 10 ml of each TPPO and TOA are added. 2.5×10^{-2} M, mixed with xylene and pre-equilibrated with 0.1 M of several acids, including HCl, HNO₃, and H₂SO₄.

A mechanical shaker shook the solution before it was allowed to settle for a while. ICP-OES values considered the distribution coefficients of zinc (II) measured in both phases were assessed before and after extraction.

$$Kd = [Zn(II)]org] / [Zn(II)]aq$$

5. Discussion And Observation:

5.1. IMPACT OF EQUILIBRATION TIME

The two-phase reaction in xylene is rapid; after separating the two phases, Utilizing ICP-OES the concentration of Zinc (II) in the aqueous phase is determined. Moreover, equilibrium is reached in less than 20 min, and 91.3% of zinc(II) is extracted. According to a thorough analysis of the trioctylamine and Triphenyl-phosphine oxide with xylene in the extraction of Zinc (II), a more extended time has no impact on the equilibrium in the extraction of zinc(II), as seen in Fig. 1.

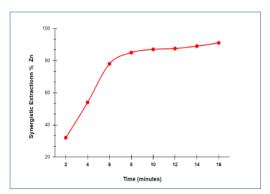


Fig.1. Time variation on zinc(II) extraction

5.2. Ph AND ITS EFFECT ON ZINC (II) EXTRACTION:

For evaluations on synergistic extraction, pH is the primary factor. The extraction percentage of zinc (II)with the extractant concentration (0.025) dramatically increases when the pH of hydrochloric acid and nitric acid increases from 1.0 -3.0, then drops and then lowers while increasing pH. When the pH rises (from 1.0 to 5.0), zinc (II) extraction with sulphuric acid steadily increases—the strength of the zinc(II) extraction mostly depends on hydrogen bonding. Fig. 2 displays the tremendous zinc(II) extraction efficiency at pH 4.0 in sulphuric acid.

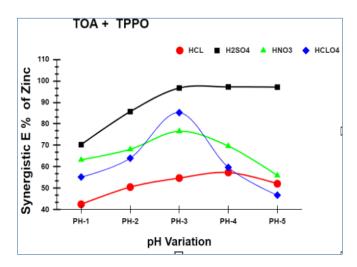


Fig.2. Effect of pH on zinc(II) extraction

5.3.IMPACT OF DILUENTS ON THE ZINC (II) EXTRACTION:

When employing trioctylamine and triphenyl-phosphine oxide with 0.025M concentrations of various solvents such as xylene, toluene, benzene, cyclohexane, carbon tetrachloride, and nitrobenzene, the impacts of diluents on the extraction efficiency of zinc (II), Using xylene, a transparent phase separation was accomplished. As a result, xylene is favoured throughout the tasks listed in(Table 1)as a diluent.

Table 1. Distribution ratio and percentage of Extraction in the H2SO₄ medium at pH 3.0 in various solvents.

Solvent	Distribution ratio	% E
Carbon tetrachloride	0.8388	34.26
Nitro-benzene	1.1204	63.22
Xylene	1.8766	96.28
Toluene	1.0022	50.12
Cyclohexane	0.7996	49.81
Benzene	1.4528	72.66

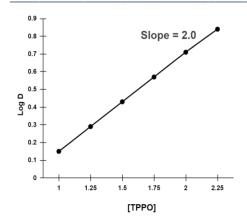
5.4. EXTRACTION OF ZINC (II) & IT'S VARIATION WITH TRIPHENYLPHOSPHINE

OXIDE:

The percentage of Zinc (II) Extraction from TPPO in Xylene at a concentration range of (0.025 to 0.005 M) revealed unexpected distribution ratios of 46.6%. As seen in Fig.3 of log D vs. log TPPO, the composition of extractable species is substantially lower, and the data provided is a straight-line equation to slope 1, which demonstrates that one molecule of triphenyl-phosphine oxide is required for the extraction of zinc (II).

Taking the logarithm and substituting the value of D in equation (1) we have

The individual zinc (II) extraction reaction from TPPO is shown in Fig. 3 due to the stoichiometric coefficients for the zinc (II) extraction reaction in graph log D against log [TPPO] organic and the slope of unity calculated from all the acids employed.



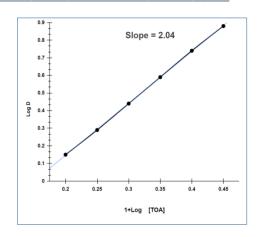


Figure 3: Extractant Variation of (Zn (II) with TPPO)

Figure 4: Extractant Variation of

(Zn (II) with TOA)

5.5. EXTRACTION OF ZINC (II) AND IT'S VARIATION WITH TRIOCTYLAMINE:

The graph of log D vs. log [TOA] in Figure 4 depicts the range of various concentrations that can occur during the extraction of zinc (II) from TOA using xylene. The data available is a straight-line equation to slope 2, demonstrating that one trioctylamine molecule is required for this process.

5.6. EXTRACTION OF ZINC (II) BY SYNERGISTIC METHOD IN THE PRESENCE OF TRIOCTYLAMINE AND TRIPHENYLPHOSPHINE OXIDE:

Experimental results indicate that the scope of the zinc(II) extraction rate is below average with a single extractant. However, the extraction of zinc(II) significantly improved when xylene was mixed with trioctylamine and triphenylphosphine oxide. Due to synergism, the results achieved in the distribution coefficient are more effective than one solo extractant, which accounts for the considerable shift in Zinc(II) extraction. Positive distribution coefficient and synergistic coefficient values denote synergism. Table -2 contains the S.C. and D values to assess synergistic effects. When S.C. & ΔD are positive, synergism takes place.

Synergistic Coefficient S.C = log D mix
$$(DTPPO + DTOA)$$

$$\Delta D = D mix - (D TPPO + D TOA)$$

D mix = distribution coefficient in the presence of binary mixture.

DTPPO = distribution coefficient in the presence of Triphenylphosphine oxide.

DTOA = distribution coefficient in the presence of Trioctylamine.

$$[Zn^{+2}]$$
 aq + $[TPPO]$ org + $[TOA]$ org <-----> $[Zn^{+2}(TPPO).(TOA)]$ org -----(6)

Therefore,

$$K = [Zn^{+2}.(TPPO).(TOA)] \text{ org}$$

$$[Zn^{+2}] \text{ aq.}[TPPO] \text{ org.}[TOA] \text{aq}$$

Table.2. Results of Zn (II) in synergistic extraction

	ITTO A I	D	D	%E	SC	ΔD
Solvent	[TOA] M	D _{TPPO}	D _{mix}			
	0.01		0.4729	53.3	0.23	1.81
	0.02		0.4815	56.7	0.24	1.80
Xylene	0.025		0.4821	56.9	0.21	1.80
	0.05	1.8766	0.4917	61.5	0.21	1.79
	0.075		0.5196	76.6	0.22	1.76
	0.1		0.8383	98.2	0.36	1.44
	0.01		0.3998	41.4	0.21	1.46
	0.02		0.5324	57.6	028	1.33
D	0.025	1.4528	0.5340	58.3	0.28	1.33
Benzene	0.05		0.5756	58.9	0.30	1.28
	0.075		0.6328	67.8	0.33	1.23
	0.1		0.7265	72.6	0.39	1.13
	0.01		0.2583	36.2	0.18	1.15
Toluene	0.02		0.3785	44.7	0.26	1.03
	0.025		0.3997	47.3	0.28	1.01
Toluelle	0.05	1.0022	0.4323	51.9	0.30	0.98
	0.075		0.4887	56.5	0.34	0.92
	0.1		0.5011	60.1	0.35	0.91
Cyclohexane	0.01		0.1250	21.4	0.10	1.08
	0.02		0.1433	38.3	0.11	1.06
	0.025		0.1654	43.7	0.13	1.04
	0.05	0.7996	0.1668	44.2	0.13	1.04
	0.075		0.1896	47.6	0.15	1.02
	0.1		0.2004	49.8	0.16	1.01

Solvent	[TOA] M	D трро	D _{mix}	%E	SC	ΔD
Carbon tetrachloride	0.01 0.02 0.025 0.05 0.075 0.1	0.8388	0.1397 0.1408 0.1442 0.1461 0.1575 0.1612	16.7 18.9 21.1 23.4 27.8 34.2	0.11 0.11 0.11 0.12 0.12 0.13	1.11 1.10 1.10 1.10 1.09 1.08
Nitrobenzene	0.01 0.02 0.025 0.05 0.075	1.1204	0.1077 0.1139 0.1188 0.1201 0.1263	39.4 45.2 46.7 58.2 62.7	0.07 0.07 0.08 0.08 0.09	1.42 1.41 1.41 1.40 1.40

The graph of log (D mixture) vs log [TPPO] and log [TOA] together shows a straight line of slope 1, which is mentioned in Fig 5 and Fig 6, respectively. Hence, based on the observation, one molecule of TPPO and one molecule of TOA are present in a mixed extractant, which leads to synergism.

0.1284

63.2

0.09

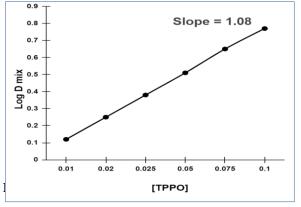
1.40

$$Dmix = [Zn(TPPO).(TOA)org -----(8)$$

$$[Zn^{+2}] aq$$

substitution values of D in D_{mix} , in equation 8 and 5 log values on both sides,

$$Log K = log [D_{mix}-D]_{Org}-log [TPPO]_{Org}-log [TOA] org \qquad -----(9)$$



0.1

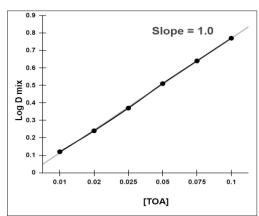


Figure 6: Log D mix vs. Log [TOA]

6. Temperature Effect:

6.1. DIFFERENTIAL SCANNING CALORIMETRY RESULTS:

Thermal characteristics of the Zinc complex $[Zn(R_3N). (Ph_3PO)X_2]$ was analyzed for temperature affect with the extractants individually in both phases in an acidic medium and binary extraction systems.

The interaction between various components must be evaluated to determine how the thermal characteristics of the synergistic mixture and the combination of extractants alter based on the amount of zinc complex with TPPO and TOA. Zn forms complexes with TOA and TPPO in xylene, [Zn(RN). (Ph3 PO)X2]. There are four peaks in the DSC curve(Figure 8), with four sequential stages with chunky peaks at the start and a sharp peak at the end, which proves four different components in the complex. The Y-axis represents the heat flow in mW, and the X-axis represents the temp in C and time in minutes. At roughly 30.53 C, the process of thermal dissociation begins, and it ends at roughly 107.4 C. The peaks' steepness indicates the change from solid to liquid. Phase (melting temperatures of 76.34C and 103.15C). The enthalpies are -21.26mJ,mol⁻¹,+26.28mJ,-139.08mJ, and -81.47kJmol⁻¹ for 15K min ⁻¹ (Figure 7), respectively.

DSC methods were used to examine the compound's thermal dissociation [12]. Two stages can be seen in the thermal dissociation of zinc by TG-DSC experiments: the ligand complex of TOA and TPPO. TOA inhibits dissociation; it makes sense that it would have a broad, blunt endothermic peak at around 103, subsequently creating a distinct endothermic peak at higher temperatures by simultaneously releasing TOA and TPPO. Furthermore, it was discovered that the main source of the total enthalpy of dissociation was the enthalpy of vaporization of TOA. After first increasing, the absorption intensity goes through two sequential peaks before gradually decreasing.

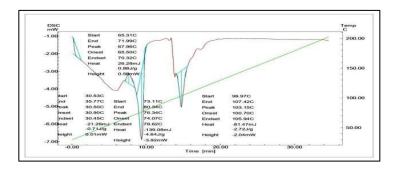


Fig 7. DSC Curve of the synergistic mixture

The equilibrium extraction constant logK is calculated using equations (5) and (9) for complex analysis.

The Van't Hoff equation was used to compute the enthalpy change from the equilibrium extraction constant values at the tested temperature range.

$$\log K = -\Delta H/2.303RT + \Delta S/2.303R$$
(10)

Figure 7 shows a straight-line plot of log K against 1/T where the slope represents the enthalpy of reaction (H $^{\circ}$) and the intercepts represent the entropy (S $^{\circ}$) value. In (Table 3), the values for G, H, and S are displayed.

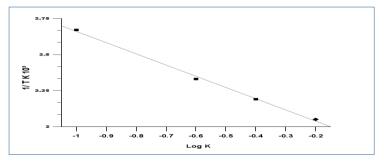


Figure .8: Temperature variation

System	Solvent	ΔH° (kJK ⁻¹ . mol ⁻¹)	ΔS° (JK ⁻¹ . mol ⁻¹)	ΔG° (kJK ⁻¹ . mol ⁻¹)
		mor)	moi)	mor)
	Xylene	- 79.56	53.02	-26.56
	Toluene	-38.90	27.90	-12.42
	Nitrobenzene	29.91	52.50	-11.72
Zn(II)	Benzene	23.86	57.49	-18.69
TPPO-TOA	Carbon tetrachloride	19.22	34.08	-23.11

Table 3. Thermodynamic Parameters

According to data, enthalpy and entropy are preferred during extractions, positive values show that bond breakage occurs during extraction and positive entropy levels have represented the release of water molecules in mixed extraction. Advantageously, the widespread breakdown of the metal's hydration sphere during adducts formation results in the release of such water molecules.

24.26

35.06

-11.82

Cyclohexane

7. FTIR RESULTS

Fourier Transform Infra-Red (FTIR) spectroscopy examines the chemistry of coordination of the solvent-extracted metal complexes in the organic phase. Trioctylamine (TOA) and triphenyl-phosphine oxide (TPPO) dissolved in xylene were part of the organic phase used to extract zinc (II) from an acidic medium. The recovered organic phase was subjected to FTIR analysis (PerkinElmer spectrum IR ES Version 10.6.2) to get information about the coordination chemistry and aggregation behavior of the Zn (II)-ligand complex in the binary solution. The spectra range's 1516–483 cm-1 region was observed. Figures 9,10 and 11 display the zinc-loaded organic phase of (TOA-TPPO-Zinc) FTIR spectra in xylene at room temperature [13]. The distinct vibrational bands of TOA (C/N), 740 cm1) and TPPO ((Ph\O\P), 1516 cm1) are affected by zinc (II)extraction.

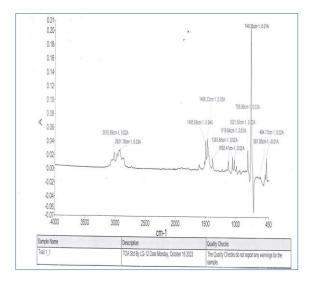


Fig.9.FTIR spectra of TOA (org)

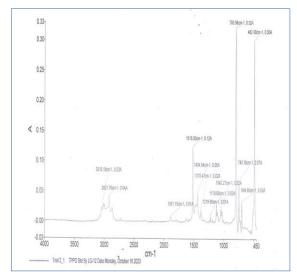


Fig 10. FTIR spectra of TPPO(org)

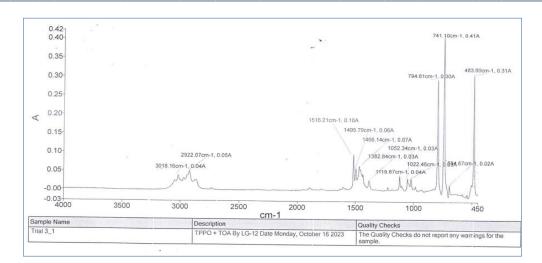
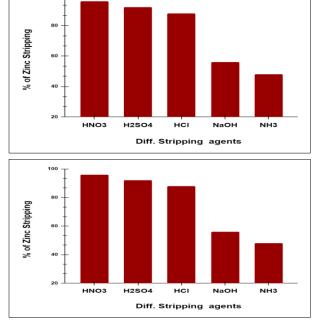


Fig 11. FTIR spectra of a synergistic mixture of TPPO +TOA (organic phase)

As seen in Figure 11, the absorption intensity rises at first, passes through three sequential maxima, and then gradually decreases in the TOA + TPPO mixture.

8. STRIPPING OF ZINC(II):

A stripping test was done to re-extract zinc (II) ions from the organic phase. This approach used 0.01 to 1.0 M concentrations of HCl, H₂SO₄, HNO₃, and NaOH solutions of 0.01 to 1.0 M. The loaded organic and aqueous phases were shaken at different volume ratios. It was found that HNO₃ at 1.0M was superior to HCl, H₂SO₄, and NaOH as an effective stripping agent for Zn (II) ions at an O/A volumetric ratio of 1:3. Two theoretical stripping stages are required to strip almost all the extracted Zn(II) from the loaded organic phase containing TOA and TPPO (Fig 6). The stripping process efficiency was equal to 96.2 %, beyond which there was no significant increase in extraction.



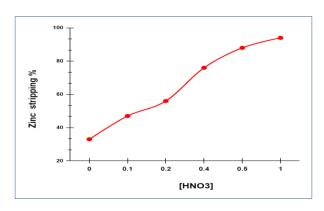


Figure 12. Stripping of Zn(II) with different agents

9. Impact Of Diverse Ions:

Zinc (II) extraction in the presence of foreign ions was investigated using the general extraction technique. The concentration of Zinc (II) (13.9 g/100 ml) was the one for which the recovery of Zn (II) tolerance limit was set at + 1% error. The findings (Table 4) demonstrate that while ions like Bi(II), Ca(II), Pd(II), Ba(II), Pb(II), Sr(II), Chlorate, Silicate, Phosphate, and Halide do not interfere even when present in the ratio 1:50, ions like Al(III), Cr (III), Ag(II), Cu(II), Sb(III), Sn(II), Co(II), Ni(II), Mn(II), Ba(II), Chlorate, Silicate, Phosphate, and Halide are tolerated in the ratio 1:30. In the 1:20 ratio, the ions with the minor tolerance limits are Fe (III) and Cr (III). Therefore, zinc (II) can be extracted even in the presence of several cations and anions. The average recovery for Zinc (II) was 99.4 + 0.2% respectively.

10. ESTIMATION OF ZINC IN NATURAL AND SYNTHETIC SAMPLES:

Analysis of actual samples of zinc (II) concentration in galvanized alloys and synthetic samples has proven the usefulness of the suggested procedure. 10 ml of aqua regia was used to dissolve a known weight of a synthetic zinc sample or galvanized alloy. After the above solution dried out completely, 10 milliliters of hydrochloric acid solution were added to the above extract. The precipitate was filtered and quantitatively cleaned to recover all of the zinc (II). Following the previously stated process, 10 milliliters of filtered solution were extracted using an equal volume of 0.025 M TPPO+TOA in xylene, and the zinc (II) content was calculated. (Table 5) presents the findings.

Table 4. The impact of Diverse ions [Zn (II)] = 3.5 g, and [TPPO+ TOA] = 0.05 M

Foreign ion	Tolerance limit	Foreign ion	Tolerance limit
Al ³⁺	367	Sn ²⁺	177
$\mathbf{A}\mathbf{g}^{\scriptscriptstyle{+}}$	763	Sb ³⁺	904
Cd ²⁺	570	Cr ⁺²	984
Fe ³⁺	270	Si ₂ O ₅ ² -	718
Mn ²⁺	468	ClO ₃ -	551
Ni ²⁺	461	PO ₄ ³ -	902
Cu ⁺²	704	Halides	867

Table 5: Estimation of zinc present in its alloys and synthetic samples

Sample	Zinc before	Zinc after	% of Zinc
	recovery	recovery	
Eveready (C-Zn)	0.80 gm/l	0.794 gm/l	99.2
battery			
Nut Bolt (galvanized)	1.0 gm/l	0.986 gm/l	98.6
Lacto Calamine lotion	0.74 gm/l	0.731 gm/l	98.7

11. Conclusion:

Thermodynamic factors determine the endothermic nature of zinc (II) extraction using a solution of triphenylphosphine oxide and trioctylamine with xylene in an acidic medium. It favors entropy due to the breakdown of hydrogen bonding during hydrolysis. These factors include pH, extractant concentration, contact time, temperature phases, distribution ratio, and stripping. Utilizing FTIR and DSC methods, the mixture's thermal dissociation was examined. The ligand combination of TOA and TPPO is one of three stages that emerge during the heat dissociation of zinc, according to TG-DSC investigations. The fact that TOA prevents dissociation may explain why it exhibits a broad, shallow endothermic peak at lower temperatures (about 110 °C).

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Conflict Of Interest:

The authors declare that they have no conflict of interest.

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