DETERMINATION OF ZINC (II)

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Zinc compound have a long but undistinguished history in oral hygiene products. Zinc salts are now in many marketed dentifrices and mouthwashes worldwide. They are used for products in which antiplaque effects have been demonstrated and in which anti " bad breath" effects have been documented.

Zinc salts have been reported to fulfill there distinct functions.

i. Reduces plaque accumulation.
ii. Reduce calculus accumulation.
iii. Reduces oral malodor.

Zinc compounds can affect oral hygiene and health status by at least four mechanisms [1]

1. Interference with microbial growth and proliferation
2. Interference with mechanism involved in the formation of microbial deposits.
3. Inhibits of the toxic effects of volatile sulphar compound.
4. Prevention of the toxic effects of volatile sulphar compound on peri dental tissue.

The zinc Ion can inhibits or after microbiological metabolic process. The Zinc ion is capable of non selectivity precipitating proteins Evans et al [2] explained the ability of Zinc salts.

The main uses of zinc are as a ingredient of alloys, such as bronze for decosting , brass , Bobbitt metal, German silver and special alloy for die costing as a protective coating for other metals to prevent corrosion, for electrical apparatus , especially dry cell batteries, household utensils, casting printing plate, building materials, automotive equipment, as reducing agent in organic chemistry, for cyanide process. Purifying fats for soap bleaching bone glue, manufactures of sodium hydrosulphide, insulin zinc salt, as a reagents in analytical Chemistry and it is a nutritional tracer element. Some colorimetric method for extraction and determination of zinc is also used [3-4]

Procedure for Extraction of Zinc (II)

An elequet of Zinc solution (containing 1mg of Zinc per ml.) was taken in a beaker, 2ml of 0.4M Sodium acetate and 1ml of 6% alcohoholic solution of Isonitrosothiocamphor was added and the mixture of solution was adjusted to 6 pH with the help of dilute solution of hydrochloric acid or sodium hydroxide. The volume of mixture was diluted to 10ml transferred into a separating funnel and shaken for a several time with an equal volume of an organic solvent. The two layer were separated out.

The compound was analyzed as anal. Calcd. For C_{20} H_{28} $N_2O_2S_2Zn$:

C 53.60%; H 6.18%; N 6.18%; S 13.88%., and Zn 14.28%,

Found C 52.64; H 6.22%; N 6.24%; S 14.81%. and Zn 14.20%

Procedure for Spectrophotometer determination of Zinc (II)

To an aqueous solution, 4ml (containing 1mcg to 20mcg) of zinc is taken in a beaker 2ml of 0.04m Sodium acetate solution and 0.5 ml of 0.5% Isonitrosothiocamphor in alcohol was added. The pH of solution was adjusted to 7 with the help of dilute solution of hydrochloric acid or sodium hydroxide, 2ml of buffer solution of sodium acetate pH 4 was added. Transferred the mixture in a separating funnel with diluting up to 10 ml. shake the mixture for 1 minute with 10 ml chloroform. Allow to separate the chloroform layer. Filter the chloroform layer through anhydrous sodium sulphate and measure the absorbance of the solution at 625 nm, using reagent blank.

The amount of zinc present was determined from a calibration curve, which was prepared as follows.

A series of solution containing known amount of zinc were treated as above method and a graph of absorbance against concentration of zinc were plotted (Refer table 21)

Procedure for mole ratio curve:

A series of mixture containing 20 mcg of Zinc & increasing amount of reagent in 1 ml of alcohol were treated as described in procedure of spectrophotometer determination of zinc. Absorbance of such extract was measured at 635 nm using chloroform as a blank (Refer Table 22)

Stability of complex:

The absorbance of solution does not show any change up to 180 min. (Refer. Table 23)

Effect of pH:

It was observed that the pH range 5-7 gives better results and at this pH range, extracted in solvent was stable up to 180 min. (Refer. Table 24)

Results and Observation:

The results of extraction studies shows that zinc can be quantitatively extracted into chloroform from an aqueous solution containing Isonitrosothiocamphor at pH 5-7 bellow and above of this pH range extraction of zinc decreases.

The value of extraction of zinc gives following order for the organic solvent used

Chloroform > Benzene > Toluene > Carbon tetrachloride > ether > butanol.

(Refer table 25 & 26)

Validation of Analytical Technique [5-7]

Parameter: The analytical procedure is prepared for the following parameter.

1. **Specificity:-** Specificity is carried out to check the interference due to diluents in analysis.
   Acceptance Criteria: The absorbance proves that there is no interference due to diluents indicating method is specific.

2. **Linearity :** Linearity is established by demonstrating that the results obtained, are directly proportional to that concentration. The Solution were prepared at different concentrations level and absorbance was measured.
   A graph of concentration v/s absorbance was plotted which is linear.

3. **Accuracy:** Accuracy of the method was established by recovery experiment known amount of zinc was added at there different concentration and analysis was performed.

Sr. No.	Qty. added in ppm	Absorbance	Quantity recovered in ppm	% Recovery
1	0	0	0	--
2	10	0.222	9.93 ppm	99.30 %
3	20	0.449	10.04 ppm	100.40 %
4	30	0.679	10.05 ppm	100.50 %
Mean recovery.				100.06%

Mean recovery of 3 level is calculated, which is 100.06 and is in acceptable limit, indicating that method is accurate under the prescribed conditions.

4. Precision: Precision of the method was established by performing assay 3 time by same analyst.

Sr.No.	Conc. of Zinc in ppm	Absorbance	% of Zinc
1	20 ppm	0.442	98.89 %
2	20 ppm	0.451	100.89 %
3	20 ppm	0.444	99.31 %
Mean			99.70 %

Mean of 3 analysis is 99.70 % and is in acceptance limit, indicated that method is validated.

5. **Robustness:** The robustness is proceeded by obtaining the analytical results on the portion of same sample by different analyst.

Sr.No.	Conc. of Zinc in ppm	Analyst	Absorbance	% of Zinc
1	20 ppm	Analyst I	0.446	99.78 %
2	20 ppm	Analyst II	0.442	98.89 %
3	20 ppm	Analyst III	0.449	100.45 %
Mean				99.71 %

Mean of results of all three analysts is 99.71 % which is in acceptable limit, and indicated that the method is validated.

Table 21

Absorbance measurement of zinc in chloroform with the help of Isonitrosothiocamphor at different wavelength .

Concentration of zinc : 20 ppm

Volume of chloroform : 10 ml

Time of equilibrium : 1 min.

Sr. No.	Wavelength	Absorbance
1	350	0.416
2	360	0.390
3	370	0.390
4	380	0.370
5	390	0.350
6	400	0.325
7	410	0..320
8	420	0.320
9	430	0.315
10	440	0.311
11	450	0.308
12	460	0.305
13	470	0.299
14	480	0.307
15	490	0.321
16	500	0.340
17	510	0.351
18	520	0.365
19	530	0.362
20	540	0.380
21	550	0.397
22	560	0.392

Cont.

23	570	0.397
24	580	0.408
25	590	0.403
26	600	0.409
27	605	0.425
28	610	0.422
29	615	0.428
30	616	0.430
31	617	0.430
32	618	0.434
33	619	0.437
34	620	0.439
35	621	0.440
36	622	0.440
37	623	0.442
38	624	0.445
39	625	0.447
40	626	0.446
41	627	0.446
42	628	0.445
43	629	0.442
44	630	0.447
45	631	0.445
46	632	0.440
47	633	0.445
48	634	0.434
49	635	0.426
50	640	0.422
51	650	0.405

Table 22

Effect of Reagent concentration on the absorbance of Zinc(II) and Isonitrosothiocamphor.

Concentration of zinc	:	20 ppm
Organic phase	:	Chloroform
Time of equilibrium	:	1 min.
Wavelength	:	625 min.

Sr.No.	Volume of Isonitrosothiocamphor	Absorbance
1	0.1	0.411
2	0.2	0.424
3	0.3	0.433
4	0.4	0.438
5	0.5	0.442
6	0.6	0.447
7	0.7	0.447
8	0.8	0.447
9	0.9	0.447
10	1.0	0.447
11	1.5	0.447
12	2.0	0.447

Table 23

Absorbance of Zinc (II) with the help of Isonitrosothiocamphor in chloroform at different pH

Concentration of zinc : 20 ppm

Organic phase : 10 ml Chloroform

Wavelength : 625 min.

Sr.No	pH	Absorbance	% extraction
1	1.0	0.292	65.32 %
2	2.0	0.295	65.99 %
3	3.0	0.365	81.65 %
4	4.0	0.388	86.80 %
5	5.0	0.440	98.43 %
6	6.0	0.447	100.00 %
7	7.0	0.447	100.00 %
8	8.0	0.432	96.64 %
9	9.0	0.426	95.30 %
10	10.0	0.409	91.49 %

Table 24

Absorbance of Zinc(II) in chloroform with Isonitrosothiocamphor as function of time.

20 ppm of Zinc(II) in 10 ml of chloroform at pH 6 equilibrated with respect to different time at 625 nm.

Sr.No.	Time in minute	Absorbance
1	1	0.447
2	5	0.447
3	10	0.447
4	20	0.447
5	40	0.447
6	60	0.447
7	90	0.447
8	130	0.447
9	180	0.447
10	240	0.445

Table 25

Absorbance measurement of Zinc(II) with different volume of chloroform

Concentration of Zinc : 20 ppm
Wavelength : 625 nm
Time of equilibrium : 1 min.

Sr.No.	Volume of chloroform in ml	Absorbance
1	5	0.881
2	10	0.446
3	15	0.292
4	20	0.221
5	25	0.174

Table 26

Extraction of Zinc (II) with Isonitrosothiocamphor using different solvent at pH 6

Organic phase : 10 ml

Time of equilibrium : 1 min.

Sr.No.	Solvent	Absorbance	% extraction
1	Chloroform	0.447	100.0%
2	Benzene	0.440	98.43%
3	Toluene	0.358	80.08%
4	Carbon tetra chloride	0.315	70.46%
5	Ether	0.302	67.56%
6	Butanol	0.115	25.72%

Table 27

Calibration Curve for Zinc (II)

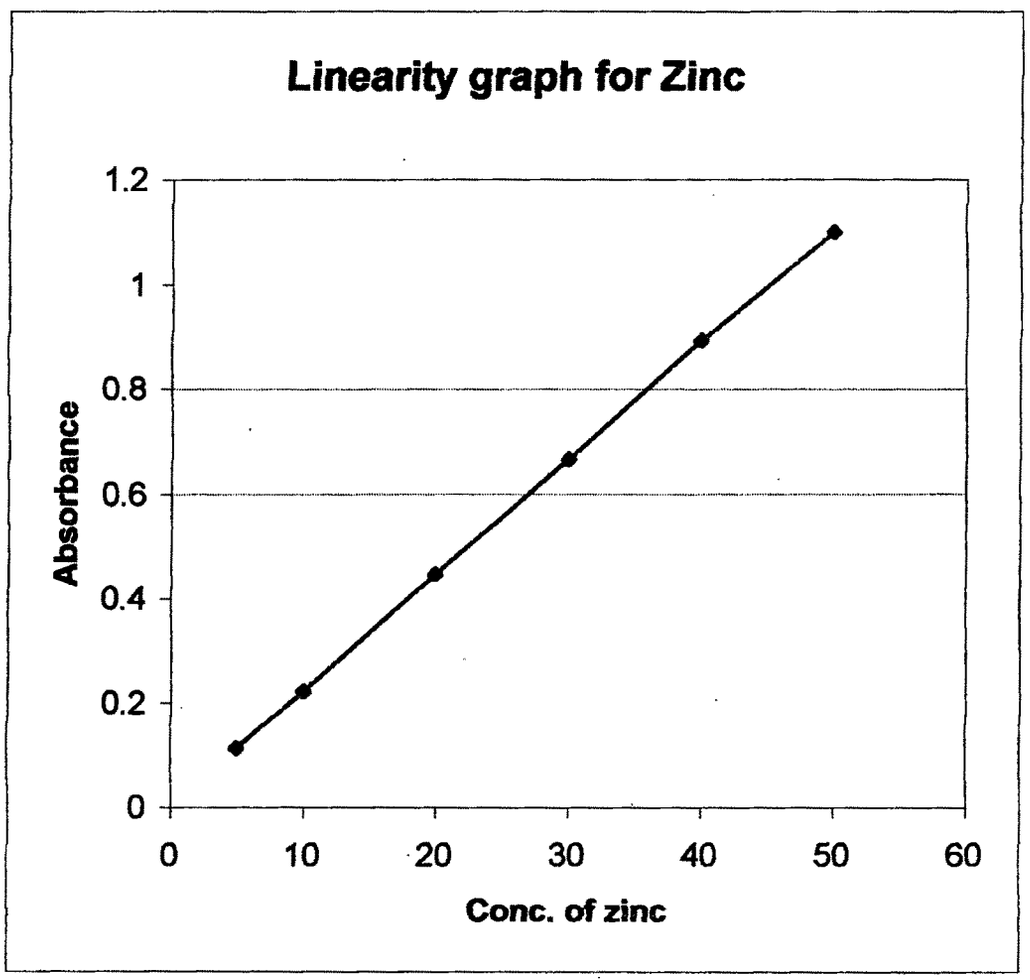
Organic phase : 10 ml
Time of equilibrium : 1 min.
Wavelength : 625 nm

Sr.No.	Conc. Of Zinc in ppm	Absorbance
1	5	0.113
2	10	0.221
3	20	0.447
4	30	0.665
5	40	0.892
6	50	>1.0

Linearity graph for Zinc

Calibration Curve for Zinc (II) at 625 nm in Chloroform.

Conc. Of Zinc in ppm	Absorbance
5	0.113
10	0.221
20	0.447
30	0.665
40	0.892
50	1.1



Infrared studies of Zinc (II)

Infrared spectrophotometers are used for recording spectra in the region 4000 cm^{-1} to 670 cm^{-1}.

The absorbance (A) is defined as the logarithm to base 10 of the reciprocal of the transmittance (T)

$$A = \text{Log}_{10} (1/T) = \text{Log}_{10} (Io/I)$$

$T = Io/I$,

Io = Intensity of incident radiation ,

I = Intensity of transmitted radiation.

The infrared absorption spectrum has a high degree of specificity and no two compounds have identical spectra small differences in structure frequently results in significant difference in spectra.

The problem noted above in making accurate infrared measurements have stimulated research on new methods of spectrochemical instrumentation.

The importance of infrared spectroscopy is obvious from its applications in a Varity of field. Infrared spectra arise from rotational and vibrational motion of the molecules. The theoretical background for the interpretation of these spectra is provided by classical and quantum mechanics.

The factors determining the intensity of an absorption band are the transition probability , frequency of the absorbed radiations can be made by studying the distribution of line intensities in the rotation absorption band of the infrared spectrum of hydrogen chloride recorded at different temperature.

The absorbance of functional group determined with the help of Infrared is as follows.

1. **Nitroso compound** : N=O bond is stable in nitroso compounds is characterized by a stretching vibration in the range [8] 1621 -1488 cm^{-1}

Nitroso compound tend to dimerise, secondary primary nitroso compound readily rearrange to oxime. In monomeric state absorbs in the 1621-1488 cm^{-1} [9] region, but in the solution they exist preferably as dime rise and then absorbs near 1290 cm^{-1}.

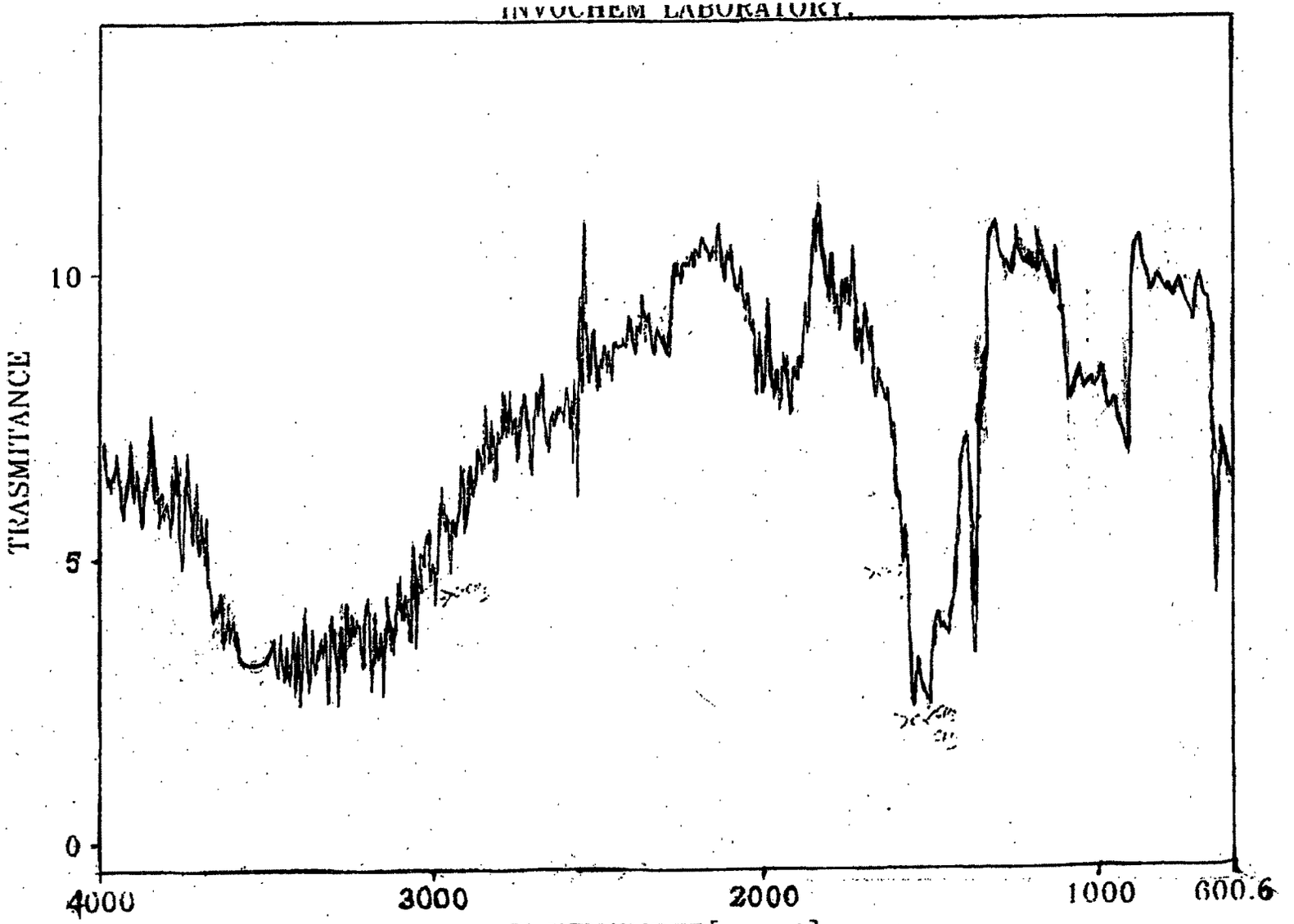
2. **→C– CH_3 group**: The mode of → C– CH_3 gives an observation at about [10] 2872 cm^{-1}

3. **>C=S group** : The range of >C=S group occurs in the range[11] of 1000-1400 cm^{-1}

4. **>C<** CH_3 **group** : The frequency of >C< CH_3 is at about [12] 1340 -1460 cm^{-1}

Two new bands around 470 and 350 cm^{-1} appeared in the spectra of all the complexes, which could be assigned to complete vibrations having contribution from υ MN υ MS.

υ – OH in free ligend disappear in the complex, and a new weak new band at 280 cm^{-1} appears. This indicates the deprotonation of the ligend molecule during the complexation. Further, the intensity of υ = C – decreases and it shifts to downward direction in the complex indicating thereby complaxation of oxygen side of the ligend.



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AMPLE : 9

B.NO. :

R.NO. :

DATE : 03/10/03

Reference:

1. Morton Pador – Oral hygine Products & Practice (Consumer product Development Resources Inc. Teaneck, New Jersey, Page 352, 1988.
2. R.T. Evans, P.J. Baker, R.A. Coburn, S.L. Fishman & R.J. Genco , J Peridentol 48:156 (1977)
3. Dr.P.D. Sethi " Analysis of drugs in Pharmaceuticals formulations" Page 375 1993.
4. D.C. Carathte " The Quantitative analysis of drugs " Page 693 1964
5. United state Pharmacopoeia (26) Page 124 2003
6. Dr. P.D. S Sethi " Quantitative analysis of drugs in Pharmaceuticals formulations" Page 34, 1993.
7. P.P. Sharma "How to Practice GLP" Page 198,2000.
8. Margarta Avarm & Gh. Maeeseu " Infrared spectroscopy" Page 312 1978
9. Silverstein, Bassler, Morril "spectrometric identification of organic compound" John Wiley and sons Inc. New yark pg. 128, 1991
10. Y.R. Sharma " Elementry Organic Spectroscopy" Page 91 1999
11. Margarta Avarm & Gh. Maeeseu " Infrared spectroscopy" Page 293 1978
12. Margarta Avarm & Gh. Maeeseu " Infrared spectroscopy" Page 124 1978