TRACE DETERMINATION OF LEAD BY AC POLAROGRAPHY

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The determination of lead in trace amounts is particularly important because of the biological hazards caused by contamination of it in various objects. A number of techniques like neutron activation analysis [1], atomic absorption analysis [2], gas-chromatography [3], high performance liquid chromatography [4], and stripping voltammetry [5] have been employed for lead determination. Amongst the polarographic techniques, DPP and DPASV are most extensively used for Pb(II) determination. AC polarographic methods are comparatively less studied. Heigl [6], Lapitskaya [7], Neeb [8], and Satiadarma [9], have reported AC methods for Pb(II). In our work, studies have been carried out using AC polarographic mode of determination.

EXPERIMENTAL

A Metrohm Polarecord E-506 along with a polarography stand E-505 (Serie 03) with a dropping mercury electrode Ag/AgCl reference electrode and a Pt electrode were used in this study. Following are the recording and other parameters maintained during the investigation.

Initial Potential	-0.2V
Potential Range	-1.5V
Drop time	1 sec
Paper Speed	60 mm min
Scan rate	6 mv/sec
Total Volume	25 ml.
Current Sensitivity	$1x10^{-9}$ to $1.5x10^{-10}$ A/mm
AC Pulse amplitude	7 mV
AC frequency	75 Hz
Phase Angle	0°
Deaeration time	15 min.

All stock and test solutions were prepared in triple distilled water using analytical grade reagents and salts.

RESULTS AND DISCUSSION

Effect of Background Electrolytes

Of the many buffers used as the medium, ammonium salts of ascorbates, citrate and phthalate were found to be good. The peak potential shifted to negative values with increase of pH of the medium. The peak were observed to be asymmetric in most cases except in 0.2 M and 0.1 M potasium biphthalate. In our studies, 0.2 M potassium biphthalate was chosen as background electrolyte. Fig 1 shows a polarogram for 1ppm Pb(II) and the blank.

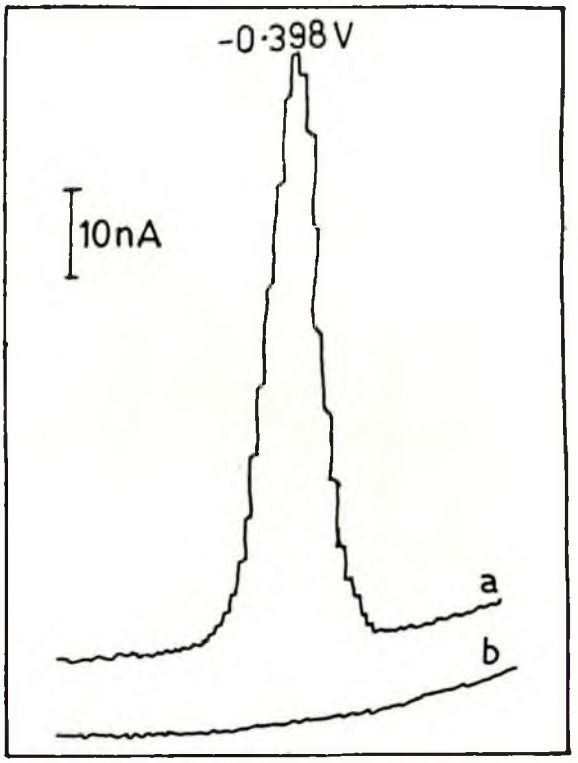


Fig. 1 A typical AC 1 Polarogram curve(a) 1 ppm Pb (II) curve(b) blank

Current-concentration relationship was observed to be linear over the range 0.01 to 0.1 ppm, 0.1 to 1.0 ppm and 1.0 to 10.0 ppm of Pb(II). The method is sufficiently sensitive so as to determine as low as 10 ppb of Pb(II).

It was found that 1 ppm Pb(II) could be measured in presence of the following impurities. Tl(I) (1:50), Ni(II) (1:25), Bi(III) (1:20), Co(II) (1:20), Mn(II) (1:20), Zn(II) (1:15), Cu(II) (1:10), Cr(VI) (1:5), As(III) (1:11), and Cd(II) (1:1). However, Sn(IV) interfered. The method is selective for Tl(I), permitting determination of Pb(II) in large excess of Tl(I).

APPLICATIONS

Simultaneous Determination of Metals

The elements that could be determined

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simultaneously in a mixture were Cu(II), Bi (III), Pb (II), Cd (II),Cr (VI) and Zn (II) or Ni (II). The potential range used was from 0 to -2.0 V with scan rate at 8 mV/s with other conditions same as described before. Fig. 2 shows the polarogram obtained

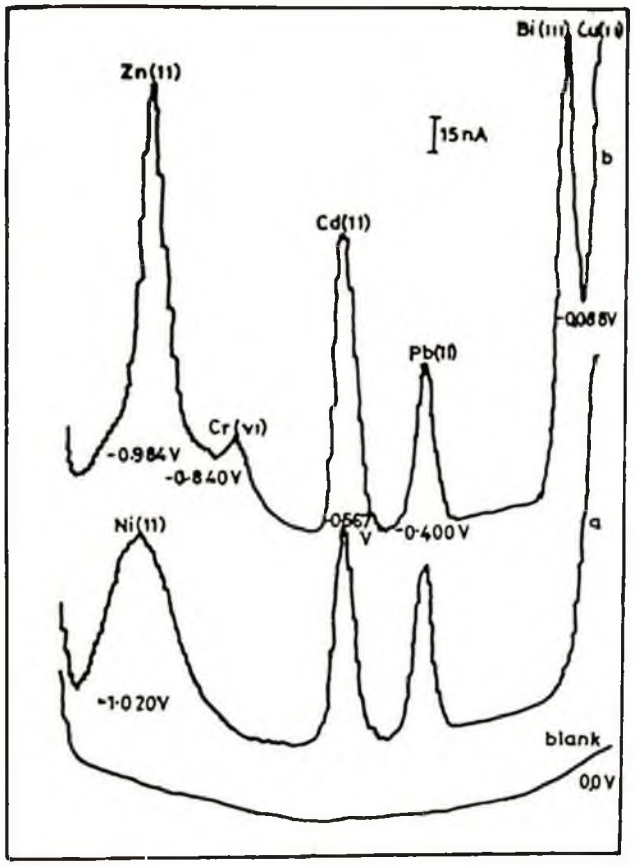


Fig. 2 Simultaneous determination:

Curve a is of Cu(II), Pb(II), Cd(II) (1 ppm each) and Ni(II) (3 ppm); Curve b is of Cu (II), Bi (III), Pb (II) (1 ppm each), Cd(II) and Zn(II) (2 ppm each) and 3 ppm of Cr(III)

Cu(II) signal being close to initial potential, it can only be detected qualitaively unless the scanning is started from small positive potentials. Metal ions are estimated by the standard addition method and the recovery of metals in both the mixtures was found to be of the order of 98%. It may, however, be mentioned that response of Cr(VI) was rather poor and a minimum of 3 ppm of Cr(VI) should be present for quantitation at the given sensitivity.

Determination of the Lead in Tin Base White Metal (BCS No.178/2)

Weighed sample (~ 0.1 g) is dissolved in 10 ml conc. nitric acid with continuous heating. The precipitate of metastanic acid formed is filtered and the residue washed with small portions of 25 ml of hot 1:1 nitric acid. The filtrate is evaporated almost to dryness to expel acid. After cooling, the contents are taken up in triple distilled water and made upto 10 ml in a volumetric flask. A 1 ml aliquout of the solution is analyzed for lead content.

Table 1 gives the amount of Pb(II) obtained by calibration method and standard addition methods along with the reported value (average of 15 observations made in each case). The values of lead determination agree with the reported value.

TABLE 1

DETERMINATION OF LEAD IN TIN BASE WHITE METAL

% Pb				
Calibration method	Standard addition method	Average	Reported value	Error
3.19	3.17			
3.18	3.20	3.19	3.18	0.01
3.20	3.20			
	RMD = 0.95%			

Determination of Lead in Vinegar

Synthetic vinegar. samples obtained from the local market were analyzed for lead content using the following procedure, 25 ml, of the sample are wet digested with 2 drops of conc. HNO_3 and heated to dryness. The residue left is taken up in triple distilled water and volume made upto 10 ml. From this solution 1 ml and 3 ml aliquots are taken and analyzed for lead content. Table 2 gives the results obtained by calibration and standard addition methods. A total of 10 recordings were performed in each case to ensure reproducibility and the average is tabulated in Table 2.

TABLE 2

DETERMINATION OF LEAD IN SYNTHETIC VINEGAR SAMPLE

Ml aliquot of sample solution	Pb Content ppm: Calibration Method	Standard Addition Method	Average
1.	0.090	0.101	
2.	0.099	0.099	0.097
3.	0.100	0.090	

RMD = 0.79%

TABLE 3

DETERMINATION LEAD IN CANNED JUCIES

Juice	ml of serum Solution	Pb Content (ppm): Calibration Method	Standard Addition Method	Average
Orange	1	0.620	0.640	
	2	0.630	0.640	0.637
	3	0.650	0.640	
Tomato	1	0.280	0.269	
	2	0.268	0.265	0.270
	3	0.270	0.268	
Pineapple	1	0.420	0.430	
	2	0.436	0.420	0.426
	3	0.428	0.420	

RMD = 0.96%, 0.89% and 1.09% for the three Juice Samples respectively.

Determination of Lead in Canned Juices

Canned juice samples are known to contain samll amounts of Pb(II). Orange, tomato and pineapple juice cans from the local market was analysed as follows. Measured amount (~25 ml) of filtered juice (using Whatman 41 paper) is digested with 10 ml. 1:1 HNO_3. The white metastannic acid is removed by filtration. The contents are cooled, than 100 ml portion of H_2O_2 (200 ml. in case of pineapple juice serum because of larger sugar content) is added gradually dropwise. When the complete carbonaceous matter is destroyed, solution is heated to eliminate excess of H_2O_2 and then further heated to near dryness. The contents are then taken up in 10 ml triple distilled water. 1,2 and 3 ml aliquots of the solution are used for measurement. A similarly processed blank is also analyzed. Table 3 gives the amount of lead obtained by calibration and standard addition method in the three juice samples respectively.

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