



## Determination of Trace Element in Galena Ore using Polarography Techniques

### KEYWORDS

Galena, Ammonia-ammonium chloride, distilled water, Polarography, AAS

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**ABSTRACT** Galena is a main source of Silver. It is associated with other elements like Pb, Cu, Cd, Sn, Ni, and Zn, which are present in trace elements. Direct current Polarography (DCP), Differential Pulse Polarography (DPP), have been successfully used for the determination of metal in ores and mineral Polarographic and Voltammetric methods are not only accurate and sensitive but are easy to handle and economic. They are highly useful and workable under ordinary laboratory conditions. In favorable cases they have proved to be superior to other techniques which are in use for such type of a purpose. Ammonia-ammonium chloride buffer as supporting electrolyte and 0.001% gelatin as maximum suppressor in a polarographic cell and final volume were made up to 100 ml with distilled water. The sample indicated the presence of -0.06/-0.08, -0.24/-0.28, -0.40/-0.42, -0.49/-0.50, -0.61/-0.63, -0.91/-0.92, -0.14/-1.16, 1.34/-1.34-1.40/-1.44 V vs. SCE in DCP/DPP mode. Indicating the presence of Ag(I), Cu(II), Pb(II), Cd(II), Sn(II), Ni(II), Zn(II), Fe(III), and Cr(III) in ores samples. Finally the obtained polarography analysis results on galena ore sample and their comparison with atomic absorption spectroscopy Method.

### INTRODUCTION

Galena is a known source of silver (1) it is a principal mineral component in the ores of the lead-zinc deposits, is a subject of intensive and versatile mineralogical and geochemical investigations. Very often it contains increased amounts of minor and trace elements some trace elements commonly found in galena are Cd, Cu, Fe, Mn, Ni, Ag and Sn, (2) In the present modern age with the progress made in the field of electronics and instrumentation an electro analyst has at his disposal some unique techniques which are especially sensitive and accurate but are easy to handle and economic. They are highly useful and workable under ordinary laboratory conditions. In favorable cases they have proved to be superior to other techniques which are in use for such type of a purpose.

A summary of more than 550 analyses, characterizing galena from the main deposits in Madan district was given by Kolkovski et al. (3) The most important additional components in galena are silver, antimony and bismuth, their content varying in the ranges (4). Ag and Sb are relatively uniformly presented in all deposits, and their average contents for the district are the same, about 660 ppm. However, the distribution of Bi in galena is rather irregular, mainly concentrated in several deposits: Spolouka, Strashimir, Laikov Choukar, Kroushev Dol, Borieva, Gradishte, Petrovitsa, Golyam Palas. Kolkovski et al. (1996) (5) considered, that these deposits form a Bi-enriched zone, situated below the upper Ag-Sb enriched zone, and followed in depth by a sulphideless zone of barren quartz, thus outlining a vertical dome-like zoning of the ore mineralization. Some other trace-elements which are in trace elements are: Te, Se, Cd, Mo, Mn, Cu and some Zn. Sn, Ni, Co were found only occasionally as traces (6-7)

Electrochemical methods, especially anodic stripping voltammetry (ASV), are still recognized as the most convenient techniques for measuring trace heavy metals in various samples of environmental/ clinical and industrial origin contaminated water samples. (8-9) Due to the capability of pre-concentrating analysis at the surface of the working electrode, ASV allows quantification of heavy metals down to a microgram or even nanogram per liter concentration level (Guzsvány et al., 2010 and Sonthalia et al., 2004). (10-11)

The determination of traces of Zn, Pb, Cu in rum samples by anodic stripping voltammetry without previous treatment or

addition of a supporting electrolyte, using a hanging mercury drop electrode, is described. (12)

Voltammetry/polarography are widely used for the simultaneous determination of metals in natural origin samples (13-15). Other determinations for which polarography is suitable include Ni(16), Co(17), Fe(18), Mn(19) and Cd(20) in rocks and V(21) and Cu(22) in ores. Stripping voltammetry using glassy carbon fiber electrode (23) and rotating glassy carbon electrode (24) has been proposed for the trace determination.

polarographic determination of trace amounts of Co, Zn and Ni soil was done by Aleskovski (25) et al. Similarly determination of Pb, Cd, Zn and Cu in surface soil (26) Many techniques have been proposed for trace determination of metal ions in samples of different origins, however the polarographic and voltammetric methods are best suited for the said purposes. (27-29)

### EXPERIMENTAL

#### SAMPLING

The ore Samples galena was procured from the Geology department of autonomous government Science College. Jabalpur. (M.P.) According to their information they had collected the kurvet village of balaghat district. (M.P.)

#### INSTRUMENTATION

Pulse Polarograph model CL-90, which was coupled with a Polarocard model LR-108 and a three electrode assembly (Elico Private Limited, Hyderabad, India) was used for precise qualitative as well as quantitative analysis of metal ions in samples of different origins. The electrode system consisted of a dropping mercury electrode as a working electrode, a coiled platinum wire as an auxiliary electrode and a saturated calomel electrode as a reference electrode.

#### CHEMICALS AND REAGENTS

I. All the chemicals used were of Anala R/BDH grade, stock solutions of ammonium chloride, ammonium tartrate, (each 1M), Ag(I), Cu(II), Pb(II), Cd(II), Sn(II), Ni(II), Zn(II), and Cr(III) (each 0.01M) were prepared by dissolving requisite amounts of their soluble salts in double distilled water.

#### PREPARATION OF SAMPLE SOLUTION

Ig. Finely pulverized ore sample was dissolved in 10 ml of

hydrobromic acid (10N) and evaporated to dryness. The dry residue was dissolved in 10 ml of nitric acid (Pure, 16 N) and again evaporated to dryness. The dry residue was again dissolved in 10 ml of ammonium hydroxide containing 3 g of tartaric acid. The final volume was made to 100 ml with distilled water Preparation of analyte and recording of voltamograms/ polarograms

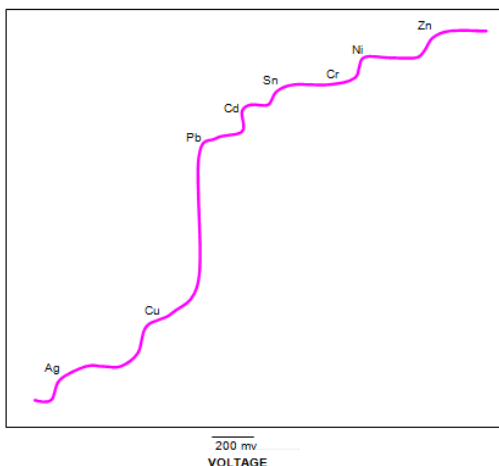
In the field of trace analysis, procedure for preparation of analyte play a very important role, it should be simple convenient, should not leave any contaminating element in the analyte and should not destroy the ions of interest at any level.

The sample solution (10 ml) was mixed with 0.1 M ammonia-ammonium chloride buffer as supporting electrolyte and 0.001% gelatin as maximum suppressor in a polarographic cell and final volume was made up to 100 ml with distilled water. The pH of the test solution was adjusted to  $8.0 \pm 0.02$  with ammonia solution. Pure nitrogen gas was bubbled through the test solution for 15 min before recording the polarogram.

**RESULT AND DISCUSS**

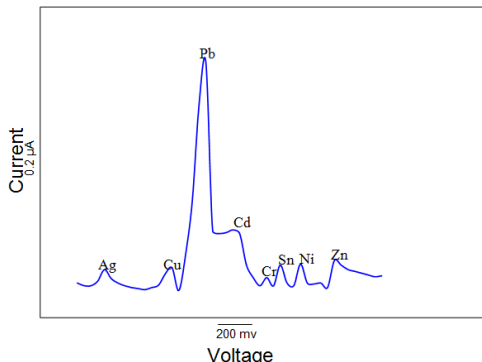
The DC and DP Polarograms of the sample saluting (Fig 3.1 a, b) showed Eight well defined waves / peaks with  $E_{1/2} / E_p$  values = -0.06/-0.08, -0.24/-0.28, -0.40/-0.42, -0.49/-0.50, -0.61/-0.63, -0.91/-0.92, -0.14/-1.16, -1.40/-1.44 V vs. SCE in DCP/DPP mode. Indicating the presence of Ag(I), Cu(II), Pb(II), Cd(II), Sn(II), Ni(II), Zn(II), and Cr(III) in the ore sample.

**DIRECT CURRENT POLAROGRAPH**



To confirm which increased the observed wave / peak height of each metal ion signal without any change in  $E_{1/2} / E_p$  values. The linear relationship between the concentration of each metal ion and the corresponding wave / peak height was also unchanged confirming the possibility of an accurate simultaneous qualitative and quantitative determination of the metal ions in the sample. They Produced well defined waves/ peaks with same  $E_{1/2} / E_p$  values. Quantitative analysis of the sample using DCP and DPP of Ag, Cu, Pb, Cd, Sn, Ni, Zn, Fe and Cr in the sample were carried out by using DCP and DPP methods. Spiked samples were prepared in order to evaluate the concentrations of each metal ion, the results are Given in

**DIFFERENTIAL PULSE POLAROGRAPH**



**Table – 1.1 : RESULTS ON THE GALENA ORE SAMPLE (mg/100 mg)\*FOR IT'S MEAL CONTENT**

| Metal Ion | Parameter | By DCP |       |        | By DPP |        |        |
|-----------|-----------|--------|-------|--------|--------|--------|--------|
|           |           | Added  | Found | % Rec. | Added  | Found  | % Rec. |
| Ag(I)     | Amount    | -      | 0.015 | -      | -      | 0.018  | -      |
|           |           | 0.012  | 0.027 | 0.012  | 0.012  | 0.030  | 100    |
|           | % Rec.    |        | 100   |        |        | 100    |        |
|           | S.D. **   |        | 0.002 |        | 0.001  |        |        |
| Cu(II)    | Amount    | -      | 1.43  | -      | -      | 1.270  | -      |
|           |           | 1.27   | 2.70  | 1.270  | 1.270  | 1.270  | 99.2   |
|           | % Rec.    |        | 99.2  |        | 99.2   | 99.2   |        |
|           | S.D. **   |        | 0.011 |        | 0.001  |        |        |
| Pb(II)    | Amount    | -      | 84.04 | -      | -      | 71.55  | -      |
|           |           | 62.160 | 146.2 | 62.16  | 62.16  | 62.16  | 99.4   |
|           | % Rec.    |        | 99.3  |        | 99.4   | 71.55  |        |
|           | S.D. **   |        | 0.020 |        | 0.02   | 133.71 |        |

|        |         |       |       |        |       |       |        |
|--------|---------|-------|-------|--------|-------|-------|--------|
| Cd(II) | Amount  | -     |       | 0.16   | -     |       |        |
|        |         | 0.124 |       | 0.28   |       |       | 0.186  |
|        | % Rec.  |       | 99    |        | 0.124 | 99.2  | 0.310  |
|        | S.D. ** |       | 0.004 |        |       | 0.003 |        |
| Sn(II) | Amount  | -     |       | 0.0125 | -     |       |        |
|        |         | 0.125 |       | 0.03   |       |       | 0.014  |
|        | % Rec.  |       | 99.2  |        | 0.125 | 99.1  | 0/0139 |
|        | S.D. ** |       | 0.003 |        |       | 0.001 |        |
| Cr(II) | Amount  | -     |       | 0.025  | -     |       |        |
|        |         | 0.03  |       | 0.05   | 0.03  |       | 0.05   |
|        | % Rec.  |       | 99.1  |        |       | 99.6  | 0.08   |
|        | S.D. ** |       | 0.004 |        |       | 0.003 |        |
| Zn(II) | Amount  | -     |       | 0.015  | -     |       |        |
|        |         | 0.065 |       | 0.080  |       |       | 0.014  |
|        | % Rec.  |       | 100   |        | 0.065 | 100   | 0.079  |
|        | S.D. ** |       | 0.004 |        |       | 0.002 |        |
| Ni(II) | Amount  | -     |       | 0.12   | -     |       |        |
|        |         | 0.015 |       | 0.135  |       |       | 0.017  |
|        | % Rec.  |       | 99.6  |        | 0.015 | 100   |        |
|        | S.D. ** |       | 0.007 |        |       | 0.006 | 0.032  |

The results indicated that the percentage recovery is over 99% for all the metal ions, with high accuracy and precision of the determination.

TABLE – 1.2 FINAL RESULTS ON GALENA SAMPLE AND THEIR COMPARISON WITH AAS

| Metal ion | Polarographic                        | AAS    |
|-----------|--------------------------------------|--------|
|           | ( mg g <sup>-1</sup> of the sample ) |        |
| Ag (I)    | 0.15                                 | 0.16   |
| Cu (II)   | 14.3                                 | 15.03  |
| Pb (II)   | 840.40                               | 840.40 |
| Cd (II)   | 1.65                                 | 1.60   |
| Sn(II)    | 0.125                                | 0.123  |
| Cr (III)  | 0.25                                 | 0.25   |

|         |      |      |
|---------|------|------|
| Ni (II) | 0.15 | 0.16 |
| Zn (II) | 1.12 | 1.10 |

#### CONCLUSION

The final analysis results for the sample. The comparative data of Polarographic and AAS data are in good agreement for the Cu, Pb, Cd, Sn, Ni, Zn and Cr content of the sample but AAS failed to determine Ag under existing situations. However the statistical data speaks the superiority of the polarographic method for such an analysis over AAS method. The statistical and percentage recovery data also support the above.

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