Suggested Standard Test Method for Laboratory Evaluation of Zinc Sacrificial Anode Test Specimens

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Abstract - *The ASTM (American Society for Testing and Materials) standard test method for laboratory evaluation of Magnesium Sacrificial Anode test specimens is available as ASTM International Standard: G97-97(Re-approved 2007). But for laboratory evaluation of Zinc Sacrificial Anode test specimens, the ASTM or any other standard test method is not available as of today. So herewith, an attempt is made to suggest Standard Test Method for Laboratory Evaluation of Zinc Sacrificial Anode Test Specimens. This test method is intended to be used for quality assurance by anode manufacturers or anode users.*

Key Words: Zinc Sacrificial Anodes; Saturated Calomel reference electrode; Copper Coulometer with Anodes; Ampere hours; Current efficiency; Uncertainty analysis

1. INTRODUCTION

While working on Doctoral Degree, on the topic "Cathodic Protection of Embedded Steel bars in Concrete by Sacrificial Anodes", my main emphasis was on Sacrificial Zinc Anodes. Hence it is evident that purity of zinc anode is most important aspect of my study. Research review revealed that there is no standard test method for evaluation of sacrificial zinc anodes by ASTM or any other international standard. Therefore, an attempt is made to suggest a test method for laboratory evaluation of sacrificial zinc anode test specimens.

2. EXPERIMENTAL WORK

For conducting experimental work on Zinc Anodes Test Specimens, the basis of ASTM (American Society for Testing and Materials) international standard, Designation: G97-97(Reapproved 2007) titled as Standard Test Method for Laboratory Evaluation of Magnesium Sacrificial Anode Test Specimens for Underground Applications is referred for Scope, Summery of Test Method, Apparatus, Reagents, Precautions, Specimen Preparation, and Procedure. Calculations and Report is done as per the values obtained for Zinc sacrificial anodes. Modification made in the referred standard is as follows:

Reagents- Test Electrolyte Saturated Sodium Sulfate-Zinc Oxide Solution- Add 5.0g of reagent grade $Na_2SO_4.2H_2O$, 0.1 g of reagent grade ZnO_2, 2% NaCl to 3000mL of type IV or better reagent grade or distilled water.

Precision and Bias- Reproducibility of results to be compared with other labs is out of scope for this study.

3. CALCULATIONS & REPORTS

Zinc Anode-
Diameter = 11.5 mm
Effective Length = 100 mm
Effective Area = 37.15 cm^2
Current density of Zn = 0.039mA/cm^2
So, Current applied = 0.039 × 37.15
= 1.4488
= 1.45mA*

*As per referred standard for Mg, current applied is 1.6mA, but as per above calculations for Zn, it is 1.45mA

The experiment was carried out over the period of 14 days and readings obtained are recorded in following table:

S. No.	Initial Weight 21/03/2013 gm	Final Weight 04/04/2013 gm	Difference in Weight gm	Loss/Gain	OCP volts		
					1st day	7th day	14th day
Zinc Anodes							
Pot 1	114.1693	113.5642	0.6051	Loss	-0.698	-0.697	-0.692
Pot 2	107.7736	107.1635	0.6101	Loss	-0.695	-0.693	-0.685
Pot 3	102.4363	101.8647	0.5716	Loss	-0.710	-0.708	-0.706
Pot 4	106.0892	105.5172	0.5720	Loss	-0.703	-0.697	-0.693
Pot 5	108.0262	107.4671	0.5595	Loss	-0.702	-0.697	-0.693
Copper Anodes							
Wire A	0.9252	1.5402	0.6150	Gain			
Wire B	1.0065	1.6107	0.6042	Gain			
Strip A1	71.9464	71.7560	0.1964	Loss			
Strip A2	70.9680	70.0165	0.9515	Loss			
Strip B1	71.4567	70.7802	0.6765	Loss			
Strip B2	71.2405	70.5981	0.6424	Loss			

Average current applied = 1.45 mA

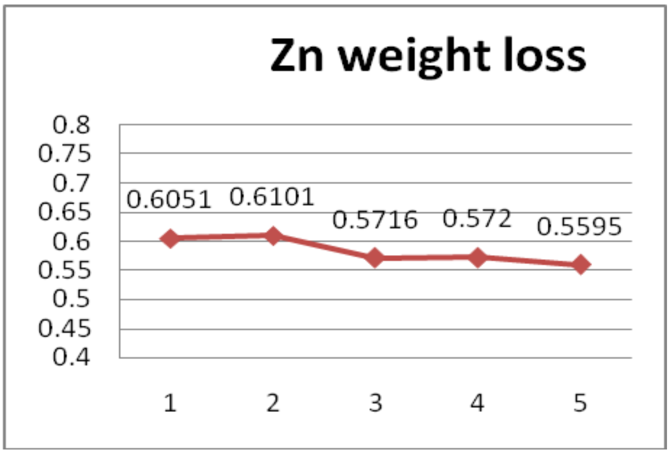
Ampere Hours-
Current = 1.45mA
Hours = 14 × 24 + 12 = 348
Ampere hours = 348 × 1.45 × 10^{-03}
= 0.5046 ....Used

Std. Equivalent Wt. of Zinc = Molecular wt./ Valency
= 65.38/2
=32.69gm.

Std. hypothetical wt. loss for Zinc coulombs consumed (constant) = 96500 Amp. Sec.

= 95500/3600
= 28.6Amp. Hrs.

Hence,
Standard weight loss Zinc
For 32.69gm = 26.80Amp. Hrs.
? = 0.5046Amp. Hrs.
Std. wt. loss = 0.6154gm.



Current efficiency calculations-

Current efficiency = Actual Wt. loss / Std. Wt. loss
So, Current efficiencies of Zinc Anodes used in,

Pot 1 = (0.6051/0.6154) × 100
=98.37%
Pot 2 = (0.6101/0.6154) × 100
=99.13%
Pot 3 = (0.5716/0.6154) × 100
=92.92%
Pot 4 = (0.5720/0.6154) × 100
=92.99%
Pot 5 = (0.5595/0.6154) × 100
=91.70%

Average Current efficiency = 95.032%

4. RESULTS

(a) Average current efficiency for zinc anodes = 95.032%

(b) Average OCP (Open Circuit Potential) = -0.6938

(c) Ah/g value = 0.0448

(d) Average difference of OCP values = 0.0091V (as per uncertainty analysis discussed below)

5. DISCUSSIONS

5.1 Uncertainty Analysis: Precision and Bias

- The following criteria should be used to judge the acceptability of results at the 5% significance level.

- Repeatability- Results (on identical test specimens) by the same operator should be considered suspect if the mean from five specimens differs by more than the following from the mean obtained from a duplicate test on five specimens.

Ah/g value	0.06Ah/g
Open circuit potential	0.02V

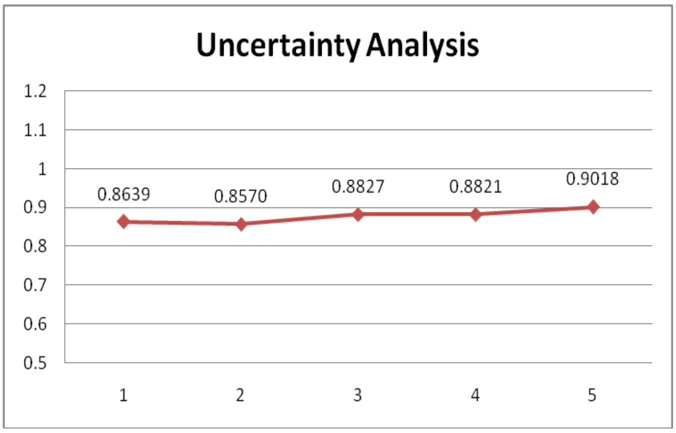
5.2 Uncertainty Analysis Calculations

- Zinc Anodes-
  Calculation of Ah per mass loss for each test specimen:

  $Ah/g = (Ah)/(M_{Zn1} - M_{Zn2})$
  Where:
  M_{Zn1} = initial mass of zinc test specimen, g, and
  M_{Zn2} = final mass of zinc specimen, g.

1. Ah/g = 0.5046/0.6051
= 0.8639
2. Ah/g = 0.5046/0.6101
= 0.8570
3. Ah/g = 0.5046/0.5716
= 0.8827
4. Ah/g = 0.5046/0.5720
= 0.8821
5. Ah/g = 0.5046/0.5595
= 0.9018

Repeatability= Max. Ah/g Value – Min. Ah/g Value
= 0.9018 – 0.8570
= 0.0448 (which is well below permissible 0.06)



OCP = (-0.706) – (-0.693) Volts
= -0.013 Volts (which is well below permissible 0.02)

Copper Anodes-
Copper coulometer, the Ah calculation is :

$$Ah= (0.8433Ah/g\ Cu)\ (M_2-M_1)$$

Where:
M_2= final mass of copper coulometer wire gm, and
M_1= initial mass of copper coulometer wire gm.

If an electronic coulometer is used, the Ah calculation is as follows:

Ah=(ampere second measurement from coulometer)/(3600)
Coulometer A

$$Ah = 0.8433 \times (1.5402 - 0.9252) = 0.5186$$

Coulometer B

$$Ah = 0.8433 \times (1.6107 - 1.0065) = 0.5095$$

Repeatability= Max. Ah Value – Min. Ah Value
= 0.5186 – 0.5095
= 0.0091 (which is well below permissible 0.02)

6. CONCLUSIONS

1. Current efficiency for pure Zinc anodes ranges above 95%, hence obtained results are satisfactory and experimentation is successful.

2. As OCP (Open Circuit Potential) as per *table-1* are negative, which indicates that purity of Zinc anodes is high and anode efficiency is high.

3. Ah/g value obtained is 0.0448, which is well below permissible value of 0.06 as per uncertainty analysis.

4. OCP value obtained is 0.0091V, which is well below permissible value of 0.02 as per uncertainty analysis.

5. This is a suggested laboratory evaluation method for zinc sacrificial anodes, for further consideration and evaluation by international standards.

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- Mr. Mourya (KCPL)
- Mr. Pravin (KCPL)
- Mr. Sunil Chavan (KCPL)
- Akhilesh Shinde
- Mrs. Kaveri Shinde

8. REFERENCES

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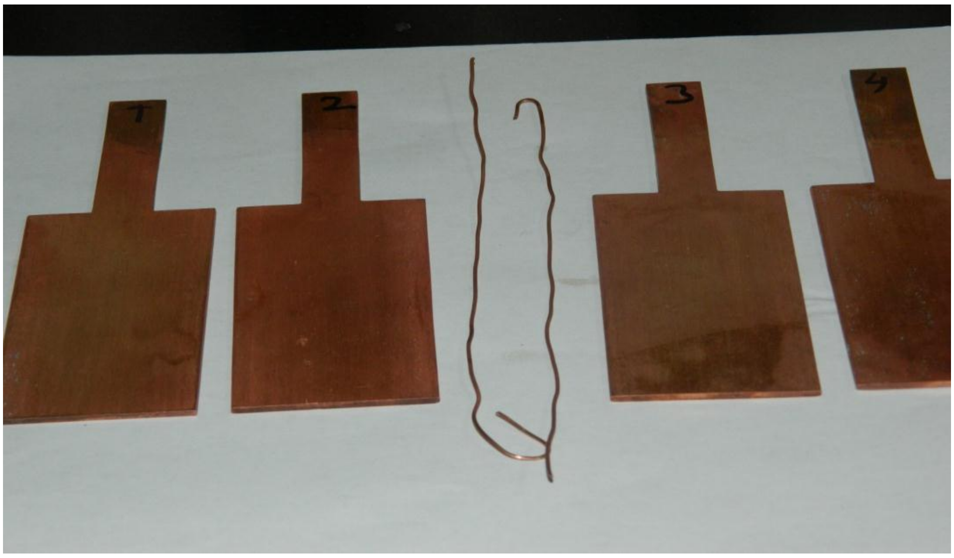
9. SUPPLEMENTARY DATA

Actual photographs of experimental setup, zinc anodes and other apparatus are as follows:

M S Test Pots

M S Test Pots cleaning

Cu Coulometer Beaker



Copper Coulometer Anodes

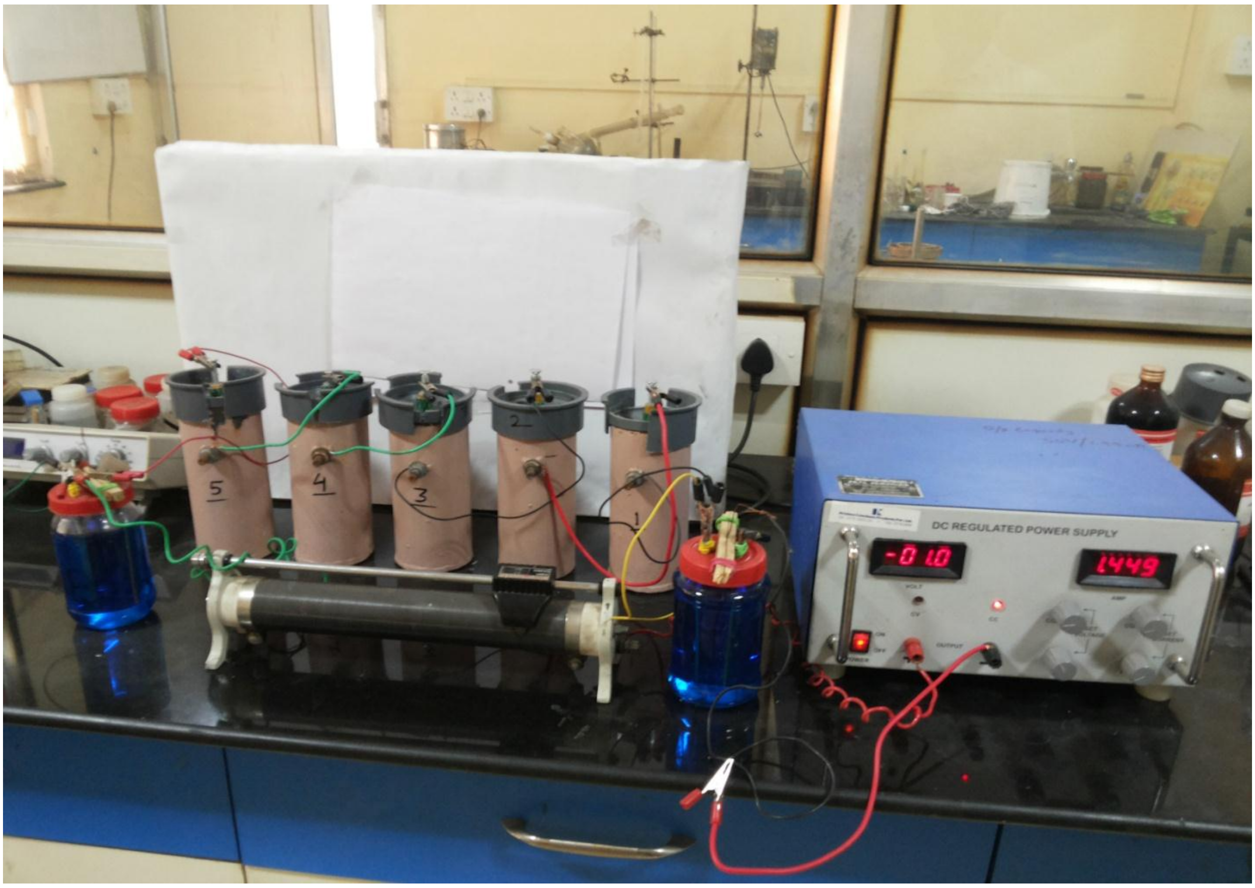
Calomel Referance Electrode

Drying Oven

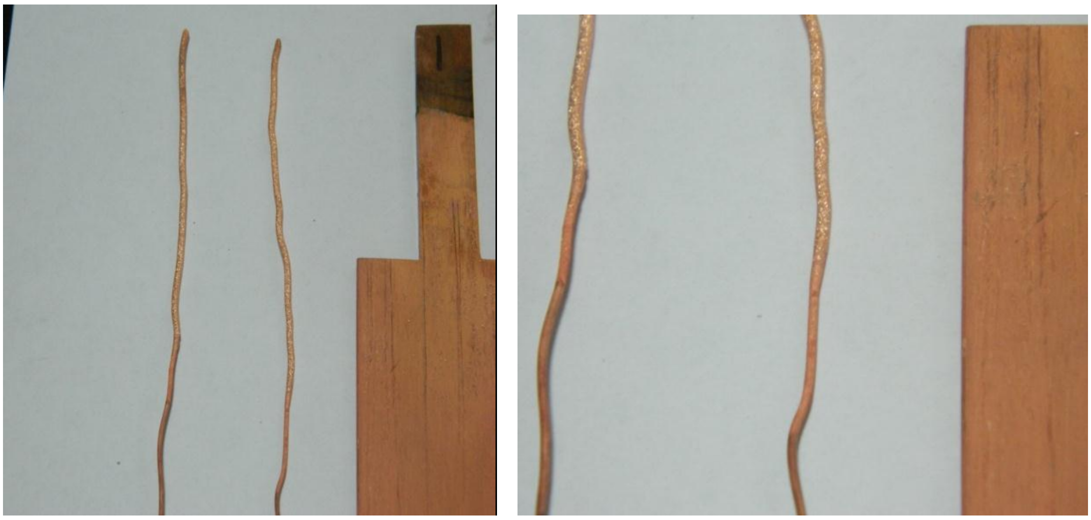
Electronic Balance

D C Power Source

Zinc Anodes



Experimental Set-up



Weight gain by Cu wires