



Electrochemical Analysis of Beryllium Using DC Polarography and Differential Pulse Polarography in an Oxalic Acid Medium

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ABSTRACT

To see how beryllium acts in an electric field, we employed direct current (DC) polarography and differential pulse polarography (DPP) with a falling mercury electrode. Oxalic acid was employed to keep beryllium ions stable and stop hydrolytic interferences. The effects of supporting electrolyte, oxalic acid concentration, pH, beryllium concentration, and mercury column height were systematically studied. When the conditions were just right (0.08 M oxalic acid, pH 2.7), beryllium showed a definite reduction wave with a half-wave potential of -1.92 V compared to a saturated calomel electrode (SCE). Differential pulse polarography showed higher sensitivity, which was shown by a clear peak at -1.86 V. The linear correlation between diffusion, peak currents, and concentration indicates that the electrode process is controlled by diffusion. The method that was created worked well on samples of mining waste, showing that it was very selective and reliable for analysis.

Keywords: DC polarography, Differential pulse polarography, Oxalic acid, Dropping mercury electrode.

INTRODUCTION

People utilise beryllium a lot in the aerospace, nuclear, electronics, and metallurgy areas because it is incredibly strong. But since it is bad for you, it is crucial to locate it in very small amounts in samples from the environment and industry. Most of the time, standard analytical methods require expensive and specialised tools. Electroanalytical techniques, particularly polarographic methods, provide a sensitive, selective, and economical approach for the analysis of metal ions.[1]

The way metal ions act in polarography is greatly affected by complex formation, supporting electrolyte, and solution pH. Oxalic acid forms stable compounds with beryllium, which inhibits hydrolysis and improves wave characteristics. The present work underscores the systematic DC polarographic and DPP studies of beryllium in an oxalic acid medium, evaluating the method's appropriateness for quantitative determination in real samples.[2]

MATERIALS AND METHODS

Chemicals and Solutions

All of the chemicals used were of analytical reagent grade. We used water that had been distilled twice to make beryllium stock solutions. We generated new oxalic acid solutions with varied quantities of the acid in them. Potassium chloride and acetate buffer solutions worked as extra electrolytes. To get rid of any dissolved oxygen, all of the solutions were flushed with high-purity nitrogen gas before any measurements were taken.[3-5]

Tools and Situations

To take polarographic measurements, we employed a microprocessor-based pulse polarographic analyser with a falling mercury electrode as the working electrode and a saturated calomel electrode as the reference. All testing was conducted at room temperature.[6,7]

METHODS

Effect of the Supportive Electrolyte

The kind of supporting electrolyte had a considerable effect on how beryllium acted in polarography. Oxalic acid generated the most stable and clearly defined reduction wave among the media analysed, due to strong complex formation (Table 1).[8]

The DC curve represent in Fig 1.

Effect of Oxalic Acid Concentration

Increasing oxalic acid concentration resulted in a gradual shift of the half-wave potential towards more negative values, indicating enhanced complex formation between Be(II) and oxalate ions. An oxalic acid concentration of 0.08 M was found optimal (Table 2).[9]

Effect of pH

The shape and stability of the wave changed a lot depending on the pH of the fluid. Waves were not well defined at lower pH levels because the complexation was not complete. Hydrolysis caused waves to be warped at higher pH levels.[10] A smooth sigmoidal wave was created at pH 2.7.

Table 1: Effect of supporting electrolyte on of Be(II)

Medium	$-E_{1/2}$	I_d (μA)
0.1 M KCl + 0.1 M Oxalic acid	2.00	3.88

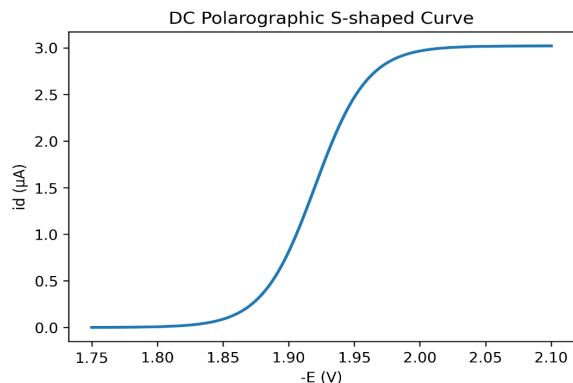


Figure 1: DC polarographic sigmoidal wave of Be (II)

Table 2: Effect of oxalic acid concentration

Oxalic acid (M)	$-E_{1/2}$ (V)	I_d (μA)
0.09	1.93	3.90
0.11	1.96	3.35
0.12	1.98	3.96

The diffusion current at half-wave potential was almost half of the limiting current, demonstrating that diffusion (Table 3).[11]

How the Height of the Mercury Column Affects

The limiting current increased in direct proportion to the square root of the mercury column height, so confirming the Ilković equation and illustrating diffusion-controlled behaviour (Table 4).

Differential Pulse Polarography study

Under optimal conditions, the differential pulse polarograms of beryllium exhibited a pronounced, symmetrical peak at -1.86 V relative to SCE. The peak current went up in a straight line when the concentration went up, which showed that it was more sensitive than DC polarography. Table 5 shows the DPP determination of the Be (II) curve shown in Figure 2.[12,13]

Interference Studies

There was no effect from typical alkali and alkaline earth metals. Transition metal ions created peaks that were easy to discern from one another. Oxalic acid in excess was able to effectively block any modest interference from Al(III) and Fe(III) at larger concentrations (Figure 3).[14-16]

Table 3: Effect of pH on polarographic parameters

pH (Supporting Electrolyte)	$-E_{1/2}$ (V)	I_d (μA)
2.8	1.92	3.02
3.0	1.93	3.05
3.2	1.94	3.06

Table 4: Effect of mercury column height

h (cm) (mercury column)	\sqrt{h}	i_l (μA)
35	5.91	0.230
40	6.32	0.240

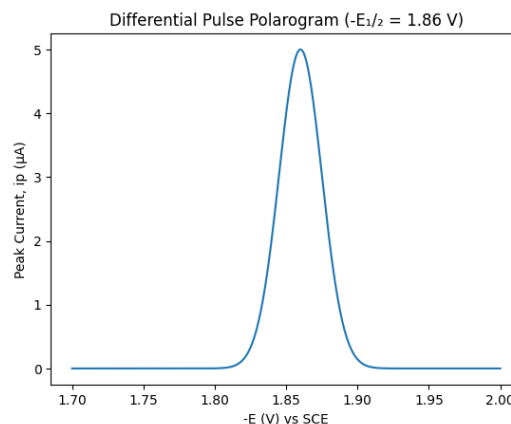


Figure 2: DPP curve

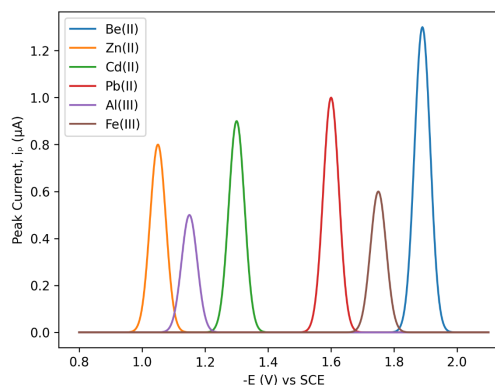


Figure 3: Effect of coexisting metal ions on DPP determination of Be (II)

Table 5: Comparative determination of beryllium in industrial (mining wastewater) samples by different analytical techniques

S. No.	Industrial Area sample	Beryllium conc. industrial sample		
		UV-visible	Graphite AAS	ICP-MS
1.	Mining waste water	Be-Erichrome Complex $\lambda = 537$ nm	0.09 $\mu g/L$	0.2 $\mu g/L$

Samples Analysis

We obtained samples of mine waste, filtered them, digested them in acid, adjusted the pH, and looked at them under the best DC and DPP circumstances. The method showed that it could accurately and consistently find beryllium. Table 5 represents various analytical methods for the identification of beryllium metal.

CONCLUSION

The new DC polarographic and differential pulse polarographic technologies are simple to use, very sensitive, and very good at finding

beryllium. Oxalic acid helps beryllium stay stable and makes waves easier to see. Differential pulse polarography is a more sensitive method that works well for looking at small amounts of environmental and industrial materials.

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