

Pyrrolidindithiocarbamate as a Reagent for Electro Analysis

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Abstract

Pyrrolidinedithiocarbamate have been extensively studied and reported as a complexing reagent for a large number of metal ions. The present paper deals with the result of amperometric titration of titled metal ion (sodium selenite) with PDTC which is a amperometric reagent. PDTC has advantages as an analytical reagent for metal analysis because of its complexation in acidic media. The electrochemical studies (polarography) of P.D.T.C. were described in this paper.

Keywords: PDTC, Amperometry, Sodium Selenite.

Introduction

Polarography and Voltammetry are the names of analytical methods based on current potential measurements in the electrochemical cell. The analytical signal is the current, normally a faradic current, which flows through the cell during the reaction of the analyze at the working electrode with a small surface¹⁻⁸. The analyze may be a cation, an anion or a molecule. Amperometry is a highly sensitive and convenient analytical technique. The electrode used in amperometric titrations must be polarized. Amperometric titrations have even wider range of applications than polarography because even electro-inactive substances can be determined using electro-active titrant. Electrochemical studies of dithiocarbamates at mercury electrodes have been conducted. Generally redox behavior associated with a ligand is very much dependent on the nature of the electrode, the solvent and the experimental conditions used. A comprehensive study of the dithiocarbonates in non – aqueous media⁸⁻¹⁰ has shown that reversible redox processes are common and that the measured potentials are dependent on both the metal and the alkyl groups in dithiocarbamate complexes. Many workers have since confirmed that Ferric dithiocarbamates are readily oxidized and reduced by electrochemical methods in a range of solvents¹⁰⁻¹².

Aim of the Study

To estimate the voltametry study with newear reagent and this mathod uses for clinical use like estimation of blood or tissue samples.

Electrochemical Behavior of Pyrrolidine Dithio Carbamate (PDTC)

Electro chemical studies of PDTC at mercury electrodes have been conducted¹²⁻¹³, generally redox behavior associated with a ligand is very much dependent on the nature of the electrode, the solvent and the experimental conditions used. During such studies, the mercury complexes form at the mercury electrode, which suggests that oxidation of the electrode, occurs in preference to oxidation of the Dithio Carbonate ligand. A comprehensive study of the electro chemistry of the first row transition metal^{8,11} has shown that reversible redox processes are common and that the measured potentials are dependent on both the metal and the alkyl groups in dithiocarbonate complexes.

Reagents

0.001M solution of PDTC was prepared in Doubly Distilled water. PDTC was also recrystallized at room temperature from a solution of ethyl alcohol by adding ether. Buffer solution of pH 5.3 ± 1.2 were prepared, pH of solution also adjusted through dil. Sodium hydroxide and hydrogen chloride. 0.01 KC1 also prepared in Distilled H₂O.

Procedure

A Polarographic cell containing 20 m.l. of KC1 and 20 m.l. of PDTC solution. Add 0.1 M freshly prepared gelatin solution (Maximum supressiour). Add Buffer solution in this cell. This solution is deoxygenated with the stream of nitrogen and the Polarographed from – 0.2 to + 0.3 volt.

Result and Discussion

In buffer solution of pH 5.3 ± 1.2 a definite polarographic wave is exhibited when id is plotted against concentration, a linear relationship results. The half-wave potential becomes more negative with increasing

concentration so measurements of limiting current cannot be made between set potentials. The half wave potential of PDTC is 0.351 and id is 2.7. The electrochemical data for dithiocarbonate complexes are consistent with the concept of O-donor properties dominant over weak π -back donation thereby producing complexes that are relatively easy to oxidize and conversely hard to reduce.

Electrochemical measurements have been established that the oxidation and reduction potentials for PDTC with metals. The redox potentials of the complexes are dominated by electron density at the metal center rather than by differences in electron configuration of the metal ion.

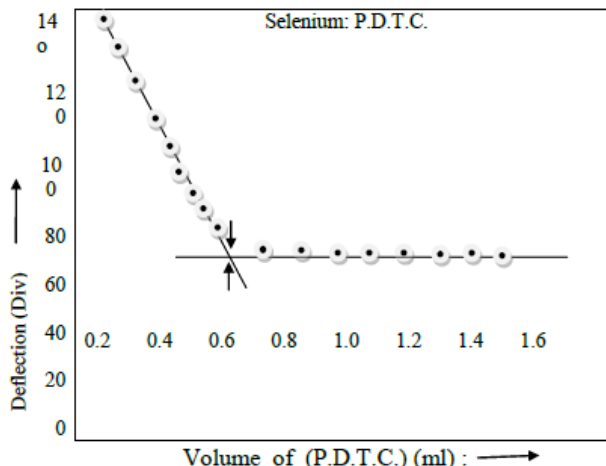
Pyrrolidine-Di-thio Carbonate as an Amperometric Reagent

A typical polarographic wave of P.D.T.C. is shown in fig. at pH 5.3 ± 1.2 in 0.1 M potassium chloride. It was observed that the diffusion current is proportional to the P.D.T.C. concentration. Hence P.D.T.C. has been successfully employed as an amperometric reagent for the precise determination of titled metals. For amperometric estimation of the metal (sodium selenite) using P.D.T.C. as the reagent, experimental sets were prepared keeping known concentration of P.D.T.C. in 0.1 M KCl at pH 5.3 ± 1.2 . The Plateau potential of P.D.T.C. wave i.e. -0.23 V vs SCE was applied Amperometric titration were performed in the usual way by adding standard solution of metal ion. A dark-green precipitation was observed in the test solution. The current was noted with multi-flex galvanometer. The reverse L Shape curve was obtained because metals are reduced under the experimental conditions. This curve is resulted for the current-volume plots. The result of amperometric titrations with P.D.T.C. are shown in table.

Amperometric Determination of Selenium (IV) with P.D.T.C.

Plateau potential = -0.23 V vs. SCE
 pH = 5.3 ± 1.2 .
 Ionic Strength (μ) = 0.1 M (KCl)
 Temperature = $28.0 \pm 1.0^\circ\text{C}$

S. No.	Approximate Concentration (M)	Amount of Se(IV)		Percentage Error (%)
		Taken (mg)	Found (mg)	
1.	2.10×10^{-4}	0.1667	0.1662	-0.42
2	4.20×10^{-4}	0.3330	0.3350	+0.60
3	6.30×10^{-4}	0.5000	0.5020	+0.40
4	8.40×10^{-4}	0.6660	0.6600	-0.45
5	1.00×10^{-3}	0.8330	0.8300	-0.36
6	1.40×10^{-3}	1.1670	1.1720	+0.43
7	1.90×10^{-3}	1.5000	1.5100	+0.66
Standard Deviation			= ± 0.016	
Coefficient of variation			= 1.10	



Concentration of Se (IV) = 0.04 mm/ml,
 P.D.T.C. = 0.06 mm/ml

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