

and then reappeared and persisted at much higher temperatures. As a general rule more extensive carbonizing results in metal ion emission at higher temperatures. When filaments were carbonized for 20 min. or longer at 2200°, no U^+ ions were observed until the temperature exceeded about 1800°. Then UC_2^+ ions were observed also but at an intensity factor of 100 below that of the U^+ ions.

It seems that metal ions may arise from several parent molecules. It is probable that they come from uranium metal (low temperature emission) and from one or more refractory carbides (high temperature emission). Positive identification of UC_2^+ ions was made by mass determination and by resolution of the two uranium isotopes in the sample. It is likely that UC and possibly U_2C_3 exist on the filaments also, even though they were not observed as stable gaseous species.⁶ The temperature at which metal ions are emitted is consistent with the known physical properties of the metal and carbides.⁷

The oxide ions seem to have a variety of parents also. The trioxide may be present on the filament after evaporation of the sample and be lost at relatively low temperatures. It is formed also from UO_3 by an air leak. As CO_2 is reduced, CO^+ ions are formed. In addition, UO^+ ions are produced when air is leaked into a source which has been emitting metal ions only. Prolific emission of both UO^+ and UO_2^+ ions may be obtained by such an air leak. In one experiment UO^+ ions first appeared. Then UO_2^+ ions appeared and grew to an intensity one hundred times that of the original metal ions. At the same time the metal ion beam increased by a factor of five before it decreased and eventually disappeared. It is probable that the intense oxide

beams produced by an air leak are associated with the burning of the highly reactive uranium carbides. Oxide ions produced in this manner can be observed at temperatures far below that at which metal ions can be produced from the same source in the absence of the leak.

Preliminary experiments with 0.1 microgram samples of the alpha emitting U^{232} suggest that well over 90% of the sample may be lost before the temperature for maximum metal ion emission is reached. Apparently, the uranium is lost during reduction before the more refractory carbides are formed. Experiments are under way to devise procedures to minimize these losses. Multiple filament assemblies will be used to investigate the problem. With a multiple filament it should be possible to separate some of the surface effects from the chemical effects. For example, the effect of the carbonizing procedure on the work function of the surface is unknown but is susceptible to study by the multiple filament technique.

Conclusions

Single filament ion sources of tungsten or rhenium will not produce uranium metal ions in the absence of reducing agents other than the filament metals. Deposition of carbon before, during, or after deposition of the sample will ensure subsequent production of metal ions to the exclusion of oxide ions unless the pressure is high. It is probable that the success of single filament sources in the analysis of uranium as metal ions is due to the formation of refractory carbides which decompose at high temperatures.

POLAROGRAPHY OF SOME METAL COMPLEXES WITH TRIETHYLENETETRAMINE

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Received August 17, 1967

The complexes of cadmium, copper, lead, zinc, nickel and cobalt with triethylenetetramine have been studied by means of the dropping mercury electrode. The cadmium, copper and lead complex are reversibly reduced to the amalgam at any concentration of the reagent. In alkaline medium the divalent cobalt complex is easily oxidized by air to a red-colored trivalent cobalt complex. The nickel, zinc and trivalent cobalt complex showed an irreversible reduction at the D.M.E. The half-wave potentials and the diffusion current constants of the various complexes are given.

Introduction

Triethylenetetramine (abbreviated "trien") forms stable complexes with certain metals. The composition and stability of the complexes have been determined by potentiometric titration.^{1,2} The cadmium complex also has been studied by means of the dropping mercury electrode by Douglas, *et al.*³ They claim that the complex is irreversibly reduced at trien concentrations less than 0.01 *M*. More recently the polarography of the copper complex has been thoroughly investigated by Jonassen and co-workers.⁴

Hitherto, the polarographic behavior of the complexes formed with other metals has not been investigated. The present paper is an extensive study of the polarography of the cadmium, lead,

copper, zinc, nickel and cobalt complexes formed with trien.

Experimental

Materials.—The technical grade triethylenetetramine, obtained from Fluka A. G., Switzerland, was purified as described by Reilley and Schmid.⁵ The remaining chemicals were reagent grade and used without further purification. Approximately 0.1 *M* stock solutions of the metal salts were prepared by dissolving 0.1 mole of the corresponding salt in redistilled water and diluting to one liter. The stock solutions were standardized by complexometric titration with EDTA. Standard solutions of trien were prepared and standardized following the procedure given by Reilley and Skeldon.⁶ Five-trites molar phosphate buffer was used as supporting electrolyte. The buffer was prepared by adding potassium hydroxide to phosphoric acid, and its pH measured with a pH meter. In order to avoid precipitation of metal phosphates, 1 *M* ammonia buffer was used as indifferent electrolyte for the lead, zinc and nickel complexes. The pH of the electrolyte was adjusted to the desired value by adding hydrochloric acid to the ammonia solution. Triton X-100, obtained from Rohm and Haas Co., Philadelphia, was found to be effective as maximum suppressor.

Apparatus and Technique.—Polarograms were recorded

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Polarography Of Metal Complexes

Ian S. Longmuir



Polarography Of Metal Complexes:

Polarography of Metal Complexes David Richard Crow, 1969 Introduction to Polarography and Allied Techniques Kamala Zutshi, 2006 This book is divided into two parts. The first explains the basic principles and techniques involved in polarography. It also explains polarography of metal complexes and organic compounds. The second part is devoted to allied techniques. It explains miscellaneous polarographic methods and their analytical applications. Advances in D.C. polarography and the principles and control of corrosion are also highlighted in this section. A simple and systematic approach is followed throughout the book. Suitable references are provided at the end of each chapter and review questions are included at the end of the book. The book would serve as a useful text for B.Sc. Hons and M.Sc. Chemistry students.

Metal Complexes in Aqueous Solutions Arthur E. Martell, Robert D. Hancock, 2013-06-29 Stability constants are fundamental to understanding the behavior of metal ions in aqueous solution. Such understanding is important in a wide variety of areas such as metal ions in biology, biomedical applications, metal ions in the environment, extraction, metallurgy, food chemistry, and metal ions in many industrial processes. In spite of this importance, it appears that many inorganic chemists have lost an appreciation for the importance of stability constants and the thermodynamic aspects of complex formation. With attention focused over the last thirty years on newer areas such as organometallic chemistry, this book is an attempt to show the richness of chemistry that can be revealed by stability constants when measured as part of an overall strategy aimed at understanding the complexing properties of a particular ligand or metal ion. Thus, for example, there are numerous crystal structures of the Li ion with crown ethers. What do these indicate to us about the chemistry of Li with crown ethers? In fact, most of these crystal structures are in a sense misleading in that the Li ion forms no complexes or at best very weak complexes with familiar crown ethers such as 12-crown-4 in any known solvent. Thus, without the stability constants, our understanding of the chemistry of a metal ion with any particular ligand must be regarded as incomplete. In this book, we attempt to show how stability constants can reveal factors in ligand design which could not readily be deduced from any other physical technique.

POLAROGRAPHY OF METAL COMPLEXES. DR. CROW, 1969 **Metals Speciation Separation and Recovery** James W. Patterson, 1987-07-05 This comprehensive book covers metals chemistry, separation chemistry, and metals separation processes. State-of-the-art papers give new and recent developments and future research needs.

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Electrochemistry of Biological Molecules Glenn Dryhurst,2012-12-02 Electrochemistry of Biological Molecules presents a fairly complete summary of the electrochemistry of the more important groups of nitrogen heterocyclic molecules including purines and pyrimidines and their nucleosides and nucleotides polynucleotides and nucleic acids pteridines flavins pyrroles porphyrins and pyridines Topics covered range from the theory and instrumentation of electrochemistry to various biological molecules including pteridines isoalloxazines flavins and flavin nucleotides Comprised of nine chapters this book begins with an overview of electrochemical techniques and their use to study biological materials followed by a discussion on the theory and instrumentation of electrochemistry with emphasis on their significance and utility as well as their principles and circuits Subsequent chapters explore nitrogen heterocyclic molecules such as purines and pyrimidines and their nucleosides and nucleotides polynucleotides and nucleic acids pteridines flavins pyrroles porphyrins and pyridines The electrochemistry of biologically important pyridines is considered This monograph should be of value to electrochemists biochemists and biologists

Advances in Polarography Ian S. Longmuir,2013-10-22 Advances in Polarography Volume 2 covers the proceedings of the Second International Congress held at Cambridge in 1959 in honor of the 70th birthday of Professor Heyrovsky This volume is composed of 35 chapters and begins with intensive discussions on the theoretical and fundamental aspects as well as pertinent equations in polarography Considerable chapters are devoted to the chemical and metallurgical applications of the technique with emphasis on the trace determination of certain compounds The remaining chapters explore other application of specific polarographic technique such as nicotinic acid iso benzpyrylium salts and metal complex analysis

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