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FINAL REPORT

1 October 1976 - 30 June 1979

"STUDIES IN ELECTROANALYTICAL CHEMISTRY AND RAMAN SPECTROSCOPY IN INORGANIC SOLVENTS"

Contract N00014-77-C-00$\overset{0}{4}$

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Work on electroanalytical chemistry, generally involving on-line computerized instrumentation, is summarized. Studies in chloroaluminate melts, predominantly involving n-butylpyridinium chloride-aluminum chloride, are summarized. Studies involving Raman spectroscopy, potentiometry, infrared spectroscopy and voltammetry are summarized.

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INTRODUCTION

This is the Final Report on Contract N00014-77-C-004, "Studies in Electroanalytical Chemistry and Raman Spectroscopy of Inorganic Solvents." Work on this Contractual phase was initiated October 1, 1976 at a level of $46,000, October 1, 1977 at a level of $20,000 (see below) and October 1, 1978 at a level of $50,000. This latter figure will be decreased to $30,000 as a result of a reduction and early termination (June 30,1979) of this present contract as a results of the Principal Investigator leaving Colorado State U. sometime during June, 1979. During the period July 1, 1977 - June 30, 1978, the Principal Investigator was in Washington, D.C., serving as a program manager at the Air Force Office of Scientific Research. Because of that, and residual funding from FY-76, the FY-1977 funding was at the decreased level indicated.

Work carried out under this Contract encompassed two major areas; the first involved studies in electroanalytical chemistry, generally involving on-line computerized instrumentation. The second area involved studies in molten salt chemistry, intended to reflect the introduction of spectroscopic methodology into our studies in this area.

ELECTROANALYTICAL RESEARCH

The electroanalytical work was an outgrowth of work carried on during the period 1974-1976 on a prior O.N.R. Contract. This work will be discussed first, mainly by reference to Technical Reports published under this contract, and the resultant journal publications.

Technical Report No. 1 -"DIGITAL SIMULATION OF DIFFERENTIAL PULSE POLAROGRAPHY WITH INCREMENTAL TIME CHANGE" by J. W. Dillard, John A. Turner and R. A. Osteryoung, November, 1976, was published in Analytical Chemistry 49, 1246 (1977). Explicit finite difference simulations of TAST, normal pulse and differential pulse polarography for the reversible case were developed for conserving computer time. The method involved changing the time increment during the simulation. Results were shown to conform to accepted theory and included sphericity and the DME compression of the diffusion layer due to the drop expansion. The dc faradaic distortion of the differential pulse mode was also studied. The simulation gave a factor of five savings in computer time over simulators of the standard type.

Technical Report No. 2 - "SQUARE WAVE VOLTAMMETRY AT THE DROPPING MERCURY ELECTRODE: THEORY" by J. H. Christie, J. A. Turner and R. A. Osteryoung, February 1977, was published in Analytical Chemistry 49, 1899 (1977). The theoretical aspects of square wave voltammetry at the dropping mercury electrode were presented. The technique involved scanning the entire potential range of interest on a single drop of a DME. Asymmetries in the waveform as well as variations in current measurement parameters were discussed. Indications that previous uses of the waveform may not have utilized all its capabilities were presented.

Technical Report No. 3 - "SQUARE WAVE VOLTAMMETRY AT THE DROPPING MERCURY ELECTRODE: EXPERIMENTAL" by John A. Turner, J. H. Christie, M. Vukovic and R. A. Osteryoung, April 1977, was published in Analytical Chemistry 49, 1904 (1977). Experimental verification of the theoretical work presented in Technical Report No. 2 for square wave voltammetry at the dropping mercury electrode was given. Experiments used ferric oxalate and cadmium (II) in HCl and confirmed excellent agreement with theory. Experimental peak heights and peak widths were found to be within 2% of calculated results. An example of trace analysis using square wave voltammetry at the DME was presented. The technique was shown to have the same order of sensitivity as differential pulse polarography but was much faster to perform. A detection limit for cadmium in 0.1M HCl for the system used was 7×10^{-8} M.

Technical Report No. 4 - See below.

Technical Report No. 5 - "RAPID SCAN ALTERNATE DROP PULSE POLAROGRAPHIC METHODS" by John Turner and R. A. Osteryoung, November 1977, was published in Analytical Chemistry 50, 1496 (1978). A number of alternate drop waveforms which compensate for charging currents at the dropping mercury electrode which arise as a result of drop growth were examined in connection with several rapid scan pulse voltammetric methods. Both normal and differential pulse and square wave rapid scan waveforms were applied and it was demonstrated that the alternate drop techniques could be applied to the rapid scan methodology in an acceptable manner.

Technical Report No. 6 - "ANALYTICAL IMPLICATIONS OF DIFFERENTIAL PULSE POLAROGRAPHY OF IRREVERSIBLE REACTIONS FROM DIGITAL SIMULATIONS: CALCULATIONS AND EXPERIMENTS" by James Dillard, John O'Dea and R. A. Osteryoung, April 1978, was published in Analytical Chemistry 51, 115 (1979). Explicit finite difference simulations of differential pulse polarography were developed, following the procedures developed under Technical Report No. 1, for evaluating the analytical applications of differential pulse polarography to irreversible and quasireversible electron transfer reactions. From simulation data, detailed prediction of polarographic peak shape and position as a function of pulse height, pulse time and drop time was shown to be possible. Optimum conditions for analytical application of differential pulse polarography was discussed.

Work in the electroanalytical area referred to above was performed largely during the first two years of this contract and acknowledges both Office of Naval Research and National Science Foundation support since the work was, in fact, largely initiated on and an outgrowth of, the prior O.N.R. contract which was carried to completion under the present contract. In fact, the theoretical work was largely performed under O.N.R. support, with a smaller fraction of the experimental work being O.N.R. supported.

SPECTROSCOPIC AND MOLTEN SALT RESEARCH

The molten salt work was to have involved Raman spectroscopic studies of solutes in inorganic liquids which were electrochemically of interest. Our work has focused on chloroaluminate melts, both $NaCl-AlCl_3$ and the recently discovered n-butylpyridinium chloride-aluminum chloride system, which is a "room temperature" molten salt. Experimental problems beyond our control, and those of Professor Elliot R. Bernstein, in whose laboratory the Raman work was done, required other studies on the room temperature melt, as well as spectroscopic work.

Technical Report No. 4 - "ELECTROCHEMISTRY OF Ni(II) AND THE BEHAVIOR OF OXIDE IONS IN CHLOROALUMINATE MELTS" by Bernard Gilbert and R. A. Osteryoung, August 1977, was published in the Journal of the American Chemical Society 100, 2725 (1978). The electrochemical behavior of Ni(II) was investigated in chloroaluminate melts between 175 and 210°C. Ni(II) was found to be completely soluble in the acidic mixtures where the pCl was higher than 5.5 (mol kg^{-1} scale). A well defined and behaved reduction wave was observed at 1.2 V vs. an Al reference in a NaCl-saturated reference compartment. When the pCl was decreased, precipitation of $NiCl_2$ was found to occur. This was confirmed by electrochemical and X-ray diffraction experiments. A solubility product of $10^{-12.9}$ (mol kg^{-1}) was found. The result was in disagreement with the precipitation of NiO which had previously been reported in the literature. As a direct consequence, the acid-base behavior of oxide ions in chloroaluminates was reinvestigated. Precise titration of the basic strength of oxide ions was performed in narrow pCl ranges. An equilibrium constant of 1.6×10^{-2} mol kg^{-1} was found for the general reaction

$AlOCl_2^- \rightleftarrows AlOCl + Cl^-$, two orders of magnitude higher than the previously reported value.

Technical Report No. 7 - "RAMAN SPECTRA OF MOLTEN ALUMINUM CHLORIDE-1-BUTYLPYRIDINIUM CHLORIDE SYSTEM AT AMBIENT TEMPERATURES" by Robert Gale and R. A. Osteryoung, May 1978, was published in Inorganic Chemistry 17, 2727 (1978). The Raman spectra of $AlCl_3$-1-butylpyridinium chloride liquids at ambient temperatures were recorded for the 0.75:1.0 to 2.0:1.0 molar composition range, respectively. Four absorption bands each for $AlCl_4^-$ and $Al_2Cl_7^-$ ionic species were assigned on the basis of higher temperature vibrational spectral results of $AlCl_3$-alkali metal chloride systems. The association equilibrium constant for $Al_2Cl_7^-$ ion formation, $2\ AlCl_4^- + Al_2Cl_6 \rightleftarrows 2\ Al_2Cl_7^-$, is significantly larger than that for the $AlCl_3$-MCl melts, where M represents an alkali metal cation.

Technical Report No. 8 - "POTENTIOMETRIC INVESTIGATION OF DIALUMINUM HEPTACHLORIDE FORMATION IN ALUMINUM CHLORIDE: 1-BUTYLPYRIDINIUM CHLORIDE MIXTURES" by Robert Gale and R. A. Osteryoung, November 1978, is currently in press in Inorganic Chemistry. The solvent acid-base properties of $AlCl_3$:1-butylpyridinium chloride melts from 2.2:1.0 to 0.6:1.0 molar ratios, respectively, were investigated by potentiometry. The equilibrium constant for the dissociation reaction $2\ AlCl_4^- \rightleftarrows Al_2Cl_7^- + Cl^-$ was determined to be less than 3.83×10^{-13} at 30°C. The 1-butylpyridinium cation was spontaneously reduced by elemental aluminum. The equilibrium for the dissociation reaction fit the entire potentiometric titration curve throughout the region studied; this is in distinction to the $AlCl_3$-NaCl system, where equilibria such as $2\ AlCl_4^- + 2\ Al_2Cl_6 \rightleftarrows 2\ Al_2Cl_7^-$ must be invoked in the acid range. In short, the Raman work, described in Technical Report No. 7, appears confirmed by the results of this study.

It is also verified by the experimental observation that the vapor pressure of $AlCl_3$ above the 2:1 $AlCl_3$:1-butylpyridinium chloride melt at 175°C, is insignificant compared to that over the 2:1 $AlCl_3$:NaCl melt.

A publication acknowledging O.N.R. support appeared as a chapter in a book, but was not put out as a Technical Report. This is "ACID-BASE DEPENDENT REDOX CHEMISTRY IN MOLTEN CHLOROALUMINATES" by Gleb Mamantov and R. A. Osteryoung, which appeared in "Characterization of Solutes in Non-Aqueous Solvents," edited by G. Mamantov, pg. 223-249, Plenum Publishing Co., New York (1978). This was the result of a presentation on "Acid-Base Dependent Electrochemistry in Molten Sodium Tetrachloroaluminate Solvents," presented at the Symposium on Spectroscopic and Electrochemical Characterization of Solute Species in Non-Aqueous Solvents of the American Chemical Society meeting in San Francisco in August, 1976.

Another publication acknowledging O.N.R. support is in press and will not be put out as a Technical report. This is "ELECTROCHEMICAL STUDIES IN A ROOM TEMPERATURE MOLTEN SALT" by R. A. Osteryoung, R. J. Gale, J. Robinson, R. Bugle and B. Gilbert which will appear in the Proceedings of the Second International Symposium on Molten Salts, to be published by the Electrochemical Society and edited by J. Braunstein. This was the result of a presentation at the Second International Symposium on Molten Salts at the Electrochemical Society Meeting in Pittsburgh in October, 1978.

The two publications above either review previously published work or are brief summaries of work in press.

A rather lengthy review of recent work in the room temperature melts, "CHEMICAL AND ELECTROCHEMICAL STUDIES IN ROOM TEMPERATURE ALUMINUM HALIDE-CONTAINING MELTS" by Helena Li Chum and R. A. Osteryoung, has been prepared for the book "Ionic Liquids," to be edited by D. Inman and D. Lovering and to be published by Plenum Publishing Co. This is an outgrowth of a presentation on "Acid-Base Dependent Chemistry and Electrochemistry in Chloroaluminate Melts," at the Conference on Highly Concentrated Aqueous Solutions and Molten Salts at Oxford University in July of 1978. This review will be sent out as a Technical Report in the near future.

Two manuscripts are in draft form and will ultimately appear as Technical Reports before June 30, 1979. The first of these, entitled "ELECTROCHEMICAL REDUCTION OF PYRIDINIUM IONS IN IONIC ALUMINUM CHLORIDE:1-ALKYLPYRIDINIUM CHLORIDE AMBIENT TEMPERATURE LIQUIDS" by R. J. Gale and R. A. Osteryoung, describes the chemistry, noted in Technical Report No. 8 above, of the reduction of the 1-butylpyridinium cation in the room temperature melt. The reduction was investigated in the 0.8:1 mole ratio melt by cyclic voltammetry and electrochemical pulse methods. A dimeric product of primary pyridyl radical coupling, 4,4'-tetrahydrobipyridine, forms in about 45% yield and undergoes fairly rapid chemical dissociation to the stable, electroactive 1,1'-dibutyl-4,4'-bipyridinium monocation radical. The corresponding dialkyl viologen dication reduces in two steps at half-wave potentials of -0.56 V and -0.96 V vs. Al(0) reference electrode in the 2:1 solvent, each with one-electron reversible behavior. It is anticipated that this manuscript will be submitted to the Journal of the American Chemical Society or possibly to the Journal of Electroanalytical Chemistry.

A second manuscript, "ELECTROCHEMICAL AND SPECTRAL INVESTIGATIONS OF Ni(II) COMPLEX IONS IN ROOM TEMPERATURE CHLOROALUMINATE MELTS" by R. J. Gale, B. Gilbert, and R. A. Osteryoung, has just been prepared. Absorption spectra of $NiCl_2$ dissolved in 0.8:1 and 1.5:1 $AlCl_3$:1-butylpyridinium chloride solvents at room temperature have been recorded. In basic, chloride rich, melts, the Ni(II) is present as $NiCl_4^{-2}$ with molar extinction coefficients A_{658}- 169 and A_{705} - 175

mol^{-1} cm^{-1}. (The behavior may be compared to the insolubility of Ni(II) in the basic sodium tetrachloroaluminate melt discussed in Technical Report No. 4.) The reduction of Ni(II) ion species exhibited irreversible behavior in acidic melts, but no $NiCl_4^{-2}$ reduction could be detected in the basic melt at carbon electrodes. Potentiometric experiments gave a theoretical Nernst slope for Ni(II) additions in the acidic region and a fourth power Cl^- ion dependence consistent with $NiCl_4^{-2}$ formation in the melt. At 40°C, the Ni/Ni(II) standard potential on the mole fraction sacle is +0.800 ± 0.005 V vs. the 2:1 Al reference and an equilibrium constant for the dissociation reaction, $NiCl_4^{-2} \rightleftarrows Ni^{+2} + 4\ Cl^-$ was determined to be 1.2×10^{-46}.

An infrared study of the room temperature chloroaluminate melt is in progress, and expected to be completed shortly. Results complementary to the Raman study (Technical Report No. 7) should result. This may be the first infrared absorption study of molten salts, possible, obviously, as a result of the low temperature nature of these melts. This study, by the way, has opened a new and fruitful area, since although the butylpyridinium infrared spectra is rather overwhelming, sufficient window exists to permit observation of infrared absorption bands of various solutes in the melt. This will be coupled with some of our organic electrochemistry work supported by A.F.O.S.R. It is anticipated that a Technical Report, by R. J. Gale and R. A. Osteryoung, will be issued and that the manuscript on this work will be submitted to Inorganic Chemistry.

We have recently initiated some work aimed at determining if chloroaluminates, either the high temperature sodium system or the room temperature melts, have potential as solvents for solar energy conversion via the semiconductor-photoelectrochemistry route. This work, discussed on the telephone with Dr. David Nelson, is being carried out in collaboration with Dr. Arthur Nozik of the Solar Energy Research Institute and arose as a result of the Principal Investigator's talk on the room temperature melts at the Gordon Research Conference in Santa Barbara in January, which elicited considerable interest, and subsequent conversation with Dr. Nozik, who has considerable expertise in this area. This represents a deviation from our Contract proposal, and a letter outlining this was sent to O.N.R. At this writing, preliminary results of capacity measurements have been obtained on a reduced TiO_2 crystal in H_2SO_4, simply to determine if our bridge is working properly and if the crystal is properly mounted, etc. After we had started this, a paper, "Semiconductor Tin Oxide Electrodes in Molten Sodium Chloroaluminate at 175°C," by I. Uchida, H. Urushibata and S. Toshima appeared in the Journal of Electroanalytical Chemistry 96, 45 (1979) which indicates the validity of this idea and strongly suggests that the entire spectrum of chemistry in both the high and low temperature chloroaluminate systems which we have investigated over the years can be applied to these semiconductor electrochemical studies.

Thus, in summary, during the period October 1, 1976 to the present time, eight Technical Reports have been issued, and we anticipate issuing at least four more in the near future. Eight publications have appeared acknowledging O.N.R. support, three more are in press, and at least three more will be submitted.

PERSONNEL

Individuals who were supported on this present contract for significant periods of time include:

Dr. Bernard Gilbert
Dr. James Dillard
Dr. Robert Gale
Mr. John Turner (received Ph.D.)

Individuals who were supported on this activity for three months or less include:

Mr. Mark Schure
Mr. John O'Dea
Dr. James Robinson

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