





MICROCOPY RESOLUTION TEST CHART  
NATIONAL BUREAU OF STANDARDS 1963-A

AD-A182 723 DOCUMENTATION PAGE

1a REPOF Unclassified		1b RESTRICTIVE MARKINGS	
2a SECURITY CLASSIFICATION AUTHORITY SELECTED		3. DISTRIBUTION / AVAILABILITY OF REPORT Approved for public release; Distribution unlimited	
2b. DECLASSIFICATION / DOWNGRADING SOURCE JUL 20 1987		5. MONITORING ORGANIZATION REPORT NUMBER(S) AFOSR-TR- 87-0879	
4. PERFORMING ORGANIZATION REPORT NUMBER(S) CSD		7a. NAME OF MONITORING ORGANIZATION AFOSR/NC	
6a. NAME OF PERFORMING ORGANIZATION State University of New York at Buffalo	6b OFFICE SYMBOL (if applicable)	7b ADDRESS (City, State, and ZIP Code) Bldg. 410 Bolling AFB, D.C. 20332-6448	
6c ADDRESS (City, State, and ZIP Code) Department of Chemistry State University of New York at Buffalo Buffalo, New York 14214		9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER AFOSR-84-0292	
8a. NAME OF FUNDING / SPONSORING ORGANIZATION AFOSR	8b OFFICE SYMBOL (if applicable) NC	10. SOURCE OF FUNDING NUMBERS	
8c. ADDRESS (City, State, and ZIP Code) Bldg. 410 Bolling AFB, D.C. 20332-6448		PROGRAM ELEMENT NO. 61102F	PROJECT NO. 2303
11 TITLE (include Security Classification) Studies in Ambient Temperature Ionic Liquids.		TASK NO.	WORK UNIT ACCESSION NO.
12 PERSONAL AUTHOR(S) Robert A. Osteryoung and Janet G. Osteryoung			
13a TYPE OF REPORT Final	13b. TIME COVERED FROM 1 Aug 84 TO 30 Nov 86	14. DATE OF REPORT (Year, Month, Day) 1987 June 30	15. PAGE COUNT 28
16 SUPPLEMENTARY NOTATION			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	Ionic liquids, ambient temperature molten salts, electrochemistry, chloroaluminates	
19 ABSTRACT (Continue on reverse if necessary and identify by block number) Chemical and electrochemical studies were carried out in ambient temperature molten salts composed of aluminium chloride mixed with either N-1(butyl)pyridinium chloride or 1-methyl-3-ethylimidazolium chloride. Electrochemical, spectroscopic and NMR work was carried out on electroactive polymers prepared in situ in the molten salts, or on redox polymers prepared ex situ, but investigated in the molten salts. "Neutral" melts were employed for studies of complex ion stoichiometry and chloride oxidation. A number of species, metal carbonyls, ferrocene, iodine-iodide, were investigated electrochemically as a function of melt acidity. NMR proton relaxation studies on the "tail" of the butylpyridinium cation were carried out and correlations made to melt composition and viscosity.			
20 DISTRIBUTION / AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a NAME OF RESPONSIBLE INDIVIDUAL Dr. John Wilkes		22b TELEPHONE (include Area Code) (202)767-4967	22c OFFICE SYMBOL NC

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This is a Final Report on AFOSR-84-0292, "Studies in Ambient Temperature Ionic Liquids".

The research carried out under this grant was intended to investigate molten chloroaluminate (ionic liquids), in particular those consisting of organic chlorides (RCI) and aluminum chlorides. The organic chlorides consist of either N-(1-butyl)pyridinium chloride (BuPyCl) or 1-methyl-3-ethylimidazolium chloride (ImCl), both of which form conducting liquid systems from extremely basic conditions (excess RCI) to very acid systems containing excess AlCl<sub>3</sub>, with an AlCl<sub>3</sub>:RCI mole ratio >1:1 to ca 2:1. We intended to study the electrochemistry of solute ions showing acid-base dependent electrochemistry, of polymer coated electrodes with emphasis on so called electroactive polymers, and to carry out a variety of spectroscopic and electrochemical techniques in pursuit of our studies.

What was originally to have been a three-year option grant was funded from 1 August, 1984 through 30 November, 1986. Total funds during this period amounted to \$255,262 (\$125,042 FY 84; \$130,220 FY 85). A decision was made by AFOSR not to renew the option grant beyond its second year, and a four month no-cost extension to the second year of the grant period, from 1 August 1986 through 30 November 1986, was made and approved.

In the interests of brevity, material which has been published or is in press will be covered by inclusion of the abstract rather than extensive discussion.

Appendix A contains a cumulative listing of publications under AFOSR funding on past grants and contracts. A listing of formal presentations at meetings, universities, etc. is also included.

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SUMMARY OF WORK: 1 AUGUST 1984 - 30 NOVEMBER 1986

1. Completed Work

A. Studies of Modified Electrodes

Here we report on a number of studies of polymer coated electrodes in the ambient temperature ionic liquid.

1. Polypyrrole rate studies

A manuscript entitled "Charging and Discharging Rate Studies of Polypyrrole Films in  $\text{AlCl}_3$ :1-Methyl-(3-Ethyl)-Imidazolium Chloride Molten Salts and in  $\text{CH}_3\text{CN}$ " was published in the Journal of Electroanalytical and Interfacial Electrochemistry. See Appendix A, Ref. 66.

ABSTRACT

The chronocoulometric charging and discharging of polypyrrole films in basic  $\text{AlCl}_3$ /1-methyl-(3-ethyl)-imidazolium chloride molten salts and  $\text{CH}_3\text{CN}$  have been investigated. Both process follow a  $t_{1/2}$  time dependence and are significantly faster in the molten salts. Comparison with redox polymer and porous electrode models shows that neither model is satisfactorily applicable over the entire potential region studied. The charging and discharging rates are limited by ion migration in the polymer and for potential steps in the "double layer charging" region polypyrrole behaves as a porous electrode material. In this region there is good correlation between the charging/discharging rates and the solvent conductivity.

2. Poly-ruthenium bipy/vpy films.

A manuscript entitled "Charge Transport in Poly-[ $\text{Ru}(2,2'$ -Bipyridine) $_2(4$ -Vinylpyridine) $_2$ ] $^{3+/2+}$  Films in  $\text{AlCl}_3$ /N-(1-Butyl)pyridinium Chloride and  $\text{AlCl}_3$ /1-Methyl(3-Ethyl)imidazolium Chloride Molten Salts" was published in the Journal of Electroanalytical and Interfacial Chemistry. See Appendix A, Ref. 67.

ABSTRACT

Charge transport in Poly-[ $\text{Ru}(2,2'$ -Bipyridine) $_2(4$ -Vinylpyridine) $_2$ ] $^{3+/2+}$  films has been measured by chronoamperometry, chronocoulometry, linear sweep voltammetry and mediated oxidation of [ $\text{Fe}(2,2'$ -bipyridine) $_3$ ] $^{2+}$ . Charge transport follows diffusional behavior in both acidic and neutral  $\text{AlCl}_3$ /N-(1butyl)pyridinium chloride molten salts and in  $\text{CH}_3\text{CN}$ . The charge transport diffusion coefficient did not depend upon which melt was used and an average value of  $1.7 \times 10^{-6} \text{ mol cm}^{-2} \text{ s}^{-1/2}$  was obtained. This is 2.6 times smaller than the value obtained in  $\text{CH}_3\text{CN}$ . Possible reasons for this are discussed.

### 3. A new polymer film.

A manuscript entitled "Electrochemistry and Spectroelectrochemistry in  $\text{CH}_3\text{CN}$  and Aluminum Chloride/*N*-(1-Butyl)pyridinium Chloride Molten Salts of Films Prepared by Electrochemical Polymerization of Tris(5-amino-1,10-phenanthroline)iron (II)" was published as a Note in Inorganic Chemistry. See Appendix A, Ref. 66. Since no Abstract is published, a Summary follows.

#### SUMMARY

$[\text{Fe}(5\text{-NH}_2\text{-phen})]^{2+}$  was electropolymerized in  $\text{CH}_3\text{CN}$ . Electrochemistry on the polymer was carried out in both  $\text{CH}_3\text{CN}$  and in the ambient temperature molten salts. Spectroelectrochemistry was employed to measure the  $E_{1/2}$  value of the polymer redox couple in  $\text{CH}_3\text{CN}$ . The electrochemical behavior of the polymer in the acidic molten salt showed complex behavior indicative of specific interaction, perhaps adduct formation, with  $\text{AlCl}_3$ .

### 4. Polythiophene

A manuscript entitled "Electrochemistry of Polythiophene and Polybithiophene Films in Ambient Temperature Molten Salts" has been accepted for publication in the Journal of the Electrochemical Society. See Appendix A., Ref. 69.

#### ABSTRACT

Polythiophene and polybithiophene polymer films were deposited on platinum, tungsten and glassy carbon electrodes by anodic oxidation of the monomer in ambient temperature molten salts consisting of a mixture of aluminum chloride and 1-methyl-3-ethylimidazolium chloride. The formation reaction of polythiophene and polybithiophene is totally irreversible. The polymer films are conductive in the oxidized state and non-conductive when reduced as indicated by the shape of cyclic voltammetric curves for the polymers and for ferrocene oxidation on electrodes covered by different thicknesses of polymer. Results of ferrocene oxidation on electrodes coated by polythiophene suggest that these polymer films are porous. The films obtained in these molten salts are more stable and their electrochemical behavior appears to be less complicated than that of films prepared in acetonitrile solutions.

## B. Studies in Neutral Melts

The unique features of the neutral melts were utilized in a variety of different studies.

### 1. Butyrypyridinium reduction.

A manuscript entitled "Electrochemical Reduction of N-(1-Butyl)Pyridinium Cation in 1-Methyl-3-Ethyl-Imidazolium Chloride-Aluminum Chloride Ambient Temperature Ionic Liquids" has been published in *Electrochimica Acta*. See Appendix A, Ref. 70.

#### ABSTRACT

The reduction of N-(1-Butyl)pyridinium cation ( $\text{BuPy}^+$ ) has been studied in the ambient temperature ionic liquid, 1-methyl-3-ethyl-imidazolium chloride ( $\text{ImCl}$ )-aluminum chloride. A one-electron wave with convective-diffusion controlled limiting current was observed and the diffusion coefficients of  $\text{BuPy}^+$  were measured in neutral and basic ( $\text{MC} = 0.6$ )  $\text{ImCl-AlCl}_3$  melts. The  $D/T$  was found to be almost independent of melt composition.  $E_{1/2}$  and peak potentials measured in neutral and basic melts were practically independent of melt composition; however a small but significant shift to less negative potentials was observed in the absence of chloride ions (i.e. in pure  $\text{AlCl}_3$ ). This effect was attributed to ion pair formation ( $\text{BuPy}^+\text{-Cl}^-$ ). Coulometry indicated an "n" value of 1 for the reduction of the  $\text{BuPy}^+$  cation. About 15% of the primary product was transformed into the electroactive viologens.

### 2. Determination of complex ion stoichiometry

A manuscript entitled "Electrochemistry in Neutral Ambient Temperature Ionic Liquids. Part I. Studies of Iron(III), Neodymium(III) and Lithium(I)" has been published in *Inorganic Chemistry*. See Appendix A, Ref. 71.

#### ABSTRACT

An ambient-temperature "neutral" ionic liquid composed of aluminum chloride and either N-(1-butyl)pyridinium chloride or 1-methyl-3-ethyl-imidazolium chloride,  $\text{BuPyCl}$  or  $\text{ImCl}$ , respectively, has been employed in studies that take advantage of their unusual properties. These include an extended electrochemical window, readily controlled additions of excess chloride (basic) or aluminum chloride (acid), and the fact that the physical properties of the "neutral" melt do not change about the 1:1 mole ratio of  $\text{AlCl}_3$  to  $\text{RCl}$ .  $\text{Li}^+$  was found to be reducible in the "neutral"  $\text{AlCl}_3\text{-ImCl}$  melt, and its diffusion coefficient was found to be  $0.6 \times 10^{-7} \text{ cm}^2 \text{ s}^{-1}$ . The stoichiometry of the complex formed between  $\text{Nd(III)}$  and  $\text{Cl}^-$  in the molten salt system was investigated by what is essentially an amperometric titration and was found to be  $\text{NdCl}_6^{3-}$ . The structure of the  $\text{Fe(III)}$  chloro complex that exists in basic or acidic melts just slightly varying in composition from the

neutral melt was also investigated; a constant value for the diffusion coefficient-viscosity product for the complex in both systems suggests no change in structure.

### 3. Sb-chlorocomplex ions

A paper entitled "Studies of Antimony(III) in Ambient Temperature Ionic Liquids" has been published in Inorganic Chemistry. As no Abstract was published, a Summary is provided. See Appendix A, Ref. 72.

#### SUMMARY

An indirect method, which takes advantage of the unbuffered nature of the neutral ambient temperature ionic liquid, is used to determine that the Sb(III) chloro complex existing in the neutral  $AlCl_3$ - $ImCl$  melt is  $SbCl_2^+$ .

### 4. Reaction of chloride with proton

A manuscript entitled "Hydroquinone as a Proton Donor in Ambient Temperature Chloroaluminate Ionic Liquids: Reaction with Chloride Ion" has been published in Inorganic Chemistry. See Appendix A, Ref. 73.

#### ABSTRACT

The reaction of water and hydroquinone ( $H_2Q$ ) as proton donors in either N-1-butylpyridinium chloride ( $BuPyCl$ )- or 1-methyl-3-ethylimidazolium chloride ( $ImCl$ )-aluminum chloride ambient temperature ionic liquids has been investigated. Amperometric titrations of free chloride ion indicate that  $H_2Q$  reacts with  $2 Cl^-$ , suggesting that  $HCl$  is undissociated in the molten salt. Comparison of the  $D/\eta T$  values, where  $D$ ,  $\eta$ , and  $T$  are the diffusion coefficients, viscosity, and temperature, respectively, suggest that both water and  $H_2Q$  give rise to two  $HCl$  molecules. Other observations regarding the interaction of water with the ambient temperature molten salt are reported.

### C. General Electrochemistry

Under this category we report primarily electrochemical studies of solutes in the ambient temperature molten salt systems; a bit of other work is included, however.

#### 1. Ferrocene electrochemistry

A paper, "Electrochemical Studies of Ferrocene and Ferrocenium Ion in Aluminum Chloride-N-1-Butylpyridinium Chloride Ionic Liquid" was published in Inorganic Chemistry. See Appendix A, Ref. 74.

## ABSTRACT

Electrochemical studies of the ferrocene/ferrocenium ion system have been carried out in  $\text{AlCl}_3$ -N-1-butylpyridinium chloride ionic liquid at  $40^\circ\text{C}$  as a function of melt composition. The ferrocene/ferrocenium electron-transfer process is reversible and the formal potential of the ferrocene/ferrocenium cation couple varies only slightly with wide changes of solvent acidity. Ferrocene and ferrocenium ion are both stable in neutral butylpyridinium tetrachloroaluminate. In basic (excess BuPyCl) melts, ferrocene is stable, while the ferrocenium cation is decomposed by  $\text{Cl}^-$  to  $\text{Fe}(\text{Cp})_2$  and  $\text{FeCl}_4^-$ . In acidic (excess  $\text{AlCl}_3$ ) melts ferrocene is oxidized to ferrocenium ion by traces of oxygen and reacts with products of the reaction of water with the solvent. The ferrocenium cation is stable in the acid melt.

## 2. Iodine electrochemical-spectrophotometric studies

Two papers on this subject, one primarily electrochemical, the other spectrophotometric, have been published. The first, "Electrochemical Studies of Iodine in an Aluminum Chloride-Butylpyridinium Chloride Ionic Liquid: Part II. Neutral and Basic Solvent Composition" was published in the Journal of Electroanalytical and Interfacial Chemistry. The second, "Spectrophotometric Studies of Iodine Complexes in an Aluminum Chloride-Butylpyridinium Chloride Ionic Liquid" was published in Inorganic Chemistry. See Appendix A, References 75 and 76.

## ABSTRACT 1

The electrochemical behavior of iodine in an ambient temperature molten salt system, aluminum chloride-N-(1-butyl)pyridinium chloride (BuPyCl), have been studied in basic (excess BuPyCl) and neutral (1.0:1.0  $\text{AlCl}_3$ :BuPyCl mole ratio) melt compositions. Acid-base interactions of iodine in different oxidation states with the ionic solvent are observed. High stability of triiodide ion in neutral butylpyridinium tetrachloroaluminate indicates relatively weak intermolecular interactions in this solvent. In basic solutions polyhalogen equilibria involving iodine in different oxidation states and chloride ions are established. In iodine and tetraethylammonium triiodide solutions a mixture of  $\text{ICl}_2^-$ ,  $\text{I}_2\text{Cl}^-$ ,  $\text{I}_3^-$  and  $\text{I}^-$  forms. The formation constants of  $\text{I}_2\text{Cl}^-$  and  $\text{I}_3^-$  and the equilibrium constant of  $\text{I}_2\text{Cl}^-$  disproportionation are estimated.

## ABSTRACT 2

Iodine, iodine chloride, tetraethylammonium iodide, and tetraethylammonium triiodide solutions in aluminum chloride-N-1-butylpyridinium chloride ambient temperature ionic liquids have been studied by UV-visible spectrophotometry. The different oxidation states of iodine show a distinct dependence on the solvent acidity. Iodine(+) exists in the form of  $\text{ICl}$  in acidic melts and in the form of  $\text{ICl}_2^-$  in basic  $\text{AlCl}_3$ -BuPyCl mixtures.

Molecular iodine does not form strong complexes in acidic or neutral solutions. Triiodide ion is stable in neutral butylpyridinium tetrachloroaluminate, but not in acidic melts. In solutions of iodine or triiodide in basic  $\text{AlCl}_3$ -BuPyCl ionic liquids equilibrium mixtures of  $\text{I}_2\text{Cl}^-$ ,  $\text{I}_3^-$ ,  $\text{ICl}_2^-$  and  $\text{I}^-$  are formed. The charge transfer bands observed for iodide solutions in neutral and basic melts reveal the formation of iodide ion-butylpyridinium cation ion pairs. Similar bands in pure basic  $\text{AlCl}_3$ -BuPyCl mixtures indicate the association of chloride ions and butylpyridinium cation.

### 3. Tetrachloroaluminate dissociation constant

A paper entitled "Potentiometric Studies of the Chlorine Electrode in Ambient Temperature Chloroaluminate Ionic Liquids: Determination of Equilibrium Constants for Tetrachloroaluminate Ion Dissociation" was published in *Inorganic Chemistry*. See Appendix A, Reference 77.

#### ABSTRACT

The potentiometric behavior of the chlorine electrode has been investigated at 40° C in aluminum chloride-N--butylpyridinium chloride and in aluminum chloride-1-methyl-3-ethylimidazolium chloride ionic liquids. In basic (excess organic chloride) melts, the Nernstian behavior of a tungsten electrode in solutions containing  $\text{Cl}_2(\text{g})$  indicates two electrode reaction stoichiometries:  $\text{Cl}_3^- + 2\text{e}^- = 3\text{Cl}^-$ , when chloride ion is in excess over chlorine, and  $3\text{Cl}_2 + 2\text{e}^-$ , when there is an excess of chlorine. Formal potentials of the  $\text{Cl}_2/\text{Cl}_3^-$ ,  $\text{Cl}_3^-/\text{Cl}^-$ , and  $\text{Cl}_2/\text{Cl}^-$  couples have been determined in both ionic solvents, and the stability constant for the  $\text{Cl}_3^-$  ion has been obtained. The difference between the  $E^0(\text{Cl}_3^-)$  values in these two solvents reflects the difference between ionic association constants for the two organic chlorides. In acidic (excess  $\text{AlCl}_3$ ) melt compositions, slow equilibria and reactions of chlorine with the organic cations impede potentiometric measurements with the chlorine electrode. The reversible chlorine electrode potential could be measured only in acidic  $\text{AlCl}_3$ -BuPyCl melts, and the tetrachloroaluminate ion dissociation constant  $\log K_{\text{BuPyCl}} = -17.0 \pm 0.5$  has been obtained. In the acidic 1:1:1  $\text{AlCl}_3$ -ImCl system, the half-life of chlorine is ca. 1 min; thus, the chlorine electrode cannot be used in this system. The tetrachloroaluminate ion dissociation constant in the latter system ( $\log K_{\text{ImCl}} = -17.1 \pm 0.5$ ) was calculated from the difference of the aluminum electrode potentials in acidic  $\text{AlCl}_3$ -BuPyCl and  $\text{AlCl}_3$ -ImCl ionic liquids.

### 4. Carbonyl electrochemistry

A paper on the electrochemistry of some carbonyls, "Electrochemical Oxidation of Some Metal Carbonyls in Ambient Temperature Ionic Liquids" was published in *Electrochimica Acta*. See Appendix A, Reference 78.

## ABSTRACT

The electrochemical oxidation of  $\text{Cr}(\text{CO})_6$ ,  $\text{W}(\text{CO})_6$  and  $\text{Fe}(\text{CO})_5$  was studied in  $\text{AlCl}_3$ :N-(1-butyl)pyridinium chloride (BuPyCl) molten salt at  $40^\circ\text{C}$ . In the 1:1 to 2:1 mole ratio of  $\text{AlCl}_3$ /BuPyCl,  $\text{Cr}(\text{CO})_6$  was found to be reversibly oxidized forming stable  $\text{Cr}(\text{CO})_6^+$ . Further oxidation of  $\text{Cr}(\text{CO})_6^+$  generated  $\text{Cr}(\text{CO})_6^{3+}$  which was not stable. Iron pentacarbonyl and tungsten pentacarbonyl in 1:1 to 2:1 melts each showed single oxidation with some stability of their cations.  $\text{Fe}(\text{CO})_5^+$  was more stable than  $\text{W}(\text{CO})_6^{2+}$ . In the melt of <1:1 mole ratio no evidence was found for the formation of stable  $\text{Fe}(\text{CO})_5^+$ .

### 5. Ionic association in ambient temperature melts

A manuscript entitled "On Ionic Association in Ambient Temperature Chloroaluminate Molten Salts: Analysis of Electrochemical and Conductance Data" was published in the Journal of the Electrochemical Society. See Appendix A, Reference 79.

## ABSTRACT

A simple model based on the validity of the Nernst-Einstein equation and a constant mobility-viscosity product was applied to the interpretation of conductance data for  $\text{AlCl}_3$ -RCl (R = N-(1-butyl)pyridinium or 1-methyl-3-ethylimidazolium) ambient temperature ionic liquids. The dissociation constants of  $\text{RAlCl}_4$  and  $\text{RAl}_2\text{Cl}_7$  were calculated, and the experimental values of specific conductivity were reproduced with an error not exceeding 5% from ionic mobilities calculated from electrochemically measured diffusion coefficients. This calculation suggests that about 50% of the ions are associated into ion pairs. For very basic ( $\text{MC} = 0.6$ ) melts, the formation of ion aggregates is postulated.

### D. Techniques

Here we discuss work primarily dealing with NMR studies of solutes or the molten salt itself.

#### 1. Relaxation rate studies

A manuscript entitled "Relaxation Time Measurements in N-(1-Butyl)pyridinium-Aluminum Chloride Ambient Temperature Ionic Liquids" has been accepted for publication in J. Phys. Chem. See Appendix A, Reference 80.

## ABSTRACT

Proton spin-lattice relaxation times ( $T_1$ ) have been determined for the protons attached to the butyl moiety on the N-(1-butyl)pyridinium cation present in the room temperature molten salt system composed of mixtures of aluminium chloride with N-(1-butyl)pyridinium chloride at various mole ratios.

Reorientational correlation times have been estimated from the data. From the viscosity dependence of these correlation times for protons in various positions on the butyl chain, it is shown that strong cation-anion interactions occur only when chloride anion is present in the melt. Also, the field dependence of the relaxation times has been analyzed to yield correlation times for the random motion giving rise to a relaxation mechanism. The behavior of these experimentally derived correlation times with melt composition and viscosity suggests the following: in acidic melts both overall rotation and internal motion contribute to the relaxation of the alkyl chain protons, while in basic melts, the motion in the terminal methyl group protons is effectively uncoupled from the overall rotation of the BuPy<sup>+</sup> cation.

## II. Work in Progress

Aspects of work in progress at the conclusion of this grant period will be briefly touched upon; it is anticipated that most of these activities will be continued and/or completed under the new AFOSR grant.

### A. Studies of Modified Electrodes

Work on the polymerization mechanisms of pyrrole and fluorene is in progress and will be continued and brought to a conclusion. Simultaneous EPR-electrochemical studies, primarily on polypyrrole, are in progress and will be continued.

### B. Studies in Neutral Melts

Efforts to determine something about the depolarization of the Al electrode by chloride are in progress and should be completed during the initial year of the new grant. Work on this particular topic is also supported, in part, by the Office of Naval Research.

### C. General Electrochemistry

Studies on the quinone-hydroquinone system are in progress, utilizing both electrochemical and spectroscopic techniques. Very fast voltammetry at very small electrodes is also being utilized. This work should be finished and submitted for publication by May or June.

### D. Techniques

A great deal of NMR work is in progress. Studies involving <sup>17</sup>O in water to the melt are being carried out to aid in our understanding of the role of impurities in the solvent molten salt. Initial results were very encouraging, and a manuscript dealing with the work is in preparation. The number of oxide containing species in the acidic melt has been determined to be three, and at least two oxide species are present in the basic melts.

Gutmann donor-acceptor number determinations have been carried out utilizing NMR and electrochemical techniques. A manuscript on this should be written.

NMR studies on the supposed adduct of pyrrole with  $AlCl_3$  have been carried out to explain the inability to polymerize pyrrole in the acidic melt. A manuscript on this will be written.

### III. Reviews and Symposium Proceedings

A major review, "Organic Chloroaluminate Ambient Temperature Molten Salts" was prepared for an August, 1986, NATO workshop on Molten Salts in Camerino, Italy, and will be published. See Appendix A, Reference 81.

### IV. Interaction with Air Force Laboratories

Interactions have continued on a fairly regular basis between Seiler Laboratory personnel and workers here. Dr. John Wilkes has visited and discussions at various meetings have been held. A contractors meeting is planned for April, 1987, at Seiler Laboratory.

### V. Personnel

Personnel associated with this grant for any extended period of time during the grant period are listed below.

#### Senior Research Personnel

Dr. Laura Janiszewska  
Dr. Marek Lipsztajn  
Dr. John O'Dea  
Dr. Jean-Francois Oudard  
Dr. Francisco Uribe

#### Junior Research Personnel

Mr. Thomas Zawodzinski

APPENDIX A

Publications--Grant Related Activity - since AFOSR support initiated.

AFOSR-71-1955; 1 Jan. 1971 - 28 Feb. 1975

1. Janet Osteryoung and R. A. Osteryoung, "The Advantage of Charge Measurements for Determining Kinetic Parameters", *Electrochimica Acta*, 16, 525 (1971).
2. R. A. Osteryoung, "Introduction to the On-Line Use of Computers in Electrochemistry", Vol. II, "Application of Computers to Chemical Instrumentation", Ed. by Mattson, Mark and MacDonald, Marcel Dekker (1973).
3. L. G. Boxall, H. L. Jones and R. A. Osteryoung, "Solvent Equilibria in  $AlCl_3$ - $NaCl$  Melts", *J. Electrochem. Soc.*, 120(2), 223 (1973).
4. H. Lloyd Jones, L. G. Boxall and R. A. Osteryoung, "Organic Electrode Reactions in Fused  $AlCl_3$  Containing Solvents", *J. Electroanal. Chem.*, 38, 476 (1972).
5. L. G. Boxall, H. L. Jones and R. A. Osteryoung, "Electrochemical Studies on Ag, Fe and Cu Species in  $AlCl_3$ - $NaCl$  Melts", *J. Electroanal. Chem.*, 121, 212 (1974).
6. H. Lloyd Jones and R. A. Osteryoung, "Electrode Reactions of Aromatic Amines in Solvents Containing Fused  $AlCl_3$ , II.", *J. Electroanal. Chem.*, 49, 281 (1974).
7. R. J. Gale and R. A. Osteryoung, "Investigation of Subvalent Ion Effects During Aluminum Anodization in Molten  $NaCl$ - $AlCl_3$  Solvents", *J. Electrochem. Soc.*, 121, 983 (1974).
8. V. R. Koch, L. L. Miller and R. A. Osteryoung, "Reductive Defunctionalization of 1-substituted Adamantanes in Molten Sodium Tetrachloroaluminate", *J. Org. Chem.*, 39, 2416 (1974).
9. H. Lloyd Jones and R. A. Osteryoung, "Organic Reactions in Molten Tetrachloroaluminate Solvents", *Advances in Molten Salt Chemistry*, Vol. 3, Edited by J. Braunstein, G. P. Smith and G. Mamantov, Plenum Publishing (1975).
10. R. J. Gale and R. A. Osteryoung, "Dissociative Chlorination of Nitrogen Oxides and Oxyanions in Molten Sodium Chloride-Aluminum Chloride Solvent", *Inorg. Chem.*, 14, 1232 (1975).
11. H. L. Chum, V. R. Koch, L. L. Miller and R. A. Osteryoung, "An Electrochemical Scrutiny of Organometallic Iron Complexes and Hexamethylbenzene in a Room Temperature Molten Salt", *J. Am. Chem. Soc.*, 97, 3264 (1975).
12. D.E. Bartak and R. A. Osteryoung, "The Electrochemical Oxidation of  $N,N,N',N'$ -Tetramethylbenzidine in Molten Sodium Tetrachloroaluminate", *J. Electrochem. Soc.*, 122, 600 (1975).

13. J. Phillips, R. J. Gale, R. G. Wier and R. A. Osteryoung, "Glassy Carbon Rotating Ring-Disc Electrodes for Molten Salt Studies", *Anal. Chem.*, 48, 1266 (1976).
14. D. E. Bartak and R. A. Osteryoung, "The Redox Behavior of Tetrachloro-p-Benzoquinone-Tetrachlorohydroquinone Systems in Molten Aluminum Chloride-Sodium Chloride Solvents", *J. Electroanal. Chem.*, 74, 69 (1976).

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Meeting Presentation and Seminars--Related to Grant Activity

(\*Invited Presentations)

1971

\*R. A. Osteryoung, "Computerized Electrochemical Experimentation", Department of Chemistry Colloquia, Pennsylvania State University, University Park, PA, May 27, 1971.

\*L. G. Boxall, H. L. Jones and R. A. Osteryoung, "Electrochemical Studies in Aluminum Chloride Melts", N. W. Regional ACS Meeting, Bozeman, Montana, June, 1971.

\*R. A. Osteryoung, "Application of a Computer-Based Pulse Polarographic System in Molten Salt Studies", Gordon Research Conference on Molten Salts, Kimball Union Academy, Meriden, N.H., August, 1971.

\*R. A. Osteryoung, "Use of a Mini-Computer in Electrochemical Studies", Symposium on Mini-Computers in the Research & Teaching Laboratory, American Chemical Society Meeting, Washington, D.C., September, 1971.

1972

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Fused Salts", Baylor University (ACS Waco Section) May 8, 1972.

\*R. A. Osteryoung, "Computer Controlled Electrochemical Experimentation", Texas Christian University (Dallas-Fort Worth ACS Section) May 9, 1972.

\*R. A. Osteryoung, "Computer Controlled Electrochemical Experimentation", (San Antonio, Texas ACS Section) May 10, 1972.

\*R. A. Osteryoung, "Computer Controlled Electrochemical Experimentation", Texas A&M (College Station, Texas ACS Section) May 11, 1972.

\*R. A. Osteryoung, "Computer Controlled Electrochemical Experimentation", Midwestern University (Wichita Falls, Texas/Duncan, Oklahoma ACS Section), May 12, 1972.

\*H. L. Jones, L. G. Boxall and R. A. Osteryoung, "Organic Electrochemistry in Aluminum Halide Melts", Rocky Mountain Regional ACS Meeting, Ft. Collins, CO, June 1972.

1973

\*R. A. Osteryoung, H. L. Jones and L. G. Boxall, "Electrochemical Studies in Molten Chloroaluminates", Symposium on Fused Salt Technology, Electrochemical Society Meeting, Chicago, May 8-13, 1973.

\*R. A. Osteryoung, R. H. Abel, L. G. Boxall and B. H. Vassos, "An Introduction to the On-Line Use of Digital Computers in Electrochemistry", Plenary Lecture at Symposium on Electrochemical Measurements by Digital Computer, Electrochemical Society Meeting, Chicago, May 8-13, 1973.

1974

D. E. Bartak and R. A. Osteryoung, "Oxidation of Tetramethylbenzidine in Chloroaluminate Melts", Electrochemical Society, San Francisco, CA, May 1974.

\*R. A. Osteryoung, "Chemistry in Aluminum Chloride Melts", Fifth International Conference in Non-Aqueous Solutions, International Union of Pure and Applied Chemistry, Vienna, July 10-12, 1974.

\*R. A. Osteryoung, "Electrochemical Studies in Fused Salts", First Latin American Electrochemistry and Corrosion Meeting (ABRACO), Rio de Janeiro, Brazil, October 21-25, 1974.

1975

\*R. A. Osteryoung, "Some Applications of Pulse Techniques to Analytical Chemistry and Electrochemistry", University of Brussels, Brussels, Belgium, March 17, 1975.

V. R. Koch, L. L. Miller and R. A. Osteryoung, "Electroinitiated Fiedel-Crafts Transalkylation in a Room Temperature Molten Salt Medium", Mile High Electrochemistry Symposium, Colorado Electrochemical Society Section, Fort Collins, CO, May 3, 1975.

K. A. Paulsen and R. A. Osteryoung, "Electrochemical Studies of Sulfur in Molten Sodium Tetrachloroaluminate", Fall Meeting, American Chemical Society, Chicago, Illinois, August 1975.

\*R. A. Osteryoung, "Electrochemical Studies in Molten Sodium Chloride-Aluminum Chloride", Colorado College, Colorado Springs, Colorado, December 4, 1975.

1976

\*R. A. Osteryoung, "Chemical and Electrochemistry in Aluminum Chloride Molten Salt Systems", International Symposium on Molten Salts, Spring Meeting, The Electrochemical Society, Washington, D.C., May 2-7, 1976.

\*R. A. Osteryoung, "Electrochemical Studies in Molten Chloroaluminates", Gould, Inc., Rolling Meadows, Illinois, May 4, 1976.

\*R. A. Osteryoung, "Chemical and Electrochemical Studies in Molten Chloroaluminate Solvents", Department of Chemistry Colloquium, Purdue University, West Lafayette, Indiana, March 23, 1976.

\*R. A. Osteryoung, "Acid-Base Dependent Electrochemistry in Aluminum Halide Molten Salts", Gordon Research Conference on Molten Salts and Metals, Tilton, N.H., July 24-29, 1977.

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Molten Chloroaluminates", Georgia Institute of Technology, Atlanta, GA, October 12, 1977.

\*J. Robinson and R. A. Osteryoung, "The Electrochemistry of Selenium in Molten Sodium Tetrachloroaluminates", 3rd Annual Mile High Symposium on Electrochemistry, Rocky Mountain Section of the Electrochemical Society, Fort Collins, CO, May 7, 1977.

#### 1978

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Molten Chloroaluminates", Georgetown University, Washington, D.C., February 9, 1978.

R. A. Osteryoung and B. Gilbert, "Electrochemistry of Nickel in Molten Sodium Tetrachloroaluminates", National American Chemical Society Meeting, Anaheim, CA, March 12-17, 1978.

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Molten Chloroaluminates", Wichita State University, Wichita, KS (April, 1978).

\*R. A. Osteryoung, "Electrochemistry in Molten Chloroaluminates", Naval Surface Weapons Center, White Oak, Maryland (May, 1978).

\*R. A. Osteryoung, "Acid Base Dependent Electrochemistry in Chloroaluminate Melts", Conference on Highly Concentrated Aqueous Solutions and Molten Salts, Oxford University, Oxford, England (July 5-7, 1978).

\*R. A. Osteryoung, "Acid Base Dependent Chemistry and Electrochemistry in Chloroaluminate Melts", The University of Southampton, Southampton, England (July 10, 1978).

\*R. A. Osteryoung, R. J. Gale, J. Robinson, R. Bugle and B. Gilbert, "Electrochemical Studies in a Room Temperature Molten Salt", Second International Meeting on Molten Salts, The Electrochemical Society, Pittsburgh, PA (October 15-20, 1978).

\*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Molten Chloroaluminates", Gould, Inc., Rolling Meadows, IL, November 16, 1978.

\*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Molten Chloroaluminates", Colorado Section, American Chemical Society, University of Colorado, December 12, 1978 (Colorado Section Award Talk).

#### 1979

\*R. A. Osteryoung, "Room Temperature Molten Salts: A New Class of Solvents" Gordon Research Conference on Electrochemistry, Santa Barbara, CA, January 7-12, 1979.

\*R. A. Osteryoung, "Studies in Molten Chloroaluminates", Oak Ridge National Laboratory, Oak Ridge, TN, February 2, 1979.

\*R. A. Osteryoung, J. Robinson and R. J. Gale, "Oxidation of Aromatic Hydrocarbons in a Room Temperature Molten Salt", Symposium on Electrochemistry and Spectroscopy in Melts, American Chemical Society/Chemical Society of Japan Congress, Honolulu, Hawaii, April 1-6, 1979.

James Robinson and R. A. Osteryoung, "Electrochemical Studies on Some Aromatic Amines in a Room Temperature Molten Salt", Spring Meeting, The Electrochemical Society, St. Louis, MO, May 11-16, 1979.

\*R. J. Gale, A. J. Nozik and R. A. Osteryoung, "Electrochemical Characterization of the Semiconductor  $TiO_2$ -Room Temperatures Molten Salt Interface", Rocky Mountain Regional Electrochemical Society Meeting, Ft. Collins, CO (June, 1979).

\*G. Cheek and R. A. Osteryoung, "Electrochemical and Infrared Studies of Quinones in a Room Temperature Molten Salt", Gordon Research Conference on Molten Salt", Gordon Research Conference on Molten Salts and Metals, Brewster Academy, Wolfeboro, New Hampshire (August 20-24, 1979).

\*R. J. Gale, J. Robinson and R. A. Osteryoung, "Acid Base Dependent Chemistry and Electrochemistry in Room Temperature Chloroaluminate Melts", Plenary Lecture, 30th Meeting, International Society of Electrochemistry, Trondheim, Norway (August 26-31, 1979).

R. A. Osteryoung, G. Cheek and R. J. Gale, "Correlation of Infrared and Electrochemical Studies on Quinones in a Room Temperature Molten Salt", Fall Meeting, American Chemical Society, Washington, D.C. (September 9-14, 1979).

G. Cheek and R. A. Osteryoung, "Electrochemical and Spectroscopic Studies in Quinones in a Room Temperature Molten Salt", Northeast Regional American Chemical Society Meeting, Syracuse, NY (October 2-5, 1979).

\*R. A. Osteryoung, "Simple Chemistry in Molten Salts", State University College at Brockport, Brockport, NY, October 17, 1979.

\*R. A. Osteryoung, "Chemical and Electrochemical Studies in Molten Chloroaluminates", Colloquium, Department of Chemistry, State University College at Buffalo, Buffalo, NY, November 7, 1979.

Janet Osteryoung and T. R. Brumleve, "Reverse Pulse Polarography: Spherical Diffusion and Depletion Effects", 178th ACA National Meeting, Washington, D.C., September, 1979.

Janet Osteryoung, Koichi Aoki and R. A. Osteryoung, "Modified Differential Pulse Voltammetry: Theory", 9th NE Regional ACS Meeting, Syracuse, October, 1979.

\*Janet Osteryoung, Recent Advances in Pulse Polarography, Georgetown University, Washington, D.C., November, 1979.

#### 1980

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Molten Chloroaluminates", Canisius College, Buffalo, NY, Feb. 14, 1980.

\*R. A. Osteryoung, "Chemical and Electrochemical Studies in Molten Chloroaluminates", Colloquium, Department of Chemistry, University of Pittsburgh, Pittsburgh, PA, Feb. 28, 1980.

\*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Molten Chloroaluminates", University of North Dakota, Grand Forks, North Dakota (Department Colloquia), March 14, 1980.

\*R. A. Osteryoung, "Electrochemical Studies in Molten Chloroaluminates", Sioux Valley American Chemical Society Section, Sioux Falls, South Dakota, March 15, 1980 (ACS Tour Speaker).

\*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Chloroaluminate Melts", Pittsburgh Section, The Electrochemical Society, Spring Meeting, May 23, 1980.

\*R. A. Osteryoung, Graham Cheek and Hogné Linga, "Studies in Room Temperature Molten Salts", Third International Symposium on Molten Salts, The Electrochemical Society, Fall Meeting, Hollywood, Florida, October 5-10 (1980).

\*Janet Osteryoung, "Recent Advances in Pulse Voltammetry", Pittsburgh Conference, Atlantic City, March, 1980.

Janet Osteryoung and Edmund Hurdle, Jr., "Construction of a Potentiostat for Use in Computer-Assisted Electrochemistry", 34th Eastern Colleges Science Conference, Cortland, NY, April, 1980.

\*Janet Osteryoung, "The Use of Pulse Techniques for Detection of Intermediate Products of Electrode Reactions", J. Heyrovsky Memorial Congress on Polarography, Prague, Czechoslovakia, August, 1980.

\*Janet Osteryoung, "Recent Advances in Pulse Voltammetry", 3rd Brno Symposium on Molecular Biophysics: Electroanalysis of Biopolymers, Kupařovice Castle, Czechoslovakia, August-September, 1980.

\*Robert A. Osteryoung and Janet G. Osteryoung, "Pulse Voltammetric Methods of Analysis", Symposium on "Prospects of Industrial Electrochemistry", The Royal Society, London, England, December 10-11, 1980.

#### 1981

\*R. A. Osteryoung, "Introduction to Session", Session on High Temperature Chemistry, Gordon Research Conference on Electrochemistry, Ventura, California, January 25-30, 1981.

\*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Chloroaluminate Melts", Allied Chemical, Buffalo, N.Y., March 19, 1981.

Janet Osteryoung, R. A. Osteryoung and Timothy Brumleve, "Applications of Differential Normal Pulse Voltammetry to Irreversible Reactions", Spring Meeting, American Chemical Society, Atlanta, GA, March 29-April 3, 1981.

\*R. A. Osteryoung, R. J. Gale, J. Robinson, H. Linga and G. Cheek "Chemical and Electrochemical Studies in a Room Temperature Ionic Liquid", Symposium on Nonaqueous Electrolytes, The Electrochemical Society, Spring Meeting, Minneapolis, Minnesota, May 10-15, 1981.

\*R. A. Osteryoung and Janet G. Osteryoung, "Recent Advances in Pulse Voltammetry", Trent University, Peterborough, Canada, April 3, 1981.

\*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Chloroaluminate Melts", SRI, Inc., Menlo Park, California, July 23, 1981.

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\*R. A. Osteryoung, "Ambient Temperature Molten Salts - A New Class of Solvents", Penn-York American Society Section, Jamestown, New York November 7, 1983 (ACS Tour Speaker).

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M. Lipsztajn and R. A. Osteryoung, "Electrochemistry in Ambient Temperature Neutral Ionic Liquids", The Electrochemical Society, Spring Meeting, Cincinnati, Ohio, May 6-11, 1984.

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\*Saeed Sahami, P. G. Pickup and R. A. Osteryoung, "Studies in Ambient Temperature Chloroaluminates; Electrochemistry of Polypyridine Complexes and Polymeric Electrodes", EUCHEM Conference on Molten Salts, Elsinore, Denmark, August 19-25, 1984.

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Z. Karpinski, C. Nanjundiah and R. A. Osteryoung, "Electrochemistry of Ferrocene and Ferrocinium Ion in Ambient Temperature Ionic Liquids", The Electrochemical Society, Fall Meeting, New Orleans, LA, October 7-12, 1984.

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\*M. Lipsztajn, S. Sahami, T. A. Zawodzinski, Jr. and R. A. Osteryoung, "Amperometric Titrations in Ambient Temperature Ionic Liquids", Symposium on Spectroelectrochemistry and Electroanalysis, The Electrochemical Society, Spring Meeting, Toronto, Canada, May 12-17, 1985.

Thomas A. Zawodzinski, Jr., M. Lipsztajn, R. J. Kurland and R. A. Osteryoung, "Characterization of Lewis Acidity of a Room Temperature Ionic Liquid", General Session, The Electrochemical Society, Spring Meeting, Toronto, Canada, May 12-17, 1985.

T. A. Zawodzinski, Jr., M. Lipsztajn, R. J. Kurland and R. A. Osteryoung, "Characterization of a Room Temperature Ionic Liquid", Third Annual International Symposium for Chemistry Grad-Students, State University of New York at Buffalo, Buffalo, NY, May 22-23, 1985.

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\*R. A. Osteryoung, "Aspects of Ambient Temperature Molten Salts", Molten Salt Discussion Group, London, England, December 16, 1985.

\*R. A. Osteryoung, "Room Temperature Molten Salts as Battery Media", Conference on Physical Chemistry of Battery Electrode Reactions, University of Southampton, Southampton, England, December 18, 1985.

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\*R. A. Osteryoung, "Chemistry and Electrochemistry in Ambient Temperature Ionic Liquids", Department of Physical Chemistry Colloquium, University of Bristol, Bristol, England, February 3, 1986.

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Ambient Temperature Ionic Liquids", Department of Inorganic and Structural Chemistry, University of Leeds, Leeds, England, February 5, 1986.

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Ambient Temperature Ionic Liquids", Department of Inorganic Chemistry, University of Sussex, Falmer, England, February 27, 1986.

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Ambient Temperature Ionic Liquids", Institute of Dynamics and Thermophysics of Fluids, University de Provence, Marseilles, France, April 27, 1986.

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Ambient Temperature Ionic Liquids", Department of Organic Chemistry, University of Grenoble, Grenoble, France, April 29, 1986.

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Ambient Temperature Ionic Liquids", Atomic Energy Research Establishment (AERE), Harwell, England, May 7, 1986.

\*R. A. Osteryoung, "New Pulse Methods in Electroanalytical Chemistry", ALCOA Laboratories Centennial Technical Symposia, Electrochemistry, Nemacon, Pennsylvania, June 1 - 5, 1986.

Richard T. Carlin and R. A. Osteryoung, "Molybdenum Dimers in Ambient Temperature Molten Salts", General Session, Division of Inorganic Chemistry, Northeast Regional ACS Meeting, Binghamton, New York, June 22-25, 1986.

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Francisco Uribe, T. Zawodzinski and R. A. Osteryoung, "A Study of 1,4-Hydroquinone and 1,4-Benzoquinone in Room Temperature Chloroaluminate Melts", Northeast Regional ACS Meeting, Binghamton, New York, June 22-25, 1986.

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