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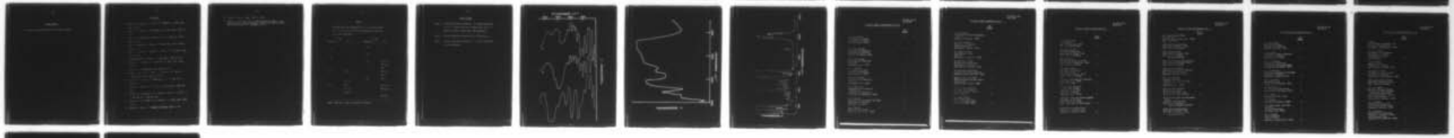

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INFRARED SPECTRAL INVESTIGATIONS OF AMBIENT MOLTEN ALUMINUM CHLORIDE: 1-BUTYLPYRIDINIUM CHLORIDE SYSTEMS

by

R. J. GALE AND R. A. OSTERYOUNG

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A direct transmission infrared technique has been applied to obtain spectra of acidic and basic $AlCl_3$: 1-butylpyridinium chloride melts. Band assignments for the $AlCl_4^-$ anion show splitting of the ν_3 mode and confirm tetrahedral distortion. Bands for the $Al_2Cl_7^-$ anion in the 200-650 cm^{-1} region for the 2:1 mole ratio melt and solid $Tl_4(Al_2Cl_7)_2$ support the premise that the ion in the liquid phase has a bent Al-Cl-Al bridge. Detailed spectra also have been obtained of the 1-butylpyridinium cation and the absence of extensive band broadening may indicate that the melt structure is quasi-crystalline.

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AlCl4(-)

Al2Cl7(-)

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by

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ABSTRACT

A direct transmission infrared technique has been applied to obtain spectra of acidic and basic $AlCl_3$: 1-butylpyridinium chloride melts. Band assignments for the $AlCl_4^-$ anion show splitting of the ν_3 mode and confirm tetrahedral distortion. Bands for the $Al_2Cl_7^-$ anion in the 200-650 cm^{-1} region for the 2:1 mole ratio melt and solid $Tl_4(Al_2Cl_7)_2$ support the premise that the ion in the liquid phase has a bent Al-Cl-Al bridge. Detailed spectra also have been obtained of the 1-butylpyridinium cation and the absence of extensive band broadening may indicate that the melt structure is quasi-crystalline.

INTRODUCTION

Structural investigations of the haloaluminate anionic species in molten mixtures by vibrational spectroscopic methods are hampered to varying extents by practical problems. For this reason, the experimentally more applicable Raman techniques in general have been preferred over infrared methods, e.g., [1,2], although a Fourier transform infrared system has been applied to obtain emission spectra of $MAlCl_4$ melts at 160-250° (M = Li, Na, K) [3]. As certain binary $AlCl_3$:alkylpyridinium halide mixtures are ionic liquids at ambient temperatures, these systems are well suited for acid-base studies, as well as having interesting implicit properties. Also, their low liquidus characteristics facilitate experimental spectroscopic studies. The primary objective of this study was to assess the extent to which additional vibrational information might be obtained for the $AlCl_4^-$ and $Al_2Cl_7^-$ anionic species.

RESULTS

Aluminum tetrachloride ion ($AlCl_4^-$)

Figure 1B illustrates the IR absorption spectrum for a 1:1 mole ratio $AlCl_3$-1BuPyCl liquid obtained by the transmission technique for the 200-650 cm^{-1} spectral region and the bands assignable to the $AlCl_4^-$ anion are reported in Table 1. An extremely broad, intense peak is centered at 490 cm^{-1} with shoulders at 476 and 525 cm^{-1}. A weak band usually could be resolved at $\sim$350 cm^{-1} despite a similarly intense band in the region due to the polyethylene windows.

Most Raman studies of the $AlCl_4^-$ anion in melts have been interpreted in terms of the ion possessing tetrahedral (T_d) symmetry [2,4,5], although Balasubrahmanyam and Nanis [6] reported nine frequency shifts for each of liquid and solid $AlCl_3$:KCl phases and proposed that the tetrahedral symmetry is distorted (C_{2v}). Their result is at variance with the more careful study of $AlCl_3$:KCl melts by Oye, et al. [5]. An undistorted tetrahedron possesses 4 Raman-active vibrations ($A_1 + E + 2F_2$) and 2 IR-active ($2F_2$) from point-group symmetry rules. In basic (chloride-rich) $AlCl_3$:1-BuPyCl, the Raman bands assigned to $AlCl_4^-$ are 351 $\nu_1(A1_1)$, 126 $\nu_2(E)$, 484 $\nu_3(F_2)$ and 184 cm^{-1} $\nu_4(F_2)$ [2]. Splitting of the weakly Raman-active ν_3 mole only can be used to determine whether or not the tetrahedral species is perturbed structurally if its band intensity permits. A better indication may be provided by splitting of the strongly IR-active ν_3 mode, when the normally inactive ν_1 and ν_2 modes may appear also. Smyrl, Mamantov and McCurry [3] observed some additional band structure from IR emission spectra for ν_3 in the molten $AlCl_3$:LiCl, NaCl and KCl spectra but decomposition of the melts complicated the evaluation of anion perturbations by their emission technique. An IR study of the solid compounds Te_2AlCl_4, Te_3AlCl_4, Se_4AlCl_4 and $NaAlCl_4$ has found that their anionic ν_3 modes are split [7] and a weak ν_1 mode at 347 cm^{-1} has been reported to occur in the spectrum of the $PCl_5 \cdot AlCl_3$ adduct $(PCl_4^+)(AlCl_4^-)$ [8]. Raman studies of the $LiAlCl_4$ melt have revealed that the ν_3 mode splits at 473, 498 and 512 cm^{-1} and that the structural distortion decreases in the alkali metal series $Li^+ > K^+ > Cs^+$ [9].

Dialuminum heptachloride ion ($Al_2Cl_7^-$)

A typical IR spectrum of 2:1 molar ratio $AlCl_3$:1-BuPyCl melt is shown in Figure 1C for the 200-650 cm^{-1} region and the bands attributable to the $Al_2Cl_7^-$ ion are listed in Table I. There is relatively little IR data available for comparison but two IR peaks at 530 and 480 cm^{-1} present in acetyl heptachlorodialuminate have been assigned to Al-Cl modes of the $Al_2Cl_7^-$ anion [10]. Previous attempts to correlate Raman vibrational data of melts to a model structure have not been particularly successful [5,9] and Oye and coworkers have been reluctant to propose a bent Al-Cl-Cl bridge because of the excessive number of vibrations predicted for such a structure over those found experimentally. Methods other than vibrational spectroscopy used to investigate the structures of dialuminum heptahalide ions, for example, X-ray crystallography of the solids KAl_2Br_7 [11], $Pd_2(C_6H_6)_2(Al_2Cl_7)_2$ [12], and $Te_4(Al_2Cl_7)_2$ [13] confirm the presence of a bent bridge. In addition, applications of the NQR double-resonance technique to solid KAl_2Br_7 have produced results consistent with the crystallographic findings ([14], references therein). As a first order approximation, it is of interest to attempt to correlate the far IR spectrum of $Te_4(Al_2Cl_7)_2$ to that of the 2:1 $AlCl_3$:1-BuPyCl melt. Figure 2 contains the spectrum of $Te_4(Al_2Cl_7)_2$ and it is apparent that the overall profile in 300-650 cm^{-1} region corresponds closely to that of the 2:1 $AlCl_3$:1-BuPyCl spectrum (Figure 1C). The frequencies and intensities of the major bands for $Al_2Cl_7^-$ ion in solid $Te_4(Al_2Cl_7)_2$, at 535(S,br), 485(s,sh), 430(m), 384(s), 375(s,sh), 338(m), 334(m,sh) and 318 cm^{-1} (m,sh), are very similar to the bands found for $Al_2Cl_7^-$ ion in the molten salt (cf. Table I). Spectra of molecules fixed in a crystal lattice are affected by symmetry rules determined by the lattice symmetry constraints as well as the molecular point-group symmetry. Nevertheless, it seems most probable that the $Al_2Cl_7^-$ anion in the melts has a bent Al-Cl-Al bridge and that the small number of vibrational modes detected previously in the Raman mode is due to the experimental difficulties of attaining detailed spectral resolution.

1-butylpyridinium cation ($C_9H_{14}N^+$)

The IR spectrum of the cation measured in a basic 0.8:1 molar ratio liquid is illustrated in Figure 3. In view of the relatively weak peak intensities in the regions 800-1150, 1200-1450, 1510-1620, 1650-2800 and >3100 cm^{-1}, it should be relatively easy in many instances to identify organic solutions in these melts. The use of differential liquid cells or background subtraction methods might also be applied advantageously to obtain vibrational information of solutes.
Spinner [15] has made a detailed analysis of the vibrational spectrum of 1-methylpyridinium cation and tentative band assignments might be possible on the basis of this data. A few differences occurred in the spectrum of an acidic 2:1 melt to the basic melt spectrum shown in Figure 3: a shoulder appeared instead of the weak band at 1256 cm^{-1}, a medium band replaced the shoulder at 1360 cm^{-1}, and the pattern of

bands at circa 1450-1500 cm^{-1} modified in intensity. Far more differences in the 600-1600 cm^{-1} region were found in the spectrum of solid 1-butylpyridinium chloride in a nujol mull and only two sharp peaks (503 and 580 cm^{-1}) were resolved in the 200-600 cm^{-1} region.

DISCUSSION

In the $LiAlCl_4$ melt, splitting of ν_3 by symmetry lowering has been explained by the tendency of the small Li^+ cation to form partly covalent Li-Cl bonds with the $AlCl_4^-$ anion and this tendency decreases in the alkali metal series $Li^+ > K^+ > Cs^+$ [10]. It is improbable that the splitting effect manifested in the $AlCl_3$:1-BuPyCl system arises from the same direct cationic polarizability behavior in view of the large, soft cation. Assuming the absence of specific non-Coulombic forces, screening by the pyridinium π-system and the alkyl group may decrease the effective dielectric constant of the solvent. The result of lowering secondary iterionic attractive and repulsive forces would thus promote contact anion-cation pair formation. It is difficult, however, to assess the relative magnitude of the splitting effect with respect to those found in the alkali metal systems, without comparative IR data.

The spectral differences between basic and acidic melts at cation frequencies could reflect a difference in the cation:anion interactions or simply be a result of packing constraints. The importance to physical properties of the nature of the anion can be considerable and the acidic ($Al_2Cl_7^-$ containing) system is liquid at far lower temperatures than the basic melts. Detailed band assignments from isotropic studies might be of value in this regard. For $C_5D_5N^+CH_3$, the asymmetric CH_3 bending band at 1452 cm^{-1} broadens in aqueous solution, thereby satisfying criterion for a low barrier to alkyl-group rotation [15]. In general, the spectra of the melts seem to be similar to that of the solid chloride salt in this regard and the rotational freedom may be restricted by a quasi-crystalline lattice type of solvent structure. More detailed IR studies could aid in answering questions as to how the rotational and molecular entropy factors are related to the low liquidus temperatures of these melt systems.

Additional applications of infrared studies on solutes in these systems will be forthcoming [16].

EXPERIMENTAL

Preparation of chemicals and melts have been described previously [2). Liquid IR cells were filled and sealed with teflon stoppers in a drybox containing a purified argon atmosphere. Spectra in the 200-650 cm^{-1} regions for the 2:1 and 1:1 melts were recorded on a Beckman IR 12 spectrometer with 0.045 inch polyethylene windows and a 0.003 inch teflon spacer. A conventional NaCl liquid cell (Fisher Scientific) was used for the 4000-650 cm^{-1} experiments. The purple solid $Te_4(Al_2Cl_7)_2$ compound was prepared according to the procedure of Couch, et al. [13] and its IR obtained in a nujol mull between polyethylene plates. An attempt to record its Raman spectrum was unsuccessful.

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TABLE I

Infrared vibrational frequencies (cm^{-1}) of chloroaluminate ions in basic and acidic $AlCl_3$:1-butylpyridinium chloride melts (room temperature).

$AlCl_4^-$		$Al_2Cl_7^-$	
Raman [2]	IR	Raman [2]	IR
		102	
126			
		163	
184			
		315	315 m,sh
			328 s,sh
			334 s
351	353 w		378 s,sh
			386 s
		434	343 m
	476 s,sh		
484	490 s		500 s,sh
	525 s,sh		$\sim$545 vs, br
	550 w,sh		
			585 m,sh

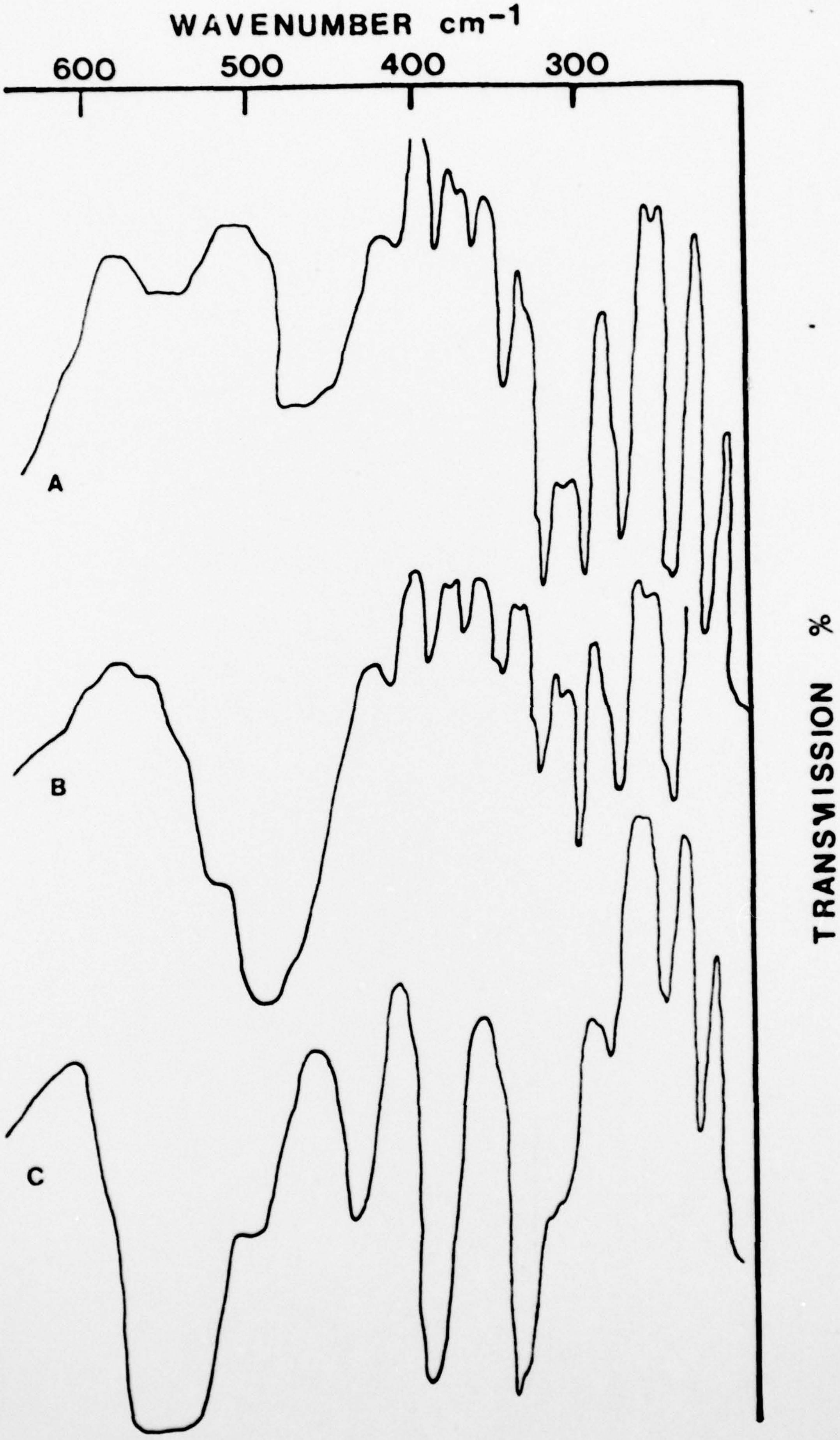
w-weak, m-medium, s-strong, sh-shoulder, br-broad

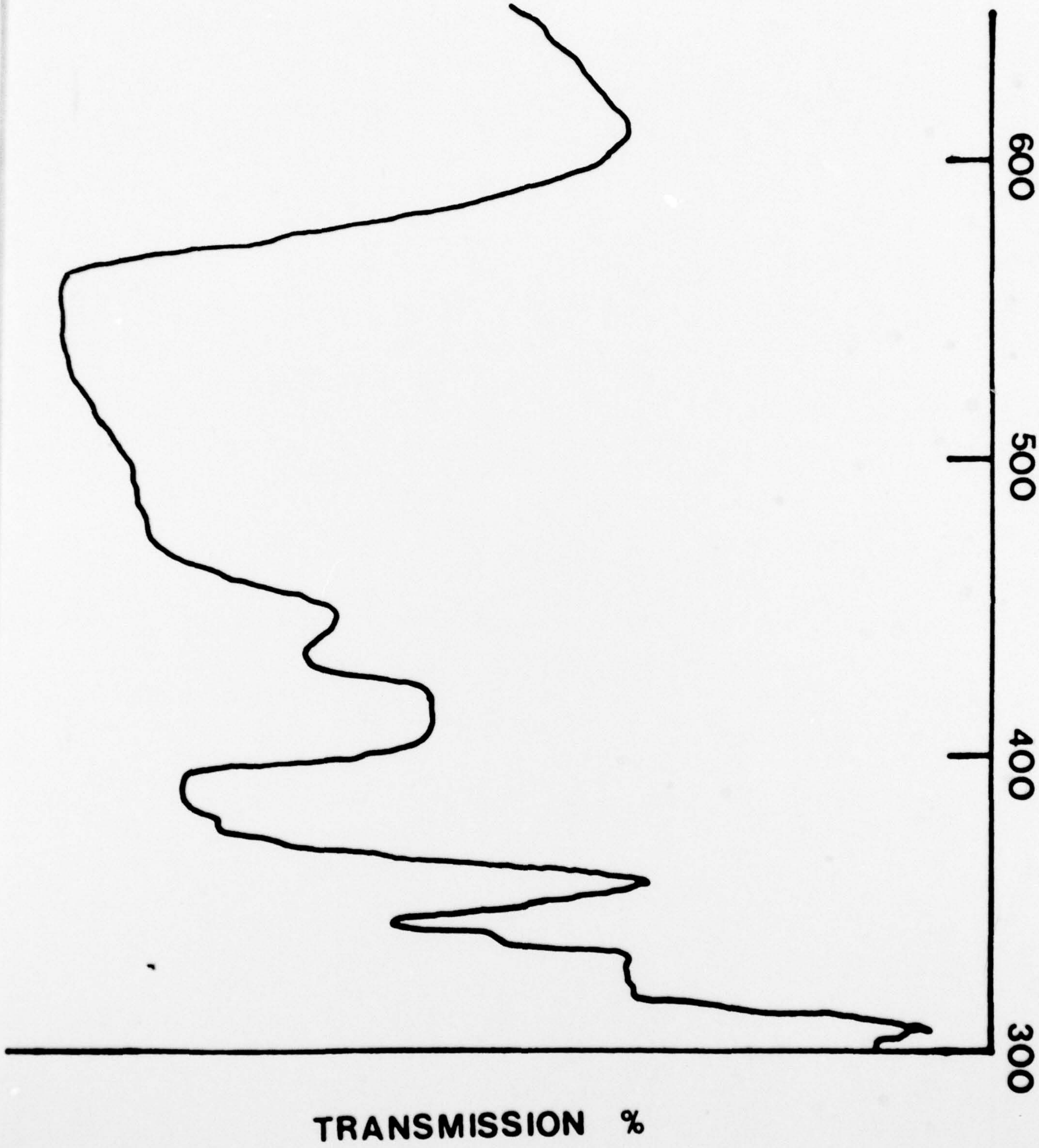
FIGURE LEGENDS

Figure 1. Infrared transmission spectra of: A. Empty polyethylene cell, B. 1:1 molar ratio $AlCl_3$:1-BuPyCl melt, and C. 2:1 molar ratio $AlCl_3$:1-BuPyCl melt, room temperature.

Figure 2. Infrared transmission spectrum of solid $Te_4(Al_2Cl_7)_2$.

Figure 3. Infrared transmission spectrum of 1:1 $AlCl_3$:1-BuPyCl melt at room temperature.


WAVENUMBER cm-1
600
500
400
300
A
B
C
TRANSMISSION %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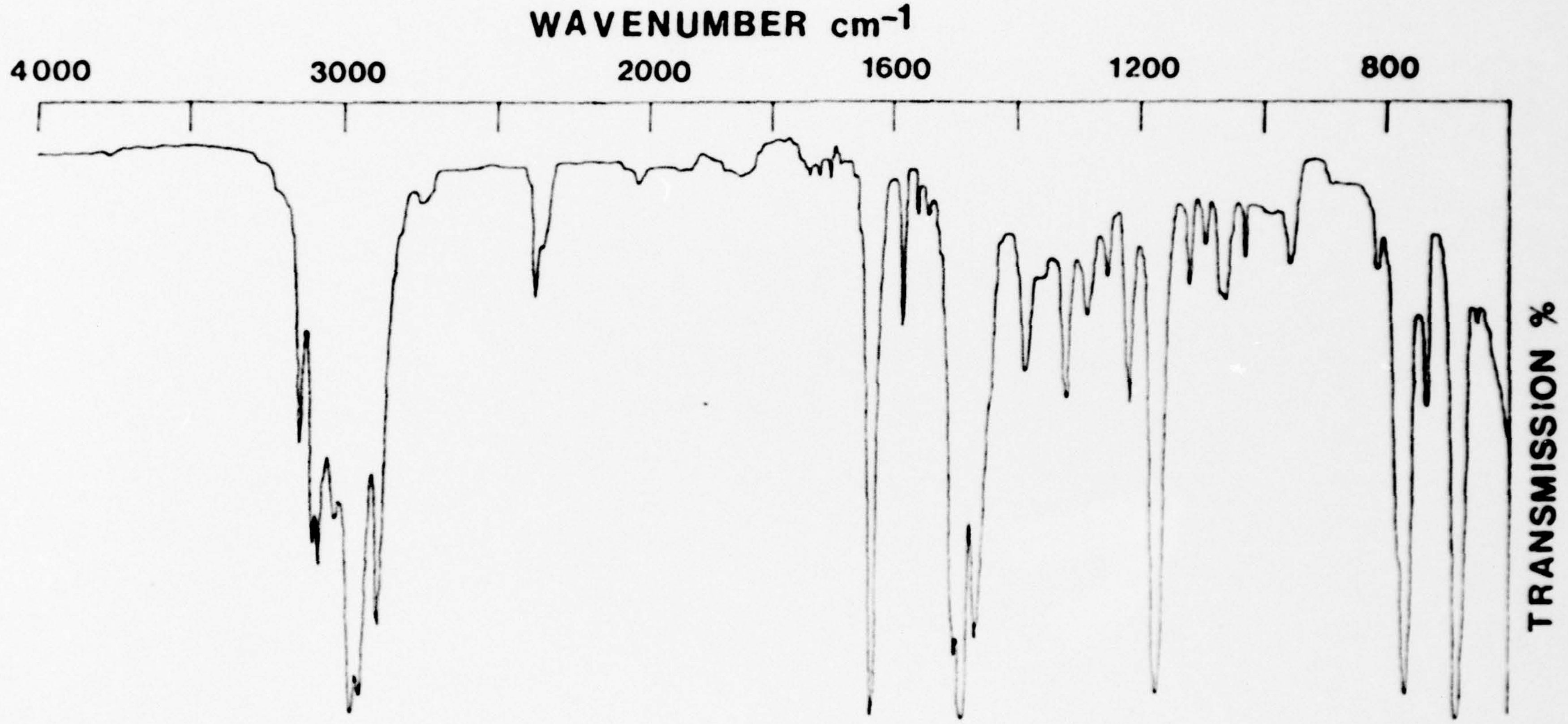WAVENUMBER cm-1
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3000
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1200
800
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