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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Ultrasonic absorption data are reported in the frequency range 1-7 MHz and 10-300 MHz for the electrolytes LiAsF ₆ and LiBF ₄ at t=25°C, in 2-methyltetrahydrofuran. The data can be described by a single Debye relaxation for LiAsF ₆ and by two Debye relaxation processes for LiBF ₄ . The observed processes are in- terpreted by a multistep Eigen type dimerization process leading to contact quadrupoles. In the case of LiAsF ₆ the products are		

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KINETICS OF QUADRUPOLE FORMATION OF LITHIUM SALTS
IN 2-METHYLTETRAHYDROFURAN AT 25°C

Technical Report #2

by

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Report

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ABSTRACT

Ultrasonic absorption data are reported in the frequency range 1-7 MHz and 10-300 MHz for the electrolytes LiAsF_6 and described by a single Debye relaxation for LiAsF_6 and by two Debye relaxation processes for LiBF_4 . The observed processes are interpreted by a multistep Eigen type dimerization process leading to contact quadrupoles. In the case of LiAsF_6 the products are solvent separated quadrupoles and contact dimers, whereas for LiBF_4 contact species predominate. Literature data for LiClO_4 suggest that the dimers are the major species present. Hence the extent of dimerization process for the three electrolytes seems to follow the order: $\text{LiAsF}_6 \approx \text{LiBF}_4 < \text{LiClO}_4$.

INTRODUCTION

In recent years research on lithium electrolytes in ether solvents has focused on studies of solvation and ion association aimed at elucidating the structure and dynamics of these systems¹. The practical aspect of this current interest is the knowledge of the species in the same solutions which are used in constructing secondary batteries with lithium electrodes². The interpretation of the results is expected to give guidance toward choosing solutions which have stability toward the electrode and a minimum internal resistance (maximum conductivity) etc.

Classical tools such as electrical conductance suffer from limitations of the developed theory to a range of concentrations far below any practical use and to insensitivity to the appearance of neutral species as dimer-ion-pairs as opposed to single ion-pairs (not to speak of outer-inner sphere species).

On the other hand a dynamic tool as ultrasonic relaxation allows for the kinetic study of the rates of formation and dissociation of various species present in solution. Evaluation of the equilibrium constants of the various species is sometimes possible. In this method the detection of a relaxation process depends on the adiabatic volume change of reaction. Solvation and desolvation phenomena then, are particularly important and outer-sphere inner-sphere processes are often studied.

2 Methyltetrahydrofuran (2MTHF) is a solvent particularly relevant for the above purposes because of the proven stability of lithium

metal in contact with it². We have studied two electrolytes LiAsF_6 and LiBF_4 and compared the results with LiClO_4 . in an effort to provide a picture of the effect of the nature of the anion on the process of ionic complexation. Later we shall extend the same study to the effect of the solvent. It is hoped that the study, when completed, will offer a rather clear picture of the competition effect of anion complexation and solvation as well as of the effect of the nature of the anion and of the solvent in such competition.

EXPERIMENTAL PART

The equipment for the ultrasonic work consisted of a Matec (Warwick, R.I.) 6600 pulser-receiver unit equipped with tuning heads covering the range 10-90 and 90-300 MHz. A Matec pulse comparator and a master synchronizer completed the unit. The twin crystal interferometric ultrasonic cell was equipped with two lithium niobate coaxially gold plated crystals cut $+36^\circ$ with respect to the Y axis and furnished by Volpey Fisher (Hopkinton, Mass.) This new set-up delivered ultrasonic decay-echoes of much improved signal over noise ratio with respect to the previously described equipment using X-cut quartz crystals³. With water as the absorbing medium, in preliminary tests, it was possible to reach the 57th harmonic of the crystal (~570 MHz) with an acceptable signal over noise ratio signal. Coupling the crystal to the delay line was accomplished with a film of

non-aqueous grease instead of the silicon grease used for aqueous solutions.

The remainder of the pulse equipment was basically the same as before³. For the measurements below 10 MHz, specifically for ~1 to 4 MHz and for 6 to 7 MHz an ultrasonic cylindrical resonator cell of new construction and a sweeper-receiver set were used. The main design of the cell is similar to the one of Eggers and Funck⁴ with some minor changes which basically consist in using a home-made teflon bellow to space the two X-cut, 1 inch, 5 MHz fundamental, quartz crystals. The bellow allows for sufficient elasticity for micrometric orientation of the two crystal holders and also holds the liquid under study without leakage. Teflon washes pressed in between the (back rubber pressed) crystals and the lips of the holders maintain a tight seal. The rest of the cell in contact with the liquid is made of stainless-steel. The authors are grateful to Dr. Theodor Funck of the Max-Planck Institute for having shown the cell during a summer visit of one of us to Goettingen. Details of the cell construction will be reported elsewhere⁵.

Thermostating of the resonator cell was provided by a circulating bath which was maintained at 25.0°C by the aid of a Bailey proportional thermoregulator (with an infrared lamp set outside the bath to eliminate thermal inertia). Initially a cryostatic unit of on-off type cooled the bath when needed.

Later its use was avoided when possible as it caused temperature oscillations of $\pm 0.1^{\circ}\text{C}$. Rather the room was air-conditioned at temperature around 70F to allow for a constant and mild heat dissipation from the resonator cell. For the materials, LiAsF_6 (α -Inorganics) was dried in vacuums at about 60°C overnight. LiBF_4 (α -Inorganics) was treated likewise. 2-Methyltetrahydrofuran (2MTHF) was distilled under reduced pressure over Na metal and benzophenone after refluxing until the boiling solution remained blue, a sign of absence of water and peroxides. Solutions were prepared by weighing the salts in situ in volumetric flasks, the same containers used for drying, and adding the freshly distilled solvent. Solutions were kept in a dessicator and used as soon as possible after preparation. No solution was used after more than 1-2 days after preparation. LiAsF_6 in 2MTHF shows a gradual yellow discoloration with time. However ultrasonic experiments carried over somewhat discolored solutions did not show any difference within experimental error of corresponding measurements performed on solutions used just after dissolution of the salts and thermostating the ultrasonic cell.

RESULTS

Figure 1 shows a representative plot of the quantity α/f^2 vs the frequency f for LiAsF_6 in 2MTHF at 25°C . The solid line is the calculated Debye function for a single relaxation process:

$$\frac{\alpha}{f^2} = \frac{A}{1 + (f/f_r)^2} + B \quad (I)$$

In the above α is the absorption coefficient of sound expressed in neper cm^{-1} , f_r the relaxation frequency, A a relaxation parameter and B the value of α/f^2 at $f \gg f_r$, not necessarily equal to the value of α_0/f^2 for the solvent.

Table I reports the relaxation parameters A , f_r and B together with the sound velocity $u(\text{cms}^{-1})$ and the maximum excess sound absorption per wavelength $\mu_m = (\frac{1}{2}A f_r u)$ for LiAsF_6 in 2MeTHF at 25°C for the concentrations c (mole dm^{-3}) investigated.

Figure 2 reports a representative plot of the quantity $\mu (= \alpha - Bf^2) \frac{u}{f}$, the excess sound absorption per wavelength, plotted vs the frequency f , for LiBF_4 in 2MTHF at 25°C . The solid line corresponds to the sum of two Debye processes according to the function:

$$\mu = 2\mu_{mI} \frac{f/f_{rI}}{1 + (f/f_{rI})^2} + 2\mu_{mII} \frac{f/f_{rII}}{1 + (f/f_{rII})^2} \quad (II)$$

where $\mu_{mI} = \frac{1}{2}A_I u f_{rI}$ and $\mu_{mII} = \frac{1}{2}A_{II} u f_{rII}$. The same data could have been expressed in a less illustrative way by:

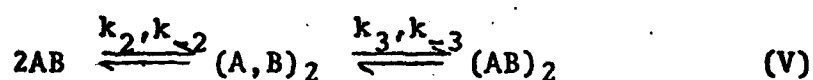
$$\frac{\alpha}{f^2} = \frac{A_I}{1 + (f/f_{rI})^2} + \frac{A_{II}}{1 + (f/f_{rII})^2} + B \quad (III)$$

In other words the data can be best described by the sum of two Debye relaxation processes. The function for a single relaxation I is unable to describe the data adequately.

Table II reports the parameters μ_{mI} , fr_I , μ_{mII} , fr_{II} and B for $LiBF_4$ in 2MTHF for all the concentrations investigated at 25°C. The sound velocities $u(\text{cms}^{-1})$ are also reported for the same systems.

DISCUSSION

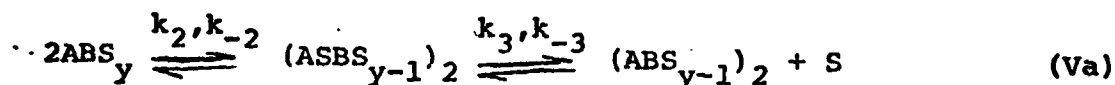
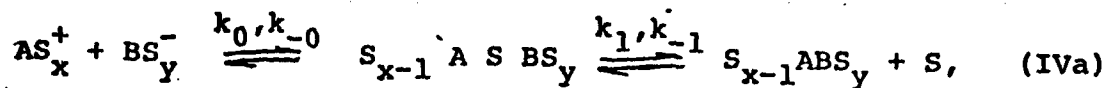
Previous studies from this laboratory¹⁻⁶ and previous data⁷ seems to convey the following picture for the process of ionic complexation of ethereal lithium salt solutions.



Where A, B and AB are the outer sphere and inner sphere ion pairs respectively, whereas $(A, B)_2$ and $(AB)_2$ are the corresponding species for the dimers or quadrupole species.

In a way processes IV and V are extensions of the Eigen multistage scheme of ionic association. It is clear that at a given concentration, in a given solvent, the predominant species (and hence the likelihood of observing a given process) will depend on the nature of B , namely its structure, donor ability and

the like. Further for a given electrolyte, the same equilibrium chain will be shifted to the left or right depending on the nature of the solvent, as we ought to write the above as:



again the "nature" of the solvent encompasses properties as permittivity, donor number, steric effects on solvation, etc. Because of the relatively high concentration (necessary to observe an ultrasonic relaxation process) and of the magnitude of the equilibrium constants (few free ions in solution), step one of scheme IV is generally unobservable in lithium ethereal solutions. The other steps have been at times reported in previous works^{1,6,7}.

In what follows we are going to interpret the reported data for $LiAsF_6$ and $LiBF_4$ according to scheme V. A comparison of these findings with $LiClO_4$ ⁷ and some generalization on the effect of the anion will be made. Scrutiny of Table I reveals that the relaxation frequency depends on concentration. It is in fact linearly dependent on c within experimental error. Further then $\ln \nu_m$ is proportional with the $\ln c$. Linear regressions applied to these data gives $r^2 = 0.996$, slope = 1.23. It has been indicated that for a dimerization process of the type:



One would expect the relaxation time τ to correlate with the forward and reverse rate constants by the linear relation

$$(\tau^{-1} = 2\pi f_r): \quad \tau^{-1} = 4k_f (\text{AB}) + k_r, \quad (\text{VII})$$

Whereas μ_m should correlate¹ with (AB) and $(\text{AB})_2$ according to:

$$\mu_m = \frac{\pi}{2\beta_s} \frac{(\Delta V_s)^2}{RT} \left(\frac{4}{(\text{AB})} + \frac{1}{(\text{AB})_2} \right)^{-1} \quad (\text{VIII})$$

Which, by the introduction of the quadrupole equilibrium constant $K_q = (\text{AB})_2/(\text{AB})^2$ reads:

$$\mu_m = \frac{\pi}{2\beta_s} \frac{(\Delta V_s)^2}{RT} \frac{K_q (\text{AB})^2}{1 + 4K_q (\text{AB})}, \quad (\text{IX})$$

Where $\beta = (\rho u)^{-1}$ is the adiabatic⁷ compressibility ρ the density, ΔV_s the adiabatic volume change due to the process VI.

In a medium of low permittivity such as 2MTHF ($\epsilon_0 = 6.2$) (if K_q is not large) one can set the total concentration equal to the concentration of the dominant specie, namely the ion pairs AB. Then from Eq. IX one would expect the slope of $\ln \mu_m$ vs $\ln c$ to be between 1 and 2 (depending on whether $1 \lesseqgtr 4K_q (\text{AB})$), as observed in the present case. Accepting the hypothesis that scheme VI describes the observed process for LiAsF_6 in 2MTHF, in a first approximation, setting $(\text{AB}) = C$ gives from linear regressions of τ^{-1} vs c : $r^2 = 0.96$, slope = 5.2×10^8 ,

int. = 1.7×10^8 , from which one calculates: $k_f = 1.3 \times 10^8 \text{ M}^{-1} \text{ s}^{-1}$,
 $k_r = 1.7 \times 10^8 \text{ s}^{-1}$ and $K_q = 0.7_6 \text{ M}^{-1}$.

Similarly by transforming Eq. IX one gets:

$$\frac{\pi(AB)^2}{2\beta_s \mu_m RT} = \frac{1}{(\Delta V_s)^2 K_q} + \frac{4}{(\Delta V_s)^2} (AB) \quad (X)$$

Setting again $AB = c$, linear regressions of the left part of equation X vs C gives: $r^2 = 0.96$, int. = $9.4_7 \times 10^{-6}$, slope = $4.7_9 \times 10^{-2}$, from which $\Delta V_s = 9.2 \text{ cm}^3/\text{mole}$ and $K_q = 1.3 \times 10^3 \text{ cm}^3/\text{mole}$ or $K_q = 1.3 \text{ M}^{-1}$, of the same order of magnitude of the figure above $K_q = 0.7_6 \text{ M}^{-1}$.

Figure 3a and b show the plots of τ^{-1} vs c and of the quantity $(\pi c^2 / 2\beta_s \mu_m RT)$ respectively. Figure 3c shows the calculated and experimental μ_m vs the concentration c.

It is interesting to compare the above results with data⁷ of LiClO_4 in 2MTHF. In this system, an ultrasonic relaxation process, independent of concentration (and assigned to the equilibrium outer sphere $\rightleftharpoons$ inner sphere quadrupoles) was observed. Hence, for LiClO_4 , the last step of scheme V is the only observable process around $c = 0.1\text{M}$. The ion-pair concentrations must be low enough for the first step of scheme V not to be visible.

It is difficult, from the isothermal data at hand, for LiAsF_6 in 2MTHF to decide whether the final product is solvated, contact quadrupole or a combination of both. What is observed

is that, at variance with LiClO_4 , a second order process is observable. The rate constant k_f is lower than what expected on a basis of a diffusion controlled rate constant between uncharged molecules in accord to Smoluchowsky⁸:

$$k_D = 8\pi RT/3000\eta, \quad (\text{XI})$$

with η the viscosity at 25°C for 2MTHF. That LiAsF_6 is probably more shifted toward the left in Scheme IV and V (in terms of the equilibrium concentrations) with respect to LiClO_4 can also be deduced from conductance results², showing the former electrolyte being much more conducting than the latter one in all the concentration range studied.

The case of LiBF_4 in 2MTHF is more complicated. Here the ultrasonic spectrum can be described as due to the sum of two close Debye relaxation processes. Inspection of Table II reveals that the relaxation at high frequency is concentration dependent, whereas the one at low frequency, within experimental error, is not concentration dependent.

We will consider the hypothesis that we are observing both steps of Scheme V.

Accordingly, as already shown⁶, one would expect two relaxation times bound by the relation:

$$\tau_{I,II}^{-1} = \frac{1}{2}(S \pm \sqrt{S^2 - 4P}), \quad (\text{XII})$$

where

$$S = \tau_I^{-1} + \tau_{II}^{-1} = 4k_2(\text{AB}) + k_{-2} + k_3 + k_{-3}$$

and

$$P = \tau_I^{-1} \tau_{II}^{-1} = 4k_2 (AB) (k_3 + k_{-3}) + k_{-2} k_{-3} \quad (XIII)$$

Notice in particular that if the condition $\tau_I^{-1} \gg \tau_{II}^{-1}$ were true then:

$$S = \tau_I^{-1} = 4k_2 (AB) + k_{-2}, \text{ and}$$

$$\tau_{II}^{-1} = \frac{P}{S} = k_{-3} + k_3 \frac{4k_2 (AB)}{4k_2 (AB) + k_{-2}}.$$

In the situation where $k_{-3} \gg k_3$, $\tau_{II}^{-1} = k_{-3}$ independent of c .

It is clear that because of the closeness of the τ 's, we cannot apply the condition $\tau_I^{-1} \gg \tau_{II}^{-1}$. However by using as first approximation $(AB) = c$ we can plot S vs. c and P vs. c .

Linear regressions applied to the quantity $S = (\tau_I^{-1} + \tau_{II}^{-1})$ vs. c gives $r^2 = 0.99$, slope $= 4k_2 = 2.63 \times 10^8$, intercepts $= k_{-2} + k_3 + k_{-3} = 1.50 \times 10^8$, hence $k_2 = 6.6 \times 10^7 \text{ M}^{-1} \text{ s}^{-1}$.

Further linear regressions of the quantity $P = (\tau_I^{-1} \tau_{II}^{-1})$ vs. c gives $r^2 = 0.95$, slope' $= 4k_2 (k_3 + k_{-3}) = 1.79 \times 10^{16}$, intercept' $= k_{-2} k_{-3} = 4.0 \times 10^{15}$.

From the above

$$\frac{\text{slope}'}{\text{slope}} = k_3 + k_{-3} = 6.8 \times 10^7, \text{ and}$$

$$k_{-2} = \text{Int} - \frac{\text{slope}'}{\text{slope}} = 1.50 \times 10^8 - 0.68 \times 10^8 = 8.2 \times 10^7 \text{ s}^{-1}$$

$$\text{also } k_{-3} = \frac{\text{Int}'}{k_{-2}} = \frac{4.0 \times 10^{15}}{8.2 \times 10^7} = 4.9 \times 10^7 \text{ s}^{-1}$$

$$\begin{aligned} \text{and } k_3 &= (k_3 + k_{-3}) - k_{-3} = 6.8 \times 10^7 - 4.9 \times 10^7 \\ &= 1.9 \times 10^7 \text{ s}^{-1} \end{aligned}$$

In summary the rate constants are:

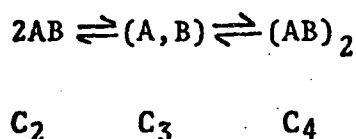
$$k_2 = 6.6 \times 10^7 \text{ M}^{-1} \text{ s}^{-1}$$

$$k_{-2} = 8.2 \times 10^7 \text{ s}^{-1}, \text{ hence } K_2 = \frac{k_2}{k_{-2}} = 0.8 \text{ M}^{-1}$$

$$k_3 = 1.9 \times 10^7 \text{ s}^{-1}$$

$$k_{-3} = 4.9 \times 10^7 \text{ s}^{-1}, \text{ hence } K_3 = \frac{k_3}{k_{-3}} = 0.4$$

The overall quadrupole formation constant K_q is related to Scheme V.



by the relation

$$K_q = \frac{C_3 + C_4}{C_2^2} = \frac{(K_3 K_2 + K_2) C^2}{C_2^2} = K_2 (1 + K_3)$$

$$\text{Where } K_2 = \frac{C_3}{C_2^2} \text{ and } K_3 = \frac{C_4}{C_3}$$

$$\text{Therefore } K_q = 1.1 \text{ M}^{-1}.$$

Figure 4a and 4b reports the plots of S vs c and of P vs c respectively.

CONCLUSION

It appears from the above that the extent of quadrupole formation for the three electrolytes LiAsF_6 , LiBF_4 and LiClO_4 in 2MTHF follows the order: $\text{LiClO}_4 > \text{LiBF}_4 \gtrsim \text{LiAsF}_6$, probably paralleling the corresponding ion-pair association (as conductance data suggest when one compares results for LiClO_4 and for LiAsF_6).

Analysis of the ultrasonic relaxation data reveal that LiAsF_6 forms quadrupoles, LiBF_4 is an intermediate case with the concentration of the contact quadrupoles being larger than the solvated species. LiClO_4 is probably an extreme case where the salt is heavily associated to quadrupoles, the free ion-pairs no longer contributing to the ultrasonic relaxation process.

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TABLE I

Li As F6 in 2 MTHF; t = 25°C.

c	fr	$A \times 10^{17}$	$B \times 10^{17}$	$u \times 10^{-5}$	$\mu_m \times 10^5$
(M)	(MHz)	(cm^{-1}s^2)	(cm^{-1}s^2)	(cm s^{-1})	-
0.60	78	170	70	1.206	800
0.516	67	157	64	1.197	630
0.41	63	126	70	1.199	476
0.36	59	110	73	1.254	407
0.25 ₇	47	96.4	66	1.226	278

TABLE II

Li BF₄ in 2MTHF; t = 25°C

c	$\mu_{mI} \times 10^5$	fr _I	$\mu_{mII} \times 10^5$	fr _{II}	$B \times 10^{17}$	$u \times 10^{-5}$
(M)	-	(MHz)	-	(MHz)	(cm^{-1}s^2)	cm s^{-1}
0.15 ₅	185	22	185	8	50	1.209
0.25	250	27	250	8	55	1.216
0.34	300	30	300	8	61	1.210
0.41	400	32	370	9.5	55	1.215
0.47	450	34	440	9	60	1.214

List of figures

Fig 1

α/f^2 vs frequency f for LiAsF_6 0.36M in 2MTHF $t=25^\circ\text{C}$

Fig 2

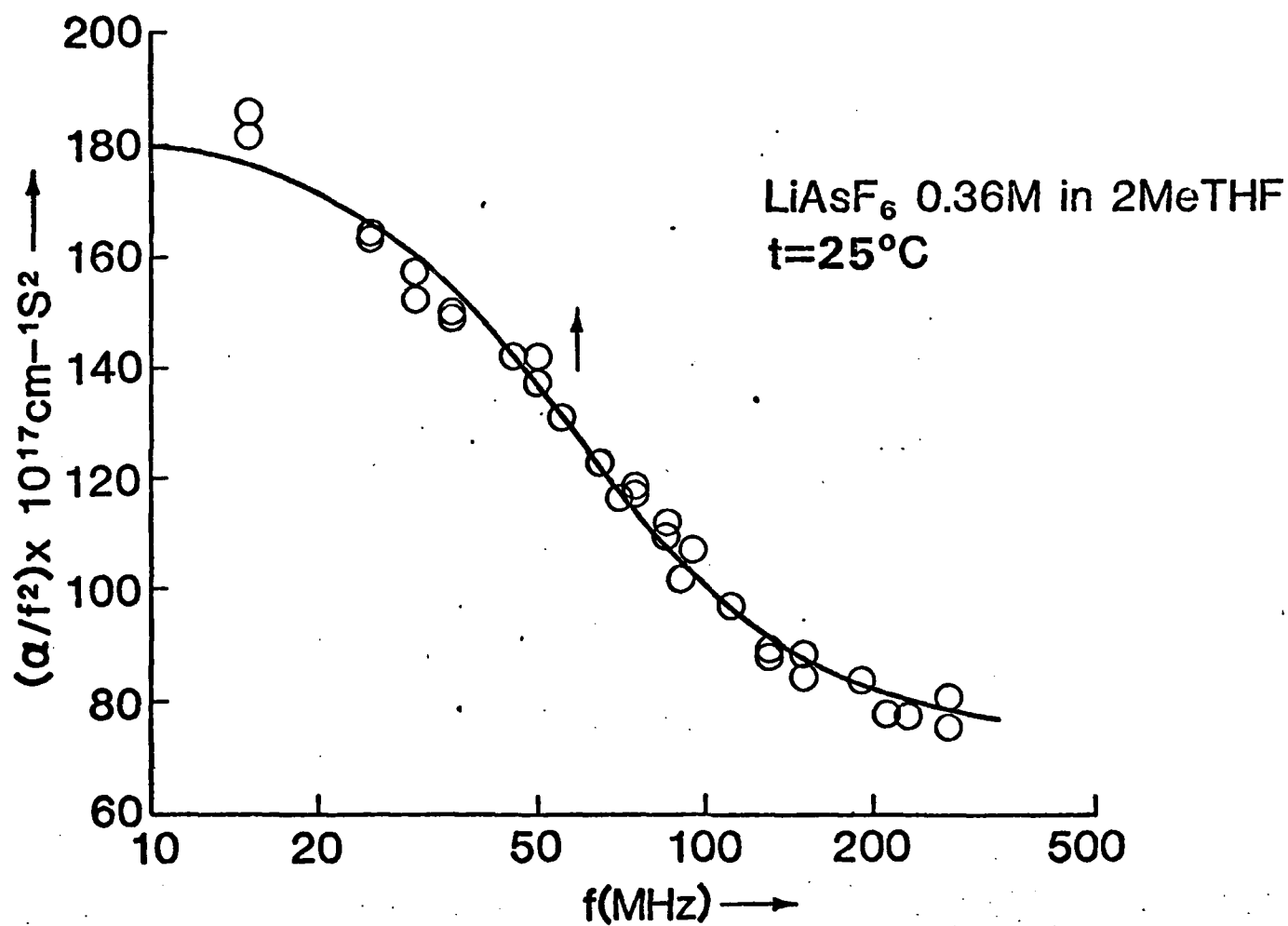
μ vs frequency f for LiBF_4 0.34M in 2MTHF $t=25^\circ\text{C}$

Fig 3

- a) τ^{-1} vs c for LiAsF_6 in 2MTHF; $t=25^\circ\text{C}$
- b) $(\pi c^2 / 2 \mu_m \beta_s \cdot RT)$ vs c for LiAsF_6 in 2MTHF; $t=25^\circ\text{C}$
- c) μ_m vs c for LiAsF_6 in 2MTHF $t=25^\circ\text{C}$
The solid line is the calculated function

Fig 4

- a) $S = (\tau_I^{-1} + \tau_{II}^{-1})$ vs c for LiBF_4 in 2MTHF; $t=25^\circ\text{C}$
- b) $P = (\tau_I^{-1} \times \tau_{II}^{-1})$ vs c for LiBF_4 in 2MTHF; $t=25^\circ\text{C}$



LiBF_4 0.34M in 2MeTHF
 $t=25^\circ\text{C}$

