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A new type of sample tube for reducing convection effects in PGSE-NMR measurements of self-diffusion coefficients of liquid samples

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Abstract

Pulsed gradient spin-echo (PGSE) NMR measurements of the self-diffusion coefficients of low viscosity liquids are greatly hampered by the effects of convection especially away from ambient temperature. Here we report on a new NMR tube designed to minimize the deleterious effects of convection. In this tube, which derives from a Shigemi symmetrical NMR tube, the sample is contained in an annulus formed from a concentric cylinder of susceptibility matched glass. The performance of this tube was demonstrated by conducting measurements on the electrochemically important $LiN(SO_3CF_3)_2$ (LiTFSI)–diglyme (DG) system. Calibrations were first made using DG at column heights of 2, 3, and 4-mm in the temperature range between −40 and 100 °C. Measurements of the diffusion coefficients of the lithium, anion, and DG were then performed to probe the solvent–ion and ion–ion interactions in the DG doped with LiTFSI. Changes in the ^1H, ^7Li, and ^{19}F PGSE-NMR attenuation curves at −40 °C provided clear evidence of interactions between the DG and lithium ion.

Keywords: Convection; Diffusion; Ion–ion and ion–solvent interactions; Low viscosity liquid; NMR tube; PGSE-NMR

1. Introduction

The translational (or self-) diffusion coefficient (*D*) of a liquid is one of the most important physical parameters for probing solution interactions, and thus additional information is gained by measuring *D* over a temperature range. In theory, the pulsed gradient spin-echo (PGSE) NMR method can easily be applied to measure variable temperature diffusion data simply by changing the temperature of the gas surrounding the sample tube. In practice, however, the sample heating (or cooling) is never completely homogeneous in an NMR probe and the resulting temperature differences (generally top-to-bottom) give rise to temperature gradients which induce convection in liquid samples. The onset of convection depends on the Rayleigh number [1–3].

Since convection is a form of translational motion, it can complicate diffusion measurements and, if unaccounted for, leads to a measured self-diffusion coefficient larger than the intrinsic value [4–8]. Convection currents, which have a range of velocities, typically occur along the long axis of the NMR tube [9–12], and thus do not produce a phase change since the flow of the spins along the direction of the gradient is exactly matched by the flow in the anti-parallel direction. PGSE experiments are typically performed by applying two equal gradient pulses of magnitude g and duration δ separated by a time interval Δ. For a single diffusing species, convection introduces a cosine modulation into the PGSE spin-echo attenuation, viz. [7,13]

$$E(g,\Delta) \sim \cos(\gamma g \delta v \Delta)\exp(-\gamma^2 g^2 \delta^2 D(\Delta - \delta/3)), \qquad (1)$$

where v is the velocity of the flow. Insidiously, however, when $v^2\Delta \ll D$ and $\delta \ll \Delta$, the presence of convection is

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generally not apparent due to the similarity between the cosine and Gaussian functions [7]. Consequently, convection-tainted PGSE data generally appears well described by the usual exponential function with an apparent diffusion coefficient of [7]

$$D_{\mathrm{app}} = D + \frac{\Delta v^2}{2}. \tag{2}$$

Thus, while D is time-independent (i.e., Δ-independent) for Fickian diffusion of a homogeneous solution of a single species, the signature of the presence of convection is the measured D increased with increasing Δ. Importantly, whereas the diffusion coefficient depends on molecular size, the flow velocity is common to all of the species in the sample.

Ideally no temperature gradients should exist within the sample, but in practice this is nearly impossible to achieve away from ambient temperature—this problem is quite apart from the probe having very stable temperature control. Numerous means of preventing convection effects have been tried including sophisticated PGSE sequences incorporating bipolar gradient pulses [14–16] and spinning the sample during the PGSE sequence [3,6,17]. Nevertheless, although reducing the effects, these 'solutions' do not prevent convection within the sample tube and introduce additional complications (e.g., more complicated pulse sequences).

Another alternative for reducing convection effects is to use short samples so as to reduce the temperature gradient. And previously we tried small sample volumes with the height set to 2 mm to prevent convection effects [18]. While such short samples were effective in reducing the convection effects for viscous liquids, measurements with small molecules, like diglyme (DG), gave unsatisfactory results. In the present work, special NMR sample tubes were designed based on a Shigemi symmetrical susceptibility-matched microtube. The conventional sample tube set is composed of an outer tube of 5 mm (4.965 mm) o.d. and a concentric cylindrical insert. In the present study, special inserts were used with a column of 2 mm diameter in the centre with heights of 2, 3, or 4 mm as shown in Fig. 1. Hence, the sample can be limited to heights of 2, 3, or 4 mm. The annular geometry of the sample results in a greatly reduced likelihood of convection while retaining high signal-to-noise and reasonable homogeneity. Since the modified tube has the same basic characteristics as a standard Shigemi tube, i.e., (reasonably) susceptibility matched and extends both above and below the sample volume, the tube is much easier to shim than the same sample volume in a standard NMR tube. The reproducible sample height leads to more consistent shimming between subsequent samples. The small sample volume also helps in reducing radiation damping artifacts [19].

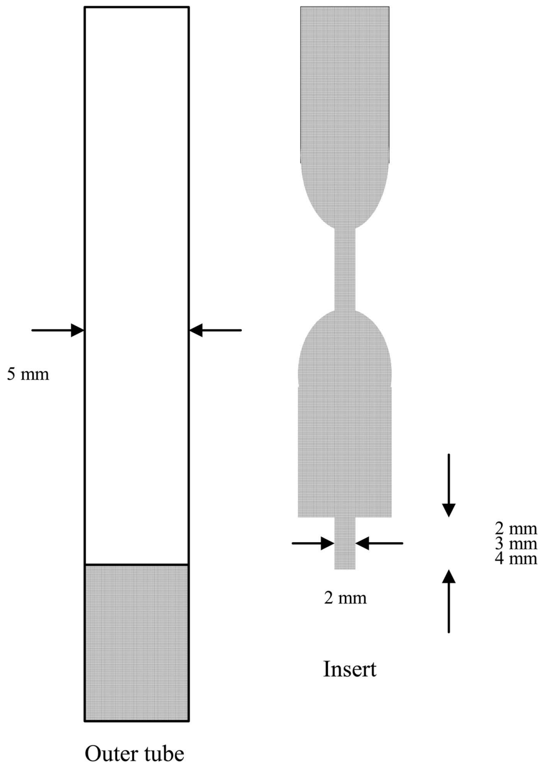

Fig. 1. Modified NMR sample tube giving an annular sample volume supplied by Shigemi, Tokyo.

The suitability and performance of this new tube was first verified by performing variable-temperature diffusion measurements on the solvent, diethylene glycol dimethyl ether (diglyme, DG, $CH_3O(CH_2CH_2O)_2CH_3$, viscosity 1.0 cP at 25 °C, freezing point −64 °C and b.p. 162 °C, as a comparison the viscosity of H_2O at 25 °C is 0.8902 cP), with excellent results being obtained for DG using 2 and 3 mm sample heights in the temperature range −40 and 100 °C.

Subsequently, the temperature dependence of the diffusion of the individual species (i.e., solvent, anion, and cation) in an electrolyte formed by doping DG with the lithium salt, $LiN(SO_2CF_3)_2$ (LiTFSI) were measured using ^1H, ^7Li, and ^{19}F PGSE-NMR, respectively. This electrolyte is commonly used as a model system in battery/ionic conductivity studies. Diglyme is the shortest oligomer of polyethylene oxide (PEO) that contains the 3 oxygen structural unit involved in coordinating lithium ions. Thus, clarifying the interaction between DG and lithium ions is of central importance in understanding the coordination of Li by $(CH_2CH_2O)_n$ chains for the development of lithium ion polymer electrolytes based on PEO. Recent solid state studies have elucidated the ion coordination structure [20,21] and the solution structure from molecular dynamics simulation [22]. In the present study, the individual self-diffusion coefficients in the liquid state afford insight into the interactions between the DG and lithium ion and ion pairing over a wide temperature range.

2. Experimental

All diffusion measurements were performed on a JEOL GSH-200 console equipped with a wide-bore 4.7 T magnet, multi/^{1}H, and ^{19}F/^{1}H JEOL diffusion probes, a JEOL pulse gradient amplifier, a TecMag RF unit, and NTNMR. The maximum gradient strength is about 10 Tm^{-1} and bipolar gradients are not possible. The temperature of the sample was controlled by a JEOL variable temperature unit. The temperature was changed in 10 °C increments in the range 100 °C to −40 °C and measurements were made both with increasing and decreasing temperature by using the modified Hahn spin-echo sequence by varying δ with Δ being set at 30, 50, 100 or 200 ms.

The sample tube used was a modified Shigemi symmetrical microtube based on a BMS-005J with a small central column at the bottom of the insert creating an annular sample volume as shown in Fig. 1. With a column height of 2 mm and the absence of sampling spinning, the full line width at half height of the H_2O signal was 4 Hz due to the close matching of the magnetic susceptibility of the glass and H_2O. These sample tubes afford reproducible sample volumes and easy sample recovery. ^{1}H PGSE-NMR calibration experiments were performed with neat DG using four sample tubes with central columns having heights 2, 3, and 4 mm inside and, for comparison, a standard Shigemi BMS-005J with a sample height of 5 mm. The multinuclear PGSE measurements of the temperature dependence of the individual diffusion coefficients in the DG-TFSI electrolyte (O:Li = 20:1 for the oxygen of DG and lithium salt) were measured using the 3 mm column sample tube between −40 and 80 °C.

3. Results

3.1. Calibration of D using DG

The temperature dependences of apparent self-diffusion coefficients of neat DG are shown in Fig. 2 for the various sample tubes. The apparent self-diffusion coefficients obtained using the 5 mm sample for Δ set to 30, 50, 100, and 200 ms (the data for 100 and 200 ms are not shown) agree in the lower temperature range and gradually deviated at higher temperatures. At 70 °C, they were 3.2, 3.9, 4.7, and 5.6×10^{-9} m^{2}s^{-1} for $\Delta = 30$, 50, 100, and 200 ms, respectively. The corresponding value obtained with the 2-mm sample tube was 2.0×10^{-9} m^{2}s^{-1}. Clearly the effects of convection on the measured self-diffusion coefficients were more apparent at longer Δ in the 5-mm sample tube. Using the 4-mm sample tube, the convection effect was greatly reduced as shown in Fig. 2, but at temperatures above 80 °C, the apparent D still deviated to larger values and the Δ-dependence of D was observed to a smaller extent. The $\Delta = 30$ and 50 ms data from the 3-mm sample tube showed good agreement. Unfortunately, the reasonably short T_2 values precluded sufficiently accurate diffusion measurements at longer Δ. In order to confirm further, measurements were made using the 2-mm sample tube between 100 and −40 °C. The results are shown in Fig. 2, in which the $\Delta = 30$ and 50 ms data are coincident over the entire temperature range (see inset). The data obtained with the 3-mm sample tube agreed well with the data obtained using the 2-mm sample tube over the whole temperature range. In the present experiments reasonable agreement was obtained below 20 °C even using the 5-mm sample tube. The convection effect in DG at lower temperatures is less significant due to the increased viscosity. Thus, the results show that the self-diffusion coefficients of DG can be measured accurately over a wide temperature range by using the 2-mm sample tube. Similarly, the 3 mm sample tube can be used for liquid samples with viscosities greater or equal to DG. The Arrhenius activation energies for DG were 13.3 ± 0.1 and 18.6 ± 0.2 kJmol^{-1} for the higher (above 0 °C) and lower temperature ranges, respectively. The previously determined value for diffusion data between 30 and 80 °C was a little smaller (11.3 ± 0.3 kJmol^{-1}) [18].

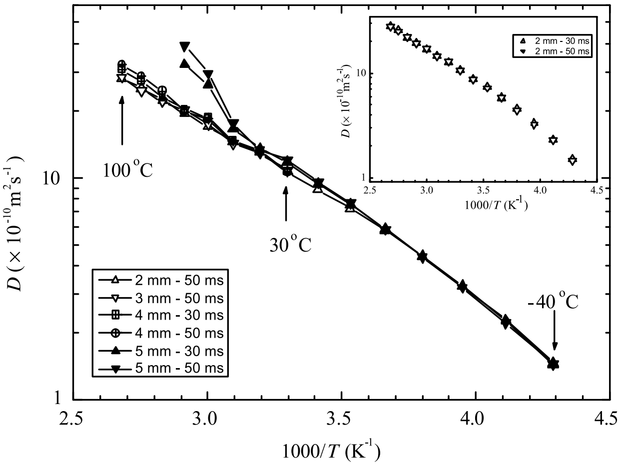

Fig. 2. The temperature dependence of the self-diffusion coefficients of DG measured using the modified sample tubes (see Fig. 1) with sample heights of 2, 3, and 4 mm and a standard Shigemi tube with a cylindrical sample volume of height 5 mm. The plots in the inset were performed using a 2 mm sample tube with $\Delta = 30$ and 50 ms.

3.2. DG electrolyte

The temperature dependencies of the diffusion of the DG, Li$^+$, and TFSI measured using $\Delta = 50$ ms and the 3-mm sample tube are shown in Fig. 3. Whilst the diffusion of the lithium ion and anion were measured using the multinuclear and F/H probes, respectively, the diffusion of the solvent DG was measured using the ^{1}H

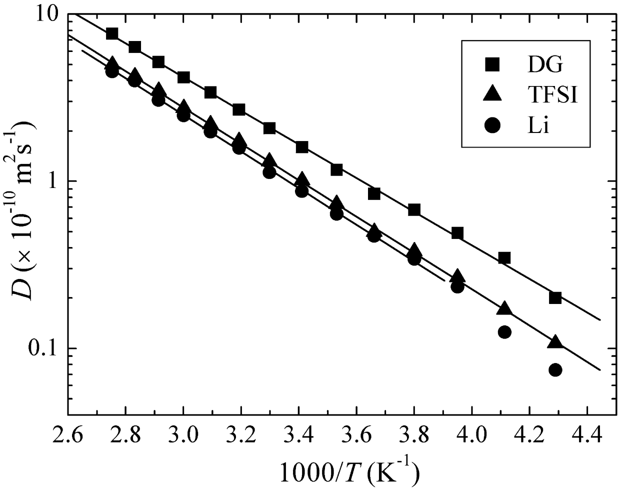

Fig. 3. The temperature dependence of the self-diffusion coefficients of the DG, lithium ion, and anion (TFSI) for the liquid electrolyte measured using the 3 mm sample tube with $\Delta = 50$ ms between −40 and 80 °C.

channel of both probes and the results were in complete agreement—verifying both the gradient strength and temperature calibrations of the probes. The linearized PGSE attenuation plots (i.e., the natural logarithm of the signal attenuation, E, versus $\gamma^2\delta^2g^2(\Delta - \delta/3)$) were linear for all the species in the temperature range between 80 and −30 °C. The determined D values are shown in Fig. 3 including estimates of the diffusion coefficients at −40 °C, which were obtained from the initial linear part of the attenuation plots (see Fig. 4). At all temperatures, the order of diffusion is DG>TFSI>Li with activation energies of 19.3 ± 0.2, 20.8 ± 0.1, and 21.0 ± 0.2 kJmol^{-1} (excluding the −40 °C Li data). The similarity of the activation energies the self-diffusion coefficients over the wide temperature range (i.e., parallel plots) suggests that the data measured using the special sample tube are very close to the intrinsic values at every temperature.

At −40 °C the linearized ^{7}Li PGSE attenuation plot (see Fig. 4) exhibited negative curvature suggestive of restricted diffusion similar to that observed in the related higher molecular weight PEO-based system [23,24]. Whereas the ^{1}H and ^{19}F PGSE attenuation plots exhibited positive curvature—possibly indicating multicomponent diffusion. Double exponential fitting of the data indicated that about 10% of the DG and TFSI data was constituted by a slowly diffusing component. The mole ratio of DG : Li is ~ 6.7:1. If one DG interacts with one Li, about 15% of all the DG molecules in the electrolyte will interact with the lithium ions at any one time.

The Stokes–Einstein equation, when applicable, implies that the diffusion coefficient of a species is inversely proportional to its hydrodynamic size. In the present system, since the diffusion coefficients of the ions are measured in the same solution, the bulk viscosity should affect the diffusing ions similarly. Thus, starting from the Stokes–Einstein equations for the solvent and the ion,

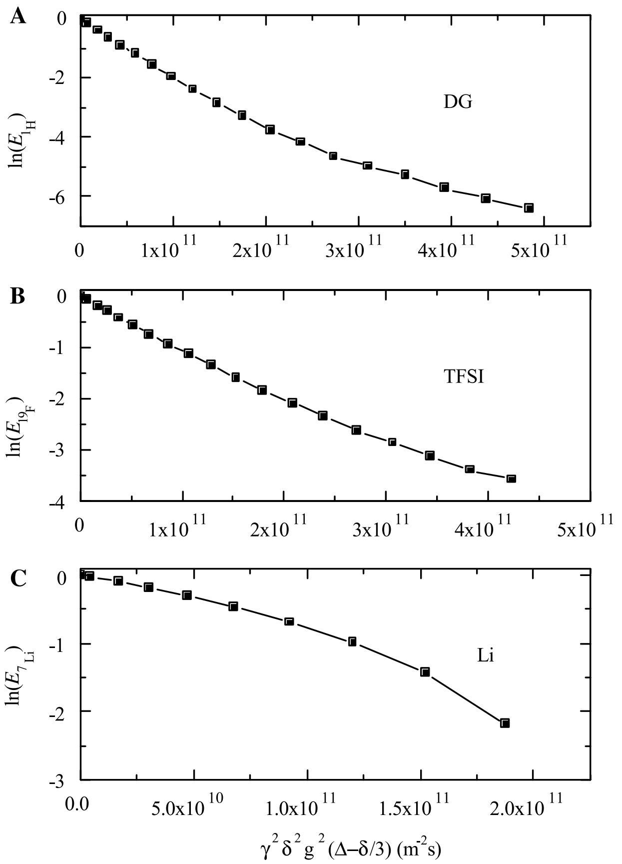

Fig. 4. The linearized PGSE attenuation profiles of (A) DG, (B) TFSI, and (C) Li ion measured at −40 °C.

$$D_{\mathrm{solv}} = \frac{kT}{c\pi\eta r_{\mathrm{s}}^{\mathrm{solv}}} \quad \text{and} \quad D_{\mathrm{ion}} = \frac{kT}{c\pi\eta r_{\mathrm{s}}^{\mathrm{ion}}}, \tag{3}$$

where c is a constant, η is viscosity, and $r_{\mathrm{s}}^{\mathrm{ion}}$ and $r_{\mathrm{s}}^{\mathrm{solv}}$ are the Stokes radii for the ions and solvent, respectively, and combining these equations and rearranging gives,

$$R = \frac{D_{\mathrm{solv}}}{D_{\mathrm{ion}}} = \frac{r_{\mathrm{s}}^{\mathrm{ion}}}{r_{\mathrm{s}}^{\mathrm{solv}}}. \tag{4}$$

R is a semi-quantitative measure of the degree of ion solvation. The temperature dependence of R_{Li} and R_{TFSI} are plotted in Fig. 5. The van der Waals radii, which are not necessarily the same as the Stokes radii, of Li and TFSI are 0.076 and 0.325 nm, respectively [25]. Since the molecular size of TFSI (Mw = 282) is larger radius than DG (Mw = 134), the R_{TFSI} values of 1.6 at temperatures above 0 °C are reasonable. Since the size of the lithium ion is much smaller than DG, the larger $R_{\mathrm{Li}} = 1.7$ above 0 °C suggests solvation of the lithium ion. Below 0 °C, R_{ion} increases, especially in the case of R_{Li}.

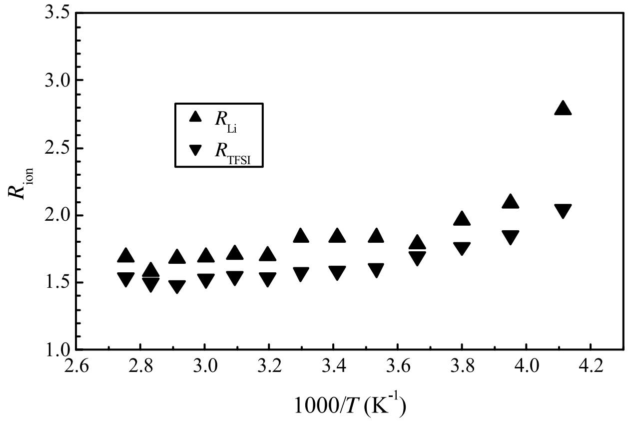

Fig. 5. The change in the R parameters (i.e., $R_{ion} = D_{DG}/D_{ion}$) of the TFSI and the lithium ion with temperature.

4. Discussion

The effect of convection on NMR diffusion measurements in low viscosity liquids away from ambient temperature is a well-known problem. The present results show that the use of the specially designed NMR tube with an annular sample volume with a height of 3 mm greatly reduces the effects of convection in liquids of low viscosity like DG. With less viscous liquids a 2 mm sample height is preferred. Although the effects of convection were not significant for DG at lower temperatures, they can be significant when measuring the diffusion of other liquids, nevertheless the use of these new sample tubes will greatly reduce artifactual attenuation in PGSE-NMR experiments.

As noted above, clarifying the interactions between DG and the lithium ion are important for electrolyte development, and to this end we have previously reported the interaction between lithium ion and the $(CH_2CH_2O)_n$ moiety in a systematic way for $n = 3$–50. However, the data from the DG electrolytes was previously excluded out of the uncertainty of the experimental data. The present diffusion data are consistent with the data for trigylme (TG), tetraglyme (TeG), and pentaglyme (PG) previously reported above 30 °C. The measuring temperature range was wider in the present study, especially with regard to lower temperatures. The linearized PGSE attenuation plots were all linear except for the measurements at −40 °C. The non-linearity of the −40 °C plots (see Fig. 4) indicates non-Fickian diffusive processes suggestive of structural formation. However, although it is known that lithium ions interact with DG [18], it is difficult to envisage how such small (length) scale structural formation could lead to the PGSE measurement of the Li ion exhibiting restricted diffusion effects. The positive curvature of the linearized ^{1}H PGSE attenuation plot suggests that the DG molecules are present as multiple components and the analysis of the data showed that about 10% of the DG diffuse more slowly, which is attributed to the interaction with the lithium ion. The similarity of the ^{19}F (i.e., TFSI) attenuation plot to that of DG, indicates that the anion diffusion is closely linked to that of DG.

The temperature dependences of the experimental parameters R_{ion} in Fig. 5 show that R_{Li} is always larger than R_{TFSI} due to solvation around the lithium ion and both values increase gradually below 0 °C, which suggests increasing ion association with decreasing temperature. By comparing ionic conductivity with ion diffusion coefficients at 30 °C, the degree of ion dissociation was previously determined to be 0.38 at a similar salt concentration [26]. We had also reported that R_{ion} becomes larger as the association of the anion and lithium is stronger [27]. The experimental finding that R_{Li} and R_{TFSI} are temperature insensitive above 10 °C suggests that the interaction of Li-DG and Li-TFSI are similar, but the gradual increase of R_{TFSI} and large increase of R_{Li} at lower temperatures are consistent with the stronger solvation of the Li ion at −40 °C. The solvation model for lithium by DG is that at higher temperatures a lithium ion diffuses together with one and a half DG molecules on average, and the number of DG molecules solvating the lithium increases as the temperature decreases.

5. Conclusions

Convection is all but inevitable in low viscosity liquid samples in NMR probes with normal gas flow temperature control. To minimize the deleterious effects of convection in PGSE-NMR diffusion measurement we have introduced a novel NMR sample tube with a short annular sample volume and verified its efficacy using DG in the temperature range −40–100 °C. By using this special sample tube, reliable self-diffusion coefficients of the ions and solvent were measured for a liquid DG-LiTFSI electrolyte system over a wide temperature. DG-lithium ion interactions were clearly observed at lower temperatures. The new NMR sample tubes afford reproducible control of the sample volume and easy sample recovery. The small sample volume also helps to prevent radiation damping artifacts.

Acknowledgments

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