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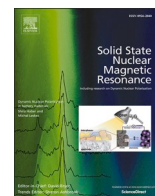
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Ultra-wideline dipolar NMR spectroscopy: Combining SEDOR and WURST-CPMG

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ABSTRACT

A method to obtain magnetic dipole-dipole coupling information for nuclei exhibiting ultra-wideline NMR spectra in disordered solids is presented. This is achieved via a constant time version of SEDOR (Spin Echo **DO**uble Resonance) employing WURST (Wideband Uniform Rate Smooth Truncation) pulses and CPMG (Carr Purcell Meiboom Gill) detection. The method – coined CT-WUDOR-CPMG (Constant Time-WURST SEDOR-CPMG) NMR spectroscopy – is tested on $\text{Ba}_2\text{TeO}(\text{PO}_4)_2$ as a crystalline model compound applying $^{125}\text{Te}\{^{31}\text{P}\}$ -CT-WUDOR-CPMG NMR. Then, $^{125}\text{Te}\{^{19}\text{F}\}$ - and $^{125}\text{Te}\{^{31}\text{P}\}$ -CT-WUDOR-CPMG are performed to obtain structural information on TeO_2 – NaPO_3 – NaF glasses.

1. Introduction

The measurement of the heteronuclear dipolar coupling between two nuclear spin systems I and S has long been established as an indispensable tool for the structural characterization of solids. In the SEDOR experiment, introduced by Kaplan and Hahn [1] as early as 1958, the heteronuclear I-S dipolar interaction, usually refocused by a π pulse in the $\pi/2 - \tau - \pi - \tau$ Hahn spin echo sequence, is recoupled by an additional π pulse applied to the non-observed spins I. Experimentally, the normalized difference between the spin echo amplitudes without (S_0) and with recoupling (S) is measured. From the evolution of $\Delta S/S_0 = (S_0 - S)/S_0$ as a function of the dipolar mixing time 2τ , the strength of the heteronuclear dipolar coupling may then be evaluated [2].

This concept may also be combined with the high-resolution conditions of a Magic Angle Spinning (MAS) NMR experiment, adding site selectivity to the approach. Among the numerous strategies developed so far, the archetypical REDOR method (Rotational Echo **DO**uble Resonance), introduced 1989 by Schaefer and Gullion [3], has evolved as the workhorse for the quantification of the heteronuclear dipolar

coupling strength. Here, the dipolar recoupling may be achieved via rotor synchronized π pulses on the non-observed (I) nucleus. As with SEDOR, from an analysis of the REDOR evolution curves, the magnitude of the dipolar coupling constant (in units of Hz) $D = \frac{\gamma_I \gamma_S \hbar \mu_0}{8\pi^2 r_{IS}^3}$ may be obtained from which internuclear distances may be deduced. Both approaches, SEDOR and REDOR (including various variants, e. g. REAPDOR [4] (Rotational Echo Adiabatic Passage **DO**uble Resonance) and TRAPDOR [5] (TRansfer of Populations in **DO**uble Resonance)) have contributed enormously to a better understanding of structural details on intermediate length scales in quite a variety of materials [6–17].

Despite recent progress in MAS NMR probe design, there are still cases in which the application of fast sample spinning does not produce a line narrowing effect, especially in the case of a large chemical shift anisotropy (CSA) in combination with a broad distribution of the isotropic chemical shift values as for example observed for ^{125}Te in oxide glasses. In these cases, CSA information may be obtained from specialized pulse sequences such as WURST-CPMG [18–22]. Here the WURST method provides a uniform excitation over a wide frequency range,

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Table 1

Molar composition (mole%) and F/Te and P/Te ratios of the glass samples studied in this work.

Sample	x	TeO ₂	NaPO ₃	NaF	F/Te -ratio	P/Te ratio
T6NN0	0	60	40	0	0	0.67
T6NN1	0.1	60	36	4	0.07	0.6
T6NN3	0.3	60	28	12	0.2	0.47
T6NN5	0.5	60	20	20	0.33	0.33

while the CPMG multiple echo acquisition mode can produce a considerably improved S/N ratio. Fourier transformation of the entire echo train, followed by magnitude calculation, then produces a spikelet pattern representative of the static powder lineshape.

While a number of different approaches are known for measuring heteronuclear interaction strengths with non-observed nuclei whose lineshapes are broadened by strong quadrupolar or CSA interactions [17,23–26], to the best of our knowledge, no dipolar recoupling experiments have been reported in which the observed nuclei have been detected by wideband excitation schemes.

To this end we have combined WURST-CPMG with SEDOR (Constant Time-WURST SEDOR-CPMG, CT-WUDOR-CPMG) NMR. The approach was validated using ¹²⁵Te{³¹P}-CT-WUDOR-CPMG experiments on the ¹²⁵Te-³¹P multi-spin system of the crystalline model compound Ba₂TeO(PO₄)₂. It was subsequently applied to obtain information about the structural organization in TeO₂ – NaPO₃ – NaF glasses employing ¹²⁵Te {¹⁹F}- and ¹²⁵Te{³¹P}-CT-WUDOR-CPMG NMR spectroscopy.

2. Experimental

Synthesis: Ba₂TeO(PO₄)₂ was synthesized according to the procedure described in Ref. [27]. The TeO₂ – NaPO₃ – NaF glass samples of series T6NNX studied here with ¹²⁵Te{¹⁹F}- and ¹²⁵Te{³¹P}-CT-WUDOR-CPMG NMR are the same as those reported in Ref. [28]. The compositions of the samples follow formula 0.6TeO₂ – 0.4[(1-x) NaPO₃-xNaF] and are listed in Table 1 together with the corresponding F/Te and P/Te ratios.

Solid-State NMR: With the exception of a ¹⁹F-MAS spin echo NMR spectrum of glass sample T6NN5, acquired with a spinning frequency of 30 kHz employing a Varian VNMRs 500 MHz spectrometer operating at 11.7 T and using a 1.6 mm T3 MAS NMR probe, all experiments were conducted at room temperature and under ambient atmosphere

employing a Bruker AVANCE III spectrometer operating at 7.05 T and using Bruker 4 mm double and triple resonance probes. All ultra-wideline experiments applying WURST pulses were performed under static conditions.

Static solid state ¹²⁵Te NMR spectra ($\nu_0 = 94.9$ MHz) were recorded using the WURST-CPMG pulse sequence (cf. Fig. 1b). WURST-80 pulses were used, with pulse lengths of 50 μ s and an excitation bandwidth of 500 kHz corresponding to a sweep rate of 10 kHz μ s⁻¹ [18–22]. Chemical shifts are reported relative to Te(CH₃)₂ at 0 ppm, referenced with Te(OH)₆ solution (1 M) as a secondary reference at 713 ppm [29].

Further details of the experimental parameters are given in the figure captions.

Pulse sequences for dipolar recoupling: The SEDOR-experiment is a straightforward expansion of the spin-echo sequence for measuring heteronuclear dipole-dipole interactions in static solids. In its original form only the echo-time was varied while the pulse lengths were kept constant. In the constant time (CT) version, CT-SEDOR (cf. Fig. 1a) [30], the echo-time τ is kept constant, while the magnitude of the dipolar dephasing is controlled by the length of the recoupling pulse, producing flip angles that are varied over the full range of 0 to 2π . Principally, this effect is governed by the probability of a spin flip which is given by $1 - \cos^2(\theta/2)$ [30]. However, this may be biased if the CSA of the dephasing nucleus exceeds the excitation bandwidth of the dephasing pulse (this effect will then increase with increasing pulse length from left to right in the WUDOR recoupling curves). We therefore analyzed the recoupling curves using the SIMPSON software package [31,32]. In CT-WUDOR-CPMG, the lineshapes of the observed nuclei are recorded by WURST-shaped pulses (cf. Fig. 1b) to provide broad uniform excitation and refocusing. To boost the S/N ratio, the signal detection is accomplished via a WURST-CPMG acquisition train, which, upon Fourier transformation and subsequent magnitude calculation, results in a spikelet pattern representing the static NMR lineshape. The constant time version was chosen since it bears the advantage of only needing one reference experiment to record the S_0 value for a series of S values. The pulse sequence for the observe channel resembles that described in Ref. [18]. Experimental details may be found in the corresponding figure captions. Pseudo-2D versions of the pulse sequence for Bruker Avance III and Avance Neo consoles are given in SI1 and SI2, respectively.

NMR lineshape analysis was performed using the DMFIT [33], ssNake [34] and SIMPSON [31,32] software. For the evaluation of the S and S_0 spikelet patterns obtained from the WUDOR-CPMG experiments,

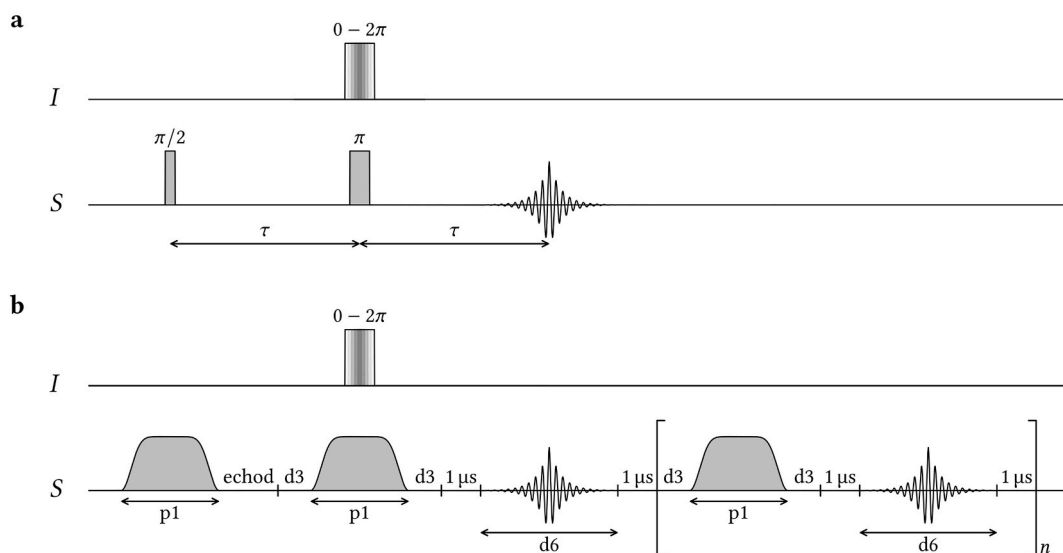


Fig. 1. Pulse sequences for a) (CT)-SEDOR NMR and b) (CT)-WUDOR-CPMG NMR. In the CT versions, the flip angle of the recoupling pulse varies between 0 and 2π . The WURST-CPMG sequence resembles that of b), without the recoupling pulse. The separation between the first two WURST pulses is given by echod + d3. The parameter echod is given by $(d6 + 2\mu s - p1)/2$.

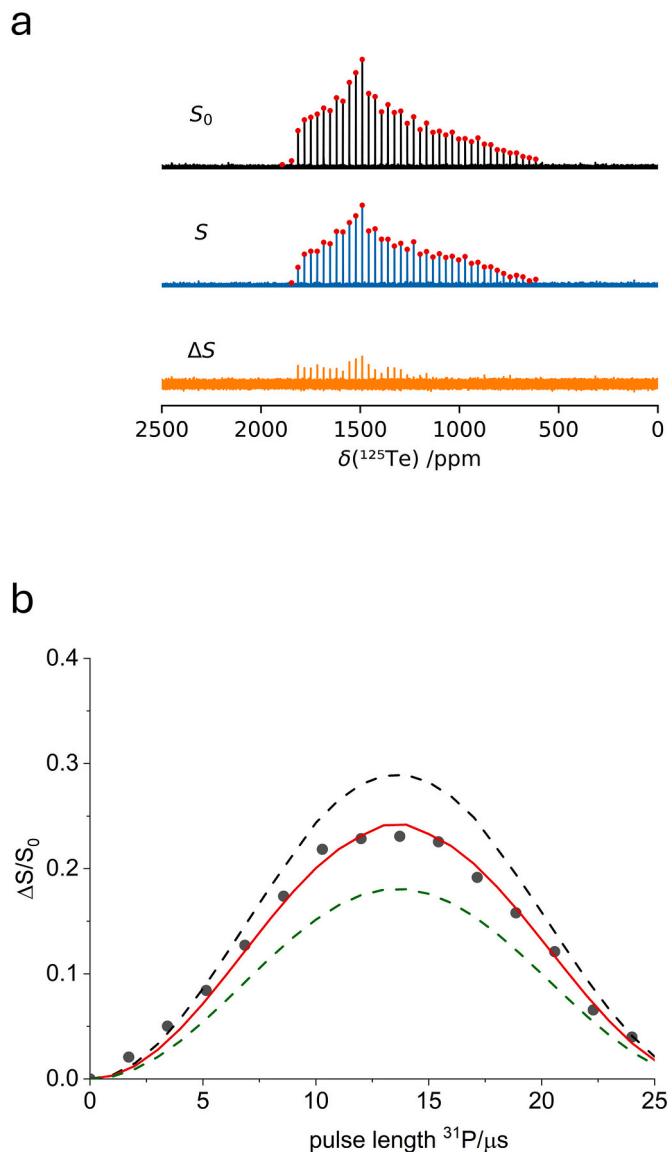


Fig. 2. a) $^{125}\text{Te}\{^{31}\text{P}\}$ -CT-WUDOR-CPMG NMR spectra for $\text{Ba}_2\text{TeO}(\text{PO}_4)_2$. Spectra marked S_0 (top) and S (middle) are without and with recoupling and the spectrum marked ΔS (bottom) is the difference spectrum. The ^{31}P pulse length is 12 μs . The red dots mark the intensities obtained employing a Python routine. b) Corresponding recoupling data (black filled circles) and SIMPSON simulations for $D = 700$ Hz (green dashed curve), $D = 814$ Hz (red curve) and $D = 900$ Hz (black dashed curve). Experimental details: recycle delay: 100 s using a saturation comb; $d_3 = 9$ μs ; $d_6 = 256$ μs ; $\text{echod} = 104$ μs ; $\nu_{\text{RF}}(^{125}\text{Te}) = 26.5$ kHz; $\nu_{\text{RF}}(^{31}\text{P}) = 36.5$ kHz; 2000 echoes were recorded.

the intensities of the individual spikelets were extracted and summed up with a Python script [35,36]. The alternative approach, co-addition of the individual echoes to produce the envelope spectra, did not lead to more reliable results. With these effective intensities the $\Delta S/S_0$ curves were calculated. The resulting dipolar evolution curves were analyzed using the SIMPSON software package [33,34]. An example code for SIMPSON simulations of CT-WUDOR-CPMG NMR experiments can be found in SI3.

3. Results and discussion

3.1. Validation on crystalline $\text{Ba}_2\text{TeO}(\text{PO}_4)_2$

To check the performance of CT-WUDOR-CPMG, we tested the

approach on the crystalline model compound $\text{Ba}_2\text{TeO}(\text{PO}_4)_2$. In $\text{BaTeO}(\text{PO}_4)_2$, Te has – via oxygen – three P neighbours with Te-P distances of 3.18 Å, 3.27 Å and 3.55 Å [27]. Taking into account all the Te-P distances within a sphere of 10 Å, an effective two-spin dipolar coupling constant of 814 Hz may be obtained by taking the root of the summed squares of the individual ^{125}Te - ^{31}P dipolar coupling constants. The ^{125}Te -WURST-CPMG and ^{31}P MAS and static NMR spectra are collected in Fig. SI4. From a simulation of these spectra, the CSA parameters for ^{125}Te ($\delta_{\text{iso}} = 1315$ ppm, $\delta_{\text{CSA}} = -700$ ppm, $\eta_{\text{CSA}} = 0.45$) and ^{31}P ($\delta_{\text{iso}} = 6.4$ ppm, $\delta_{\text{CSA}} = 62.5$ ppm, $\eta_{\text{CSA}} = 0.59$) were extracted.

The results of the $^{125}\text{Te}\{^{31}\text{P}\}$ -CT-WUDOR-CPMG NMR experiment are collected in Fig. 2. Fig. 2a shows CT-WUDOR-CPMG-NMR spectra without (S_0 ; top spectrum) and with (S ; middle spectrum) ^{31}P -

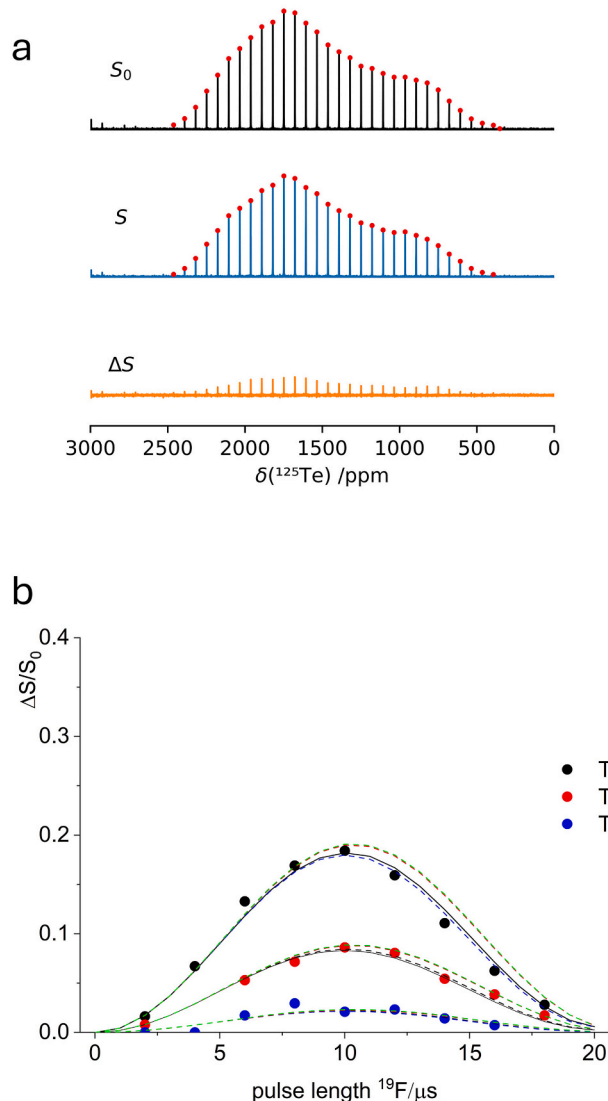
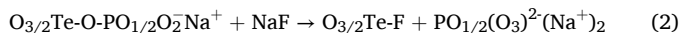
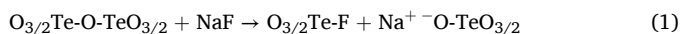


Fig. 3. a) $^{125}\text{Te}\{^{19}\text{F}\}$ -CT-WUDOR-CPMG spectra for sample T6NN5. Spectra marked S_0 (top) and S (middle) are without and with recoupling and the spectrum marked ΔS (bottom) is the difference spectrum. The ^{19}F recoupling pulse length for S is 12 μs . The red dots mark the intensities obtained employing a Python routine. b) $^{125}\text{Te}\{^{19}\text{F}\}$ -WUDOR-CPMG recoupling data for the measured samples T6NN1, T6NN3 and T6NN5. The curves represent SIMPSON simulations with $D = 550$ Hz (for T6NN1), $D = 1100$ Hz (T6NN3) and $D = 1650$ Hz (T6NN5). The Euler angles for the dipole tensor were varied. Black curves: $0^\circ 0^\circ 0^\circ$; dashed blue curves: $0^\circ 30^\circ 70^\circ$; dashed green curves: $0^\circ 70^\circ 88^\circ$; dashed red curves: $0^\circ 55^\circ 122^\circ$. Experimental details: recycle delay: 350 s; $d_3 = 10$ μs ; $d_6 = 76$ μs ; $\text{echod} = 14$ μs ; $\nu_{\text{RF}}(^{125}\text{Te}) = 25$ kHz; $\nu_{\text{RF}}(^{19}\text{F}) = 47$ kHz; 300 echoes were accumulated.

recoupling pulses. The difference spectrum is shown at the bottom. Varying the ^{31}P pulse length from 0 μs to 24 μs produces the data plotted as filled circles in Fig. 2b, showing excellent agreement with the results of the SIMPSON simulation for the theoretically calculated effective dipolar coupling constant of 814 Hz.

3.2. Application to fluorophosphotellurite glasses

Following the experimental validation of the sequence, CT-WUDOR-CPMG was used to study the heteronuclear dipolar coupling between ^{125}Te and ^{19}F as well as ^{125}Te and ^{31}P in a series of sodium fluorophosphotellurite glasses with composition $0.6\text{TeO}_2 - 0.4[(1-x)\text{NaPO}_3-x\text{NaF}]$ to obtain information about the structural organization in these glasses. The spectra obtained on the F-free sample ($x = 0$) indicate the formation of a Te-O-P polymeric network composed of metaphosphate ($\text{PO}_{2/2}\text{O}_2^-$) units linked to $\text{TeO}_{4/2}$ groups [28]. The two Te-based network forming units present in this glass are $\text{O}_{3/2}\text{Te-O-TeO}_{3/2}$ i.e. $^{\text{IV}}\text{Te}_{4\text{Te}}$ and $\text{O}_{3/2}\text{Te-O-PO}_{1/2}\text{O}_2\text{Na}^+$ i.e. $^{\text{IV}}\text{Te}_{3\text{Te},1\text{P}}$ units. In this terminology, generally used for mixed-network glasses, the coordination number is denoted by a superscripted Roman numeral, the number of bridging oxygen atoms is denoted by a superscripted Arabic numeral, and the number of atoms to which they are linked is given by a subscripted Arabic numeral. The replacement of NaPO_3 by NaF in the melt results in a marked decrease of the glass transition temperatures. Therefore, we hypothesized that increasing x along this compositional series promotes successive depolymerization of the Te-O-P network via the formation of Te-F bonds and terminal phosphate groups, following one of the two melt reaction schemes described below:



Here, reaction (1) involves breakage of a Te-O-Te linkage forming a Te-F bond and a non-bridging O atom linked to Te. Reaction (2) involves breakage of a Te-O-P linkage with formation of a Te-F bond and a non-bridging O-atom linked to phosphorus, which would then become an end group with one bridging O atom. Additional conceivable reactions involving the formation of P-F bonds were previously discounted based on ^{19}F NMR spectra [28].

Proving the formation of Te-F bonds by static ^{125}Te NMR was previously shown to be difficult as lineshapes for the different glass compositions showed only subtle changes as a function of x [28]. This could either mean that no Te-F bonds are formed or that the Te-O and the Te-F interactions influence the ^{125}Te chemical shift tensor in a similar fashion. Thus, $^{125}\text{Te}\{^{19}\text{F}\}$ -CT-WUDOR-CPMG may help to provide more decisive data. Fig. 3a exhibits the $^{125}\text{Te}\{^{19}\text{F}\}$ -CT-WUDOR-CPMG spectra for sample T6NN5 ($x = 0.5$) with the ^{19}F pulse length fixed at 12 μs without recoupling (S_0 , top, black), with recoupling (S , middle, blue) and the difference spectrum (ΔS , bottom, orange). The $^{125}\text{Te}\{^{19}\text{F}\}$ -CT-WUDOR-CPMG dipolar recoupling curves, obtained by incrementing the ^{19}F pulse lengths from 0 to 18 μs in steps of 2 μs , are shown in Fig. 3b together with the results from SIMPSON simulations, assuming a two-spin interaction with the dipolar coupling constant indicated. Since the relative orientations of the ^{125}Te chemical shift tensor and the ^{125}Te - ^{19}F dipole tensor are unknown, we checked the influence of this relative orientation by changing the Euler angles for the dipole tensor while keeping the Euler angles for the ^{125}Te CSA tensor fixed at 0° 0° 0° . The resulting recoupling curves were found to depend on the chosen relative orientation only marginally. The CSA-parameters for ^{31}P , ^{19}F and ^{125}Te , needed as input for the simulation of the dipolar recoupling curves were determined from the ^{19}F MAS spin echo spectra (acquired at 7.05 T and 11.7 T), ^{31}P MAS and static spectra (both acquired at 7.05 T) and ^{125}Te WURST-CPMG-spectra for sample T6NN5 and are given in the Supporting Information (cf. Figs. SI5 – SI7). The results, shown in Fig. 3b, clearly indicate increasing effective ^{125}Te - ^{19}F dipolar coupling constants with increasing fluorine content of the glass from 550 Hz in

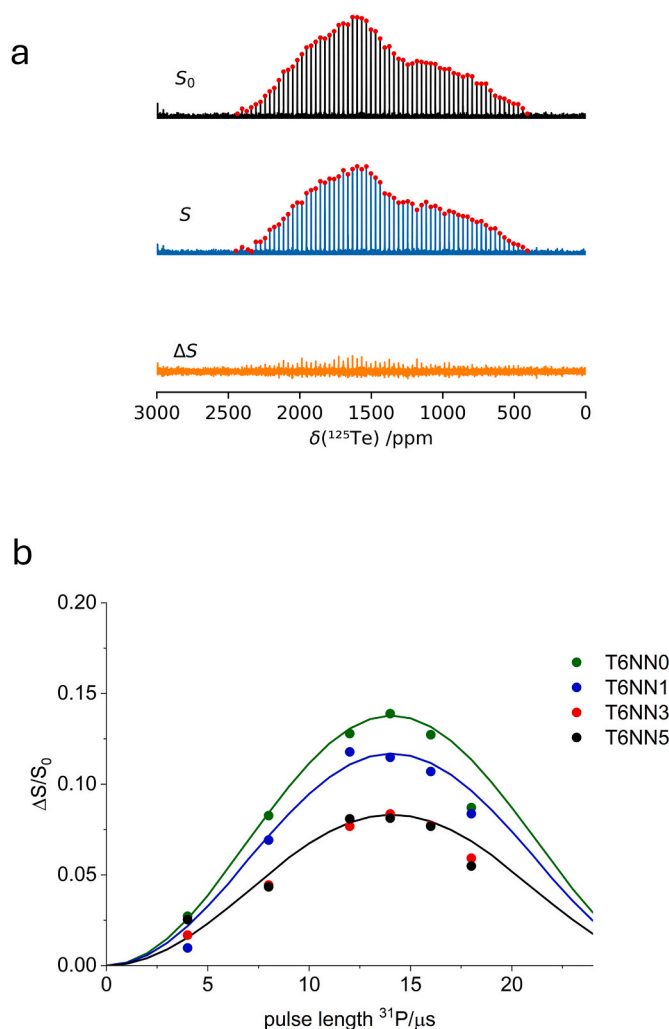


Fig. 4. a) $^{125}\text{Te}\{^{31}\text{P}\}$ -CT-WUDOR-CPMG spectra for sample T6NN5, arranged as in Fig. 3. The ^{31}P recoupling pulse length for S is 12 μs . b) $^{125}\text{Te}\{^{31}\text{P}\}$ -WUDOR-CPMG recoupling data for T6NN0, T6NN1, T6NN3 and T6NN5 together with SIMPSON simulations for $D = 460$ Hz (black curve), $D = 550$ Hz (blue curve) and 600 Hz (green curve). Experimental details: recycle delay: 350 s; $d_3 = 10$ μs ; $d_6 = 256$ μs ; $\text{echod} = 104$ μs ; $\nu_{\text{RF}}(^{125}\text{Te}) = 25$ kHz; $\nu_{\text{RF}}(^{31}\text{P}) = 35$ kHz; 300 echoes were accumulated.

T6NN1 ($x = 0.1$) to 1650 Hz in T6NN5 ($x = 0.5$). Taking into account that typical Te-F bond distances are in the range of 2.0 Å [37–40], corresponding to a dipolar coupling constant of 4500 Hz, the results in Fig. 3b indicate that not all the Te atoms, but a considerable fraction of them are involved in Te-F bonding. In fact, the conversion reactions (1) and (2) imply that a maximum of 7, 20, and 33% of the Te atoms in T6NN1, T6NN3, and T6NN5, respectively, form a Te-F single bond, whereas the remaining Te atoms experience significantly weaker magnetic dipole couplings. Thus, the clear evidence for Te-F binding provided by Fig. 3 strongly supports the conversion mechanisms (1) or (2) in these glasses.

We also used WUDOR to probe dipolar couplings beyond the first atomic coordination sphere in these glasses. Specifically, we used $^{125}\text{Te}\{^{31}\text{P}\}$ -CT-WUDOR-CPMG NMR to probe Te-O-P connectivity.

The results are collected in Fig. 4, following the same mode of presentation as in Fig. 3. We note that the extent of $^{125}\text{Te}\{^{31}\text{P}\}$ -CT-WUDOR NMR dephasing is rather small, owing to the relatively weak ^{125}Te - ^{31}P dipole-dipole coupling for this second-nearest neighbor interaction. Increased ΔS values would of course be possible to obtain via an increase of the dipolar mixing time, but in the present samples it would be at the

cost of a prohibitively worsened S/N ratio caused by spin-spin relaxation.

The simulation of the experimental data shown in Fig. 4 produces effective dipolar coupling constants of 460 Hz for T6NN5 and T6NN3, 550 Hz for T6NN1 and 600 Hz for T6NN0, respectively. As mentioned above and discussed in Ref. [28] the structure of the F-free glasses is based on randomly linked $\text{TeO}_{4/2}$ and $\text{PO}_{2/2}(\text{O}^-)_2$ units. Thus, the dominant tellurium environments in these glasses are Te species involved in zero, one or two Te-O-P linkages, i.e. $^{\text{IV}}\text{Te}_{4\text{Te}}$, $^{\text{IV}}\text{Te}_{1\text{P},3\text{Te}}$ and $^{\text{IV}}\text{Te}_{2\text{P},2\text{Te}}$ units. As expected, the effective ^{125}Te - ^{31}P dipolar coupling constant is significantly smaller in the T6NN3 and T6NN5 samples, which have significantly lower phosphate contents than the T6NN0 sample, and in which Te species having zero and one Te-O-P linkages are expected to dominate. The diminished ^{125}Te - ^{31}P dipolar interaction strength in the F-containing samples compared to T6NN0 confirms that some Te-O-P linkages have been replaced by Te-F linkages, along the lines of the mechanism described by eq. (2). On the other hand, the near-identical results obtained for T6NN3 and T6NN5 suggest that reaction (2) no longer contributes to F incorporation into the glass at $x > 0.3$. Overall, the results clearly indicate an intimate mixing of tellurite, phosphate and fluoride anions in these glasses, consistent with the model presented in Ref. [28]. More detailed analyses of the $^{125}\text{Te}\{^{19}\text{F}\}$ - and $^{125}\text{Te}\{^{31}\text{P}\}$ -CT-WUDOR-CPMG NMR recoupling curves in terms of structural models for these glasses and in the context of other complementary NMR experiments will be published elsewhere.

4. Conclusions

In this paper we introduced CT-WUDOR-CPMG NMR to obtain dipolar coupling information on observe nuclei with extremely broad resonance lines. The approach has been validated using crystalline $\text{Ba}_2\text{TeO}(\text{PO}_4)_2$. The results from $^{125}\text{Te}\{^{19}\text{F}\}$ -CT-WUDOR-CPMG NMR and $^{125}\text{Te}\{^{31}\text{P}\}$ -CT-WUDOR-CPMG NMR obtained on glasses in the system $\text{TeO}_2 - \text{NaPO}_3 - \text{NaF}$ clearly indicate intimate Te-P mixing and suggest considerable Te-F bonding. Experiments and simulations testing the applicability of CT-WUDOR-CPMG in the limit of extremely strong dipolar interactions ($D > 10$ kHz) are currently being performed in our laboratory.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.ssnmr.2026.102112>.

Data availability

We put research data including pulse sequences and simulation files into the supporting material.

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