

475 **8. Online supplemental material**

476 *8.1. Preparation of 10 mol/L LiCl from LiCl salt*

477 On a top-loading balance (Mettler-Toledo PB3002),  $(212.19 \pm 0.03)$  g of  
478 LiCl powder (equivalent to 5.0 mol of LiCl) were weighed out directly from  
479 the bottle into a tall-form, 1000 mL borosilicate glass beaker. This mass  
480 of powder had a volume of about 150 mL. The slow addition of 250 mL of  
481 distilled water (DW) was accompanied by considerable heat generation due  
482 to the exothermic hydration reaction. The goal was to dissolve all of the salt  
483 while carefully bringing the volume of the solution to 500 mL in order to

484 produce a nominally 10 mol/L LiCl solution. The salt formed a large, solid  
485 cake which dissolved slowly. The application of additional heat by means  
486 of a hotplate with occasional stirring dissolved the LiCl within 40 min, pro-  
487 ducing a volume of about 375 mL of hot solution. Further DW addition of  
488 80 mL brought the volume to  $\sim 460$  mL. The resulting solution, which was  
489 visibly turbid, was allowed to cool to room temperature over a period of 3 h.  
490 No apparent crystallization of LiCl occurred upon cooling. The solution was  
491 filtered under maximum suction but passed very slowly through a 47 mm,  
492  $0.45\ \mu\text{m}$  porosity membrane filter (Pall GN-6 Metricel, product no. 63069).  
493 The filtrate was transferred to a 500 mL glass volumetric flask, diluted to  
494 the mark and mixed. However, this filtered 10 mol/L LiCl solution soon ap-  
495 peared cloudy. It was eventually decided to allow the 10 mol/L LiCl solution  
496 to stand for several days to permit complete precipitation of any insoluble  
497 matter. It has been observed that such behavior may occur when very con-  
498 centrated solutions are prepared, presumably because the high ionic strength  
499 can affect the precipitation rate of even very insoluble compounds<sup>5</sup>. After 5  
500 d of standing, the suspension was again filtered under suction through a GN-  
501 6 ( $0.45\ \mu\text{m}$  porosity) membrane filter; the filtration rate was now relatively  
502 fast and the filtrate appeared to be clear and colorless. The 10 mol/L LiCl  
503 solution, which had a pH between 3 and 4 (measured by wide range indicator  
504 paper) and a density of  $1.205\ \text{g/mL}$  ( $\pm 1\ \%$ ,  $k=1$ ) at room temperature, was  
505 placed in a clear borosilicate glass bottle for storage. No further turbidity  
506 was observed.

507 A suggested procedure for future preparation of 10 mol/L LiCl solutions  
508 from the salt would be to add the salt (212 g) in small portions to the  
509 water ( $\sim 330$  mL to 350 mL), dissolving the added material before the next  
510 addition. Then, the solution should be allowed to stand  $\sim 1$  week to permit  
511 precipitation of insoluble matter before filtering to give the final solution.

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<sup>5</sup>One potential component of the insoluble precipitate is  $\text{BaSO}_4$ . If the Ba impurity was equal to 0.003% by weight, then 212 g of LiCl could produce as much as 0.010 g  $\text{BaSO}_4$ . This would exceed the solubility in a water solution of 500 mL (0.00123 g  $\text{BaSO}_4$ ). In the present situation, the 500 mL of 10 mol/L LiCl certainly contains less than 500 mL of water, which may further reduce the  $\text{BaSO}_4$  solubility, although the high ionic strength of the solution may counteract this effect.

512 *8.2. Preparation of 10 mol/L LiCl from Li<sub>2</sub>CO<sub>3</sub> salt*

513 We prepared 100 mL of a 10 mol/L LiCl solution starting with Li<sub>2</sub>CO<sub>3</sub>  
514 of presumably natural Li isotopic composition as an exercise to test and re-  
515 fine the procedures intended for use with isotopically enriched <sup>6</sup>Li<sub>2</sub>CO<sub>3</sub> for  
516 the PROSPECT experiment [25]. Almost exactly 37 g of lithium carbonate  
517 (from an old Baker and Adamson reagent-grade product<sup>6</sup>), approximately  
518 equal to 1.0 mol of Li, were weighed (directly from bottle) into a 250 mL  
519 glass beaker. A stoichiometric amount<sup>7</sup> of concentrated HCl (37.5 % by  
520 mass), 85 mL, was introduced into a 500 mL Erlenmeyer flask. The Li<sub>2</sub>CO<sub>3</sub>  
521 powder was added in small amounts using a plastic spoon to the Erlenmeyer  
522 flask over the course of 25 min with occasional swirling. The effervescence  
523 was easily under control; i.e., the spray and foaming were virtually completely  
524 contained within the flask, and the heating was moderated so that the solu-  
525 tion only became warm. The residue of Li<sub>2</sub>CO<sub>3</sub> in the 250 mL beaker was  
526 dissolved in a small amount of water containing a few drops of concentrated  
527 HCl and combined with the main liquid. However, the main solution in the  
528 Erlenmeyer flask was not clear. Concentrated HCl was added drop-wise with  
529 swirling until the effervescence ceased (approximately 4 mL). At this point,  
530 the solution was clear and distinctly light yellow-colored but with no visible  
531 insoluble matter. It was heated, boiled very briefly and allowed to cool over  
532 several hours to room temperature. The pH was 2 to 4 (using wide range  
533 indicator test paper) with a solution volume < 100 mL. The yellow color  
534 did not disappear upon cooling; it was suspected that the color derived from  
535 iron(III) impurity in the original salt since the previous solution prepared  
536 from LiCl salt was colorless. This yellow color was undesirable from the  
537 standpoint of producing color-quenching when mixed with liquid scintillator.  
538 Assuming it was due to Fe(III), the iron would most likely be present as the  
539 anionic chloro-complex FeCl<sub>4</sub><sup>-</sup> in this concentrated LiCl solution. As such  
540 it might be removed by using a strongly basic anion exchange resin such as  
541 AG 1 (quaternary amine functional group) just as Fe(III) is tightly retained  
542 on this resin from ≥ 6 mol/L HCl solution. Subsequently it was verified  
543 that when this LiCl solution was passed slowly through a 2 mL bed volume

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<sup>6</sup>Baker and Adamson (General Chemical Co.) reagent no. 1202, code 1872. Impurities (maximum, in %): Cl 0.005, SO<sub>4</sub> 0.25, Al<sub>2</sub>O<sub>3</sub> 0.03, CaO 0.10, Fe 0.004, heavy metals (as Pb) 0.002.

<sup>7</sup>Reaction: Li<sub>2</sub>CO<sub>3</sub> + 2HCl = 2LiCl + H<sub>2</sub>O + CO<sub>2</sub>(g); the carbon dioxide gas is evolved from the solution.

544 column of AG 1-X4, 100 to 200 mesh resin in chloride form, the yellow color  
545 was absent from the column eluate. If needed, the small amount of LiCl  
546 remaining in the resin after passing the solution could be washed out with  
547 9 mol/L HCl, leaving the Fe(III) in the resin. This 9 mol/L HCl – LiCl elu-  
548 ate could be evaporated, treated with 30 % H<sub>2</sub>O<sub>2</sub> to destroy organic traces  
549 (from the resin), re-dissolved, and combined with the main body of purified  
550 LiCl solution<sup>8</sup>. The column-purified LiCl solution (clear, colorless) was made  
551 up to 100 mL in a volumetric flask to give 10 mol/L LiCl with a measured  
552 density of 1.21 g/mL at room temperature.]

553 The final preparation of 10 mol/L <sup>6</sup>LiCl from <sup>6</sup>Li<sub>2</sub>CO<sub>3</sub> salt was carried  
554 out with the same technique. Out of 100 g of enriched-<sup>6</sup>Li lithium carbonate  
555 (95 % enrichment, origin Oak Ridge National Laboratory), 36 g were taken  
556 to prepare 100 mL of 10 mol/L <sup>6</sup>LiCl solution. The dissolution procedure  
557 followed closely that used for the natural Li<sub>2</sub>CO<sub>3</sub>, including a 500 mL Er-  
558 lenmeyer flask. The resulting solution in HCl was also yellow-colored and  
559 contained visible, undissolved black particles. Based on our experience with  
560 the natural-isotopic Li<sub>2</sub>CO<sub>3</sub>, a 2 mL bed volume column of AG 1-X4, 100  
561 to 200 mesh, chloride form anion exchange resin was prepared. The <sup>6</sup>LiCl  
562 solution was first filtered through fast filter paper (Whatman no. 41) by  
563 gravity to remove the black particles before passing through the AG 1 col-  
564 umn. The column successfully eliminated the yellow color from the eluate.  
565 <sup>6</sup>LiCl remaining on the column was stripped with 9 mol/L HCl, evaporated  
566 to dryness and treated with 30 % H<sub>2</sub>O<sub>2</sub> to destroy organic matter from the  
567 AG 1 resin<sup>9</sup>. The <sup>6</sup>LiCl recovered was about 1.5 g (out of 41.5 g <sup>6</sup>LiCl  
568 expected). All of the <sup>6</sup>LiCl was combined into a single 100 mL solution  
569 (nominally 10 mol/L <sup>6</sup>LiCl) with a measured density of 1.20 g/mL at room  
570 temperature.

571 All <sup>6</sup>LiCl solutions were stored in polyethylene bottles to allay concerns of

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<sup>8</sup>Passage of 5 mL of 0.1 mol/L HCl into the column after the 9 mol/L HCl immediately eluted the yellow substance, which gave a positive (cherry-red) spot test with 5 mol/L NH<sub>4</sub>SCN solution, a positive indicator for Fe(III). The solution was partially decolorized by the addition of HF or KF, converting the colored FeSCN<sup>2+</sup> to colorless FeF<sub>6</sub><sup>3-</sup>. While the residual yellow-colored impurity is not conclusively identified, it is removed by anion exchange chromatography.

<sup>9</sup>**Caution!** 30 % H<sub>2</sub>O<sub>2</sub> added to LiCl produces a very exothermic reaction resulting in immediate foaming/effervescence; therefore, cold H<sub>2</sub>O<sub>2</sub> must be added slowly with provisions for intercepting the spray generated by the gas liberation.

572 possible boron (a strong neutron absorber) leaching from borosilicate glass.