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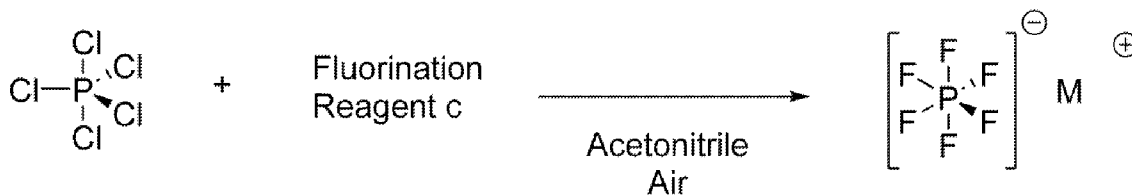
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FIG. 9



(57) Abstract: Provided herein are compositions and methods of manufacturing compositions useful in producing battery electrolyte precursors and battery electrolytes.

ELECTROLYTES AND ELECTROLYTE COMPONENTS, ADDITIVES, PRECURSORS THEREOF, AND METHODS OF MANUFACTURE

CROSS-REFERENCE

[0001] This application claims the benefit of priority to U.S. Provisional Application No. 63/521,153, filed June 15, 2023, which is incorporated herein by reference.

BACKGROUND OF THE INVENTION

[0002] Use of hazardous and toxic reagents, such as HF, to manufacture and produce battery electrolytes is dangerous and harmful to the environment. Provided herein are processes for manufacturing battery electrolytes and precursors thereof reducing and/or eliminating use of dangerous reagents.

SUMMARY OF THE INVENTION

[0003] In one aspect provided herein are methods for manufacturing battery electrolyte precursors. In some embodiments, methods provided herein comprise providing a fluorination reagent. In some embodiments, fluorination reagents are crude fluorination reagents that can be further purified to provide a purified fluorination reagent. In some embodiments, fluorination reagents are purified fluorination reagents. In some embodiments, the fluorination reagent comprises a first salt and a second salt. In some embodiments, the first salt comprises calcium and fluorine. In some embodiments, methods provided herein comprise contacting a fluorination reagent with a starting reagent to provide a battery electrolyte precursor.

[0004] In some embodiments, methods for manufacturing battery electrolyte precursors can comprise the second salt comprising an anion. In some embodiments, the anion of the second salt, when combined with Ca^{2+} to form a third salt, has a lattice energy greater than 2450 KJ/mol. In some embodiments, a powder x-ray diffraction spectrum of the fluorination reagent comprises characteristic 2θ reflections at about 21.9° , 30.3° , 31.6° , 43.4° and/or combinations thereof. In some embodiments, a powder x-ray diffraction spectrum of the fluorination reagent comprises characteristic 2θ reflections at about 28.1° , 49.0° , 52.3° , 54.1° , 60.0° , 69.7° and/or combinations thereof.

[0005] In one aspect, provided herein are methods for manufacturing fluorination reagents for providing battery electrolyte precursors. In some embodiments, fluorination reagents are crude fluorination reagents that can be further purified to provide a purified fluorination reagent. In some embodiments, fluorination reagents are purified fluorination reagents. In some embodiments, methods provided herein comprise combining a first salt with a second salt to form a mixed composition. In some embodiments, the first salt can comprise calcium and fluorine. In some embodiments, methods provided herein comprise applying mechanical force to a combination of a

first salt and a second salt to form a mixed composition. In some embodiments, methods provided herein comprise subjecting the mixed composition to a fluid composition and collecting a resultant fluid thereof. In some embodiments, subjecting the mixed composition to a fluid composition produces a solid component and a resultant fluid.

[0006] In some embodiments, methods provided herein comprise concentrating the resultant fluid. In some embodiments, concentrating the resultant fluid forms a crude fluorination reagent that can be further purified to provide a purified fluorination reagent. In some embodiments, concentrating the resultant fluid produces a reagent concentrate or precipitate. In some embodiments, methods provided herein comprise washing the fluorination reagent with a solvent to produce a reagent wash. In some embodiments, washing the fluorination reagent provides a second solid component and fluid reagent wash. In some embodiments, the reagent wash comprises a fluorination reagent. In some embodiments, the reagent wash comprises a purified fluorination reagent.

[0007] In some embodiments, methods provided herein comprise concentrating the reagent wash to form a fluorination reagent. In some embodiments, concentrating the reagent wash provides a purified fluorination reagent. In some embodiments, the purified fluorination reagent has a higher concentration of fluorine compared to the crude fluorination reagent. In some embodiments, methods provided herein comprise contacting fluorination reagents with starting reagents to provide fluorinated products. In some embodiments, fluorination reagents are purified fluorination reagents. In some embodiments, methods provided herein comprise contacting fluorination reagents with starting reagents to provide a battery electrolyte precursor.

[0008] In some embodiments, methods for manufacturing battery electrolyte precursors, and methods for manufacturing fluorination reagents for providing battery electrolyte precursors can comprise contacting fluorination reagents with starting reagents in an alkyl carbonate solvent (e.g., dimethyl carbonate). In some embodiments, fluorination reagents are crude fluorination reagents that can be further purified to provide a purified fluorination reagent. In some embodiments, fluorination reagents are purified fluorination reagents. In some embodiments, alkyl carbonate solvent is dimethyl carbonate, diethyl carbonate, dipropyl carbonate, methyl ethyl carbonate, methyl propyl carbonate, ethyl propyl carbonate, and/or combinations thereof. In some embodiments, the alkyl carbonate solvent is a fluoroalkyl carbonate solvent (e.g., trifluoroethyl carbonate, bis(trifluoroethyl) carbonate, trifluoroethyl methyl carbonate, and/or combinations thereof). In some embodiments, a combination of the fluorination reagent, the alkyl carbonate solvent, and the starting reagent is at any suitable temperature. In some embodiments, a combination of the fluorination reagent, the alkyl carbonate solvent, and the starting reagent is at a temperature of about 50 to about 150 °C. In some embodiments, a combination of the fluorination

reagent, the alkyl carbonate solvent, and the starting reagent is at a temperature of about 80 °C about or more (e.g., about 100 °C or more).

[0009] In one aspect, provided herein are methods for manufacturing battery electrolyte precursors. In some embodiments, methods provided herein comprise providing a fluorination reagent. In some embodiments, fluorination reagents are crude fluorination reagents that can be further purified to provide a purified fluorination reagent. In some embodiments, fluorination reagents are purified fluorination reagents. In some embodiments, the fluorination reagent comprises an alkali metal, fluoride, and at least one additional ion. In some embodiments, the alkali metal is lithium, potassium, or sodium. In some embodiments, methods provided herein comprise contacting the starting reagent with the fluorination reagent to provide a battery electrolyte precursor.

[0010] In one aspect, provided herein are methods for manufacturing battery electrolyte precursors. In some embodiments, methods provided herein comprise providing a fluorination reagent. In some embodiments, the fluorination reagent comprises calcium and fluorine. In some embodiments, methods provided herein comprise contacting the starting reagent with the fluorination reagent to provide a battery electrolyte precursor.

[0011] In some embodiments, methods for manufacturing fluorination reagents and methods for manufacturing fluorination reagents for providing battery electrolyte precursors provided herein can comprise adjusting the pH of the resultant fluid prior to concentrating the resultant fluid. In some embodiments, the resultant fluid can be adjusted to a pH of about 6 to about 8. In some embodiments, fluorination reagents are crude fluorination reagents that can be further purified to provide a purified fluorination reagent. In some embodiments, fluorination reagents are purified fluorination reagents.

[0012] In some embodiments, pH of the resultant fluid is adjusted with an acid. In some embodiments, the acid can comprise a strong acid, a weak acid, a polyprotic acid, and/or combinations thereof. In some embodiments, the acid can comprise phosphoric acid, hydrochloric acid, formic acid, acetic acid, benzoic acid, boric acid, silicic acid, oxalic acid, sulfuric acid, sulfurous acid, carbonic acid, and/or combinations thereof. In some embodiments, the acid can comprise hydrochloric acid, phosphoric acid, sulfuric acid, and/or combinations thereof.

[0013] In some embodiments, the resultant fluid can be adjusted to a pH of about 5 to about 10 (e.g., about 6 to about 9). In some embodiments, the fluid composition has a pH of about 7 or more (e.g., about 10 or more). In some embodiments, the fluid composition has a pH of about 12 to about 13. In some embodiments, a combination of the fluid composition and the mixed composition is at any suitable temperature. In some embodiments, a combination of the fluid composition and the mixed composition is at a temperature of about 0 to about 120 °C. In some embodiments, a

combination of the fluid composition and the mixed composition is at a temperature of 80 °C or more.

[0014] In some embodiments, a combination of the fluid composition and the mixed composition is at a temperature of 110 °C or less. In some embodiments, the mixed composition is subjected to the fluid composition for any suitable time. In some embodiments, the mixed composition is subjected to the fluid composition for about 0 hours to about 8 hours. In some embodiments, the mixed composition is subjected to the fluid composition for about 1 hour or more. In some embodiments, the mixed composition is subjected to the fluid composition for about 6 hours or less. In some embodiments, the mixed composition is subjected to the fluid composition for about 2 hours.

[0015] In some embodiments, the fluid composition has a boiling point of about 30 °C or more (e.g., about 70 °C or more, about 120 °C or more). In some embodiments, the fluid composition has a boiling point of about 240 °C or less. In some embodiments, a combination of the solvent and fluorination reagent is at any suitable temperature. In some embodiments, fluorination reagents are crude fluorination reagents that can be further purified to provide a purified fluorination reagent. In some embodiments, fluorination reagents are purified fluorination reagents. In some embodiments, a combination of the solvent and fluorination reagent is at a temperature of about -20 to about 240 °C. In some embodiments, a combination of the solvent and fluorination reagent is at a temperature of about 80 °C or more. In some embodiments, a combination of the solvent and the fluorination reagent is at a temperature of about 60 °C. In some embodiments, a combination of the solvent and fluorination reagent is at a temperature of about 235 °C or less.

[0016] In some embodiments, fluorination reagents are washed with a solvent for about 4 hours to about 48 hours (e.g., about 8 hours to about 36 hours, about 10 hours to about 28 hours). In some embodiments, fluorination reagents are washed with a solvent for about 8 hours or more. In some embodiments, fluorination reagents are washed with a solvent for about 36 hours or less. In some embodiments, fluorination reagents are washed with a solvent for about 18 hours. In some embodiments, a solvent has a boiling point of about 30 °C or more (e.g., about 70 °C or more, about 120 °C or more). In some embodiments, a solvent has a boiling point of about 240 °C or less. In some embodiments, a solvent is an organic solvent, water, an alcohol, a polar aprotic solvent, a halocarbon, and/or combinations thereof. In some embodiments, a fluid composition is an organic solvent, water, an alcohol, a polar aprotic solvent, a halocarbon, and/or combinations thereof.

[0017] In some embodiments, a solvent is acetonitrile, propionitrile, butyronitrile, toluene, 1,2-dichlorobenzene, chlorobenzene, fluorobenzene, 1,2-difluorobenzene, dichloroethane, trifluorotoluene, chloroform, sulfolane, DMF, DMSO, an alcohol (e.g., tert-butanol, tert-amyl alcohol), water, and/or combinations thereof. In some embodiments, a fluid composition is

acetonitrile, propionitrile, butyronitrile, toluene, 1,2-dichlorobenzene, chlorobenzene, fluorobenzene, 1,2-difluorobenzene, dichloroethane, trifluorotoluene, chloroform, sulfolane, DMF, DMSO, an alcohol (e.g., tert-butanol, tert-amyl alcohol), water, and/or combinations thereof. In some embodiments, a solvent is acetonitrile, propionitrile, butyronitrile, and/or combinations thereof.

[0018] In some embodiments, a fluid composition is acetonitrile, propionitrile, butyronitrile, and/or combinations thereof. In some embodiments, the mixed composition subjected to the fluid composition is provided as the first salt. In some embodiments, the first salt is a recovered waste material. In some embodiments, the first salt comprises low purity calcium and fluoride. In some embodiments, the first salt can comprise calcium and fluorine in less than 80 weight percent in total. In some embodiments, the first salt is CaF_2 or $\text{Ca}_5(\text{PO}_4)_3\text{F}$. In some embodiments, the second salt is a metal hydroxide, a metal sulphite, a metal sulphate, a carbonate, or an inorganic phosphate (e.g., a pyrophosphate).

[0019] In some embodiments, the second salt comprises NaOH , KOH , Na_2SO_3 , K_2SO_3 , KHSO_4 , CaCO_3 , H_2CO_3 , K_2CO_3 , Na_2CO_3 , $\text{K}_4\text{P}_2\text{O}_7$, $\text{Na}_4\text{P}_2\text{O}_7$, Na_3PO_4 , Li_3PO_4 , KHCO_3 , K_2CO_3 , NaHCO_3 , Cs_2CO_3 , K_2HPO_4 , KH_2PO_4 , K_3PO_4 , KPO_3 , $\text{K}_5\text{P}_3\text{O}_{10}$, K_2SO_4 , titanium phosphate, aluminum phosphate, uranium phosphate, and/or combinations thereof.

[0020] In some embodiments, crude fluorination reagents are purified at least in part using a filtration process. In some embodiments, a filtrate is concentrated and/or dried during any step or process of any method described herein. In some embodiments, the filtration process comprises passing any solution described herein through the same or a plurality of filtration modules a plurality of times (e.g., by making three or more consecutive passes through the same module and/or by passing once each through three consecutively coupled modules). In some embodiments, a fluorine recovery of a filtration process employed herein is greater than 90% (e.g., greater than 95% or greater than 99%). In some embodiments, a rejection of one or more contaminants by a filtration process employed in any method described herein is greater than 90% (e.g., greater than 95% or greater than 99%).

[0021] In some embodiments, an amount of phosphorous in the fluorination reagent is about 1 ppm to about 25 ppm (e.g., about 1 ppm, about 10 ppm, about 20 ppm, or about 25 ppm). In some embodiments, an amount of phosphorous in the fluorination reagent is about 0.015 % to about 12.5 % by weight (wt %). In some embodiments, an amount of calcium in the fluorination reagent is about 0.01 % to about 15 % by weight (wt %). In some embodiments, an amount of phosphorous in the fluorination reagent is about about 0.02 % to about 10 % by weight (wt %) (e.g., about 0.05 wt % to about 8 wt %, about 0.1 wt % to about 6 wt %, about 0.5 wt % to about 5 wt %, about 1 wt % to about 4 wt %). In some embodiments, an amount of phosphorous in the fluorination reagent

is about 0.015 % by weight (wt %) or more (e.g., about 0.05 wt % or more, about 0.1 wt % or more, about 0.5 wt % or more). In some embodiments, an amount of phosphorous in the fluorination reagent is about 5 % by weight (wt %) or less (e.g., about 3 wt % or less, about 2 wt % or less, about 1 wt % or less, about 0.5 wt % or less, about 0.1 wt % or less, about 0.05 wt % or less).

[0022] In some embodiments, a powder x-ray diffraction spectrum of the fluorination reagent comprises characteristic 2θ reflections at about 5.2° , 31.5° , 36.8° and/or combinations thereof.

[0023] In some embodiments, methods for manufacturing battery electrolyte precursors and methods for manufacturing fluorination reagents for providing battery electrolyte precursors provided herein can comprise fluorination reagents. In some embodiments, fluorination reagents are crude fluorination reagents that can be further purified to provide a purified fluorination reagent. In some embodiments, fluorination reagents are purified fluorination reagents. In some embodiments a combination of the fluorination reagent and the starting reagent comprises a reaction mixture. In some embodiments, the reaction mixture is at any suitable temperature. In some embodiments, the reaction mixture is at a temperature of about 55 to about 150 °C. In some embodiments, the reaction mixture is at a temperature of about 100 °C or less. In some embodiments, the fluorination reagent is contacted with the starting reagent for any suitable time. In some embodiments, the fluorination reagent is contacted with the starting reagent for about 12 hours or more. In some embodiments, the fluorination reagent is contacted with the starting reagent for about 14 hours to about 22 hours. In some embodiments, the fluorination reagent is contacted with the starting reagent for about 84 hours or less. In some embodiments, the fluorination reagent is contacted with the starting reagent for about 60 hours to about 80 hours.

[0024] In some embodiments, the reaction mixture further comprises a phase transfer agent, a base, and/or combinations thereof. In some embodiments, the phase transfer agent is a crown ether (e.g., 18 crown 6), a cryptand, an ionic transfer agent (e.g., tetramethylammonium chloride), and/or a hydrogen-bonding phase transfer agent. In some embodiments, the phase transfer agent is a crown ether (e.g., 18 crown 6). In some embodiments, the base is a pyridine or a derivative thereof (e.g., DMAP). In some embodiments, the reaction mixture comprises acetonitrile, propionitrile, butyronitrile, toluene, 1,2-dichlorobenzene, chlorobenzene, fluorobenzene, 1,2-difluorobenzene, dichloroethane, trifluorotoluene, chloroform, sulfolane, DMF, DMSO, an alcohol (e.g., tert-butanol, tert-amyl alcohol), water, and/or combinations thereof.

[0025] In some embodiments, the reaction mixture further comprises a reaction solvent. In some embodiments, the reaction solvent is an organic solvent, water, an alcohol, a polar aprotic solvent, a halocarbon, and/or combinations thereof. In some embodiments, the reaction solvent is acetonitrile, propionitrile, pyridine, butyronitrile, toluene, 1,2-dichlorobenzene, chlorobenzene,

fluorobenzene, 1,2-difluorobenzene, dichloroethane, trifluorotoluene, chloroform, DMF, DMSO, an alcohol (e.g., tert-butanol, tert-amyl alcohol), water, and/or combinations thereof. In some embodiments, the reaction solvent is acetonitrile, propionitrile, pyridine, butyronitrile, and/or combinations thereof.

[0026] In some embodiments, the starting reagent comprises a leaving group. In some embodiments, the leaving group is chlorine, iodine, or bromine. In some embodiments, the battery electrolyte precursor comprises at least one additional fluorine (e.g., at least two additional fluorine) compared to the starting reagent. In some embodiments, the battery electrolyte precursor is PF_3 , PF_5 , KPF_6 , or salts thereof.

[0027] In some embodiments, the at least one additional ion of the fluorination reagent comprises (i) at least one cation and at least one anion; or (ii) at least one zwitterion (e.g., psilocybin). In some embodiments, the at least one cation comprises K^+ , Na^+ , Ca^{2+} , Li^+ , or Cs^+ . In some embodiments, the at least one anion comprises a hydroxide, a sulphate, a carbonate, a phosphate, a pyrophosphate. In some embodiments, a molar ratio of the phase transfer agent to the starting reagent is about 0 to about 4. In some embodiments, a molar ratio of the base to the starting reagent is about 0 to about 2. In some embodiments, a molar ratio of a fluorine equivalent content in the fluorination reagent to the starting reagent is about 0.1 or more.

[0028] In some embodiments, a yield of the battery electrolyte precursor is about 10% or more. In some embodiments, a yield of the battery electrolyte precursor is about 20% to about 80%. In some embodiments, a concentration of the starting reagent in the reaction solvent and/or alkyl carbonate solvent is about 0.01 M to about 3 M. In some embodiments, a concentration of the starting reagent in the reaction solvent is about 1 M or less. In some embodiments, a concentration of the starting reagent in the alkyl carbonate solvent is about 1 M or less. In some embodiments, the fluorination reagent is contacted with the starting reagent under mechanochemical conditions (e.g., ball mill).

[0029] In some embodiments, the battery electrolyte precursor is contacted with an electrolyte agent (e.g., lithium salt) to provide a battery electrolyte. In some embodiments, the starting reagent is PCl_3 , PCl_5 , PF_5 , LiPCl_6 , P_4O_6 , P_2O_5 . In some embodiments, the battery electrolyte is LiPF_6 .

BRIEF DESCRIPTION OF THE DRAWINGS

[0030] The novel features of the invention are set forth with particularity in the appended claims. A better understanding of the features and advantages of the present invention will be obtained by reference to the following detailed description that sets forth illustrative embodiments, in which the principles of the invention are utilized, and the accompanying drawings (also “Figure” and “FIG.” herein), of which:

[0031] FIG. 1 illustrates an exemplary schematic of a mechanochemical method for combining one or more salts provided herein.

[0032] FIG. 2 illustrates an exemplary schematic of a process for manufacturing a fluorinating reagent provided herein.

[0033] FIG. 3 illustrates an exemplary schematic of using a fluorination reagent (e.g., a purified fluorination reagent) provided herein to synthesize a battery electrolyte or a battery electrolyte precursor.

[0034] FIG. 4 illustrates an exemplary schematic of a method of using a fluorination reagent provided herein to synthesize a battery electrolyte or a battery electrolyte precursor.

[0035] FIG. 5 illustrates an exemplary scheme of fluorinating a starting reagent provided herein.

[0036] FIG. 6 illustrates an exemplary scheme of fluorinating a starting reagent provided herein.

[0037] FIG. 7 illustrates an exemplary scheme of fluorinating a starting reagent provided herein.

[0038] FIG. 8 illustrates an exemplary scheme of fluorinating a starting reagent provided herein.

[0039] FIG. 9 illustrates an exemplary scheme of fluorinating a starting reagent provided herein.

[0040] FIG. 10 illustrates an exemplary scheme of preparing a battery electrolyte provided herein.

[0041] FIG. 11 illustrates an exemplary scheme of preparing a battery electrolyte provided herein.

[0042] FIG. 12 illustrates an exemplary scheme of preparing a battery electrolyte provided herein.

[0043] FIG. 13 illustrates an exemplary schematic of a process for manufacturing a fluorination reagent useful for preparing a battery electrolyte by methods provided herein.

[0044] FIG. 14 illustrates an exemplary schematic of a process for manufacturing a battery electrolyte precursor useful for preparing a battery electrolyte by methods provided herein.

[0045] FIG. 15 illustrates an exemplary schematic of a method of using a fluorination reagent provided herein to synthesize a battery electrolyte or a battery electrolyte precursor.

DETAILED DESCRIPTION OF THE INVENTION

Certain Definitions

[0046] As used herein and in the appended claims, the singular forms "a," "and," and "the" include plural referents unless the context clearly dictates otherwise. Thus, for example, reference to "an agent" includes a plurality of such agents, and reference to "the cell" includes reference to one or more cells (or to a plurality of cells) and equivalents thereof known to those skilled in the art, and so forth. When ranges are used herein for physical properties, such as molecular weight, or chemical properties, such as chemical formulae, all combinations and subcombinations of ranges and specific embodiments therein are intended to be included. The term "about" when referring to a number or a numerical range means that the number or numerical range referred to is an approximation within experimental variability (or within statistical experimental error), and thus the number or numerical range may vary between 1% and 15% of the stated number or numerical range. The term "comprising" (and related terms such as "comprise" or "comprises" or "having" or "including") is not intended to exclude that in other certain embodiments, for example, an

embodiment of any composition of matter, composition, method, or process, or the like, described herein, may "consist of" or "consist essentially of" the described features.

[0047] Provided herein are fluorination reagents and compositions useful for synthesizing battery electrolytes. In some instances, such reagents and compositions are useful in producing battery electrolytes in high-yields and/or without using toxic reagents, such as HF.

[0048] Provided herein are fluorination reagents and compositions useful for synthesizing battery electrolyte precursors. In some instances, such reagents and compositions are useful in producing battery electrolyte precursors in high-yields and/or without using toxic reagents, such as HF.

[0049] In some embodiments, provided herein is a method of manufacturing a battery electrolyte precursor (e.g., LiF, PF₅). In specific embodiments, any of the methods provided herein can comprise contacting a fluorination reagent with a starting reagent to provide a battery electrolyte precursor.

[0050] In some embodiments, provided herein is a method of manufacturing a battery electrolyte comprising lithium phosphorous hexafluoride or a salt thereof. In specific embodiments, any of the methods provided herein can comprise contacting a fluorination reagent with a starting reagent (e.g., a starting reagent provided herein) to provide a battery electrolyte.

[0051] Provided herein are fluorination reagents and compositions, as well as methods of making and using such fluorination reagents and compositions. In some instances, such reagents and compositions are useful in producing fluorinated products in high yield and/or without the need for use of toxic reagents, such as HF.

[0052] In some embodiments, provided herein is a method of manufacturing a fluorination reagent. In specific embodiments, the method comprises (1) combining (e.g., in the solid state, in solution, and/or combining a solid with a solution or liquid) a first salt with a second salt, the first salt comprising fluoride (e.g., and calcium); and (2) subjecting a combination of the first salt and the second salt to a (e.g., aqueous) fluid composition. In some embodiments, the resultant fluid composition is subsequently concentrated (e.g., by evaporation or other suitable method) to produce a fluorination reagent composition.

[0053] In certain embodiments, the fluorination reagent composition is further washed and/or purified with a (e.g., organic) solvent (e.g., an alcohol, such as methanol) to produce a reagent wash. In specific embodiments a (e.g., purified) fluorination reagent composition is recovered from the reagent wash (e.g., after filtering residual solids from the reagent wash). In some embodiments, separating a purified fluorination reagent from residual solids and/or separating contaminants from a resultant solution can independently comprise: centrifugation (e.g., using a decanter centrifuge and/or a disk stack centrifuge), press filtration, microfiltration, nanofiltration, ultrafiltration cross-flow membrane filtration and/or combinations thereof.

[0054] In some embodiments, crude fluorination reagents are purified at least in part using a filtration process. In some embodiments, a filtrate is concentrated and/or dried during any step or process of any method described herein. In some embodiments, the filtration process comprises passing any solution described herein through the same or a plurality of filtration modules a plurality of times (e.g., by making three or more consecutive passes through the same module and/or by passing once each through three consecutively coupled modules). In some embodiments, a fluorine recovery of a filtration process employed herein is greater than 90% (e.g., greater than 95% or greater than 99%). In some embodiments, a rejection of one or more contaminants by a filtration process employed in any method described herein is greater than 90% (e.g., greater than 95% or greater than 99%). In some embodiments, provided herein is a method of manufacturing a purified fluorination reagent, the method comprising:

- a. combining a first salt with a second salt to form a mixed composition, the first salt comprising calcium and fluoride;
- b. subjecting the mixed composition to a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
- c. concentrating the resultant fluid to produce a crude fluorination reagent (e.g., a reagent concentrate or precipitate);
- d. washing the crude fluorination reagent with a solvent (e.g., an alcohol) to produce a reagent wash (a second solid component and fluid reagent wash); and
- e. concentrating the reagent wash to form a purified fluorination reagent (e.g., the purified fluorination reagent having a higher concentration of fluorine compared to the crude fluorination reagent).

[0055] In specific embodiments, provided herein is a method of manufacturing a purified fluorination reagent, the method comprising:

- a. combining a first salt with a second salt to form a mixed composition, the first salt comprising calcium and fluoride;
- b. applying mechanical force to the mixed composition;
- c. subjecting the mixed composition to a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
- d. concentrating the resultant fluid to produce a crude fluorination reagent (e.g., a reagent concentrate or precipitate);
- e. washing the crude fluorination reagent with a solvent (e.g., an alcohol) to produce a reagent wash (a second solid component and fluid reagent wash); and

- f. concentrating the reagent wash to form a purified fluorination reagent (e.g., the purified fluorination reagent having a higher concentration of fluorine compared to the crude fluorination reagent).

[0056] In certain embodiments, provided herein are compositions or methods of providing (e.g., making, manufacturing, or the like) compositions comprising battery electrolytes or battery electrolyte precursors. In specific embodiments, a battery electrolyte provided herein comprises lithium phosphorous hexafluoride or a salt thereof (e.g., the product of any of the reactions illustrated in FIGs. 3 and 4). In yet more specific embodiments, a battery electrolyte precursor (e.g., as provided herein) is the precursor to lithium phosphorous hexafluoride, a Li-ion battery electrolyte. In certain embodiments, battery electrolytes or battery electrolyte precursors (e.g., PF_5 , KPF_6) are useful for producing battery electrolytes without the use of toxic reagents such as HF.

[0057] In certain embodiments, provided herein are compositions or methods of providing (e.g., making, manufacturing, or the like) compositions comprising reagents or reagent compositions. In some embodiments, reagents or reagent compositions provided herein are high purity and/or low-phosphorous reagents or reagent compositions. In some embodiments, presence of high purity and/or low phosphorous (e.g., a purified fluorination reagent provided herein) allows for the use of a reagent or reagent composition that produces high yield fluorination (e.g., relative to otherwise similar reagents/compositions having lower purity and/or higher phosphorous content). In certain embodiments, reagent or reagent compositions provided herein provide an improved rate of fluorination (e.g., at least about 10% improved). In certain embodiments (e.g., purified) reagents or reagent compositions provided herein have a higher fluorine content compared to (e.g., crude) reagents or reagent compositions provided herein. In some embodiments (e.g., purified) reagents or reagent compositions provide a rate of fluorination of a starting reagent (e.g., aromatic compound) that is higher when compared to a rate of fluorination provided by a (e.g., crude) reagent or reagent composition provided herein.

[0058] In some embodiments, any reagent (e.g., fluorination reagent, such as a purified fluorination reagent, or crude fluorination reagent) or (e.g., reagent) composition (e.g., any reagent or mixed composition, such as used in making of a reagent) provided herein comprises a first salt (e.g., calcium fluoride) provided herein. In certain embodiments, a reagent or reagent composition provided herein comprises a first salt and a second salt (e.g., K_2HPO_4) provided herein. In specific embodiments, a reagent or reagent composition provided herein comprises a first salt provided herein.

[0059] In some embodiments, any reagent or reagent composition provided herein comprises a metal (e.g., alkali metal, alkaline earth metal). In certain embodiments, a reagent or reagent composition comprises an alkali metal. In specific embodiments, a reagent or reagent composition

provided herein comprises an alkali metal (such as lithium, potassium, or sodium), fluoride, and (e.g., at least one additional) ion.

[0060] In certain embodiments, any composition provided herein comprises an ion (e.g., at least one additional ion herein). In some embodiments, a reagent (e.g., fluorination reagent, such as a purified fluorination reagent, or crude fluorination reagent) or (e.g., reagent) composition (e.g., any reagent or mixed composition, such as used in making of a reagent) provided herein comprises (e.g., at least one additional) ion. In specific embodiments, a reagent or reagent composition provided herein comprises at least one additional ion. In certain embodiments, a (e.g., salt or salt comprising a) composition provided herein comprises (e.g., at least one additional) ion. In specific embodiments, a (e.g., salt or salt comprising a) composition provided herein comprises at least one additional ion.

[0061] In some embodiments, an (e.g., at least one additional) ion provided herein comprises a cation, anion, and/or zwitterion. In some embodiments, an (e.g., at least one) cation provided herein comprises an alkali metal, alkaline earth metal, transition metal, other metal, cationic complex or ligand, or the like. In specific embodiments, an (e.g., at least one) cation provided herein is K^+ , Na^+ , Rb^+ , Ca^{2+} , Mg^{2+} , Fe^{2+} , Fe^{3+} , Cu^+ , Cu^{2+} , Ag^+ , Li^+ , NH_4^+ , Sr^+ , Ba^{2+} , Zn^{2+} , Cd^{2+} , Al^{3+} , $[Co(NH_3)_6]^{3+}$, or Cs^+ . In still more specific embodiments, (e.g., at least one) cation is K^+ , Na^+ , Ca^{2+} , Li^+ , Co^{3+} , Co^{2+} , U^{2+} , U^{4+} , U^{6+} , Ni^{2+} , and/or Cs^+ .

[0062] In certain embodiments, an (e.g., at least one) anion provided herein comprises a hydroxide, a sulphate, a carbonate, a phosphate, a pyrophosphate, a halide, a chlorate, a nitrate, a carbonate, a hydride, a sulfite, or the like. In specific embodiments, an (e.g., at least one) anion provided herein is a hydroxide, a sulphate, a carbonate, a phosphate, and/or a pyrophosphate.

[0063] In certain embodiments, an (e.g., at least one) zwitterion provided herein comprises an amino acid, a betaine, sulfamic acid, an acid, an aromatic compound, and/or a phospholipid. In specific embodiments, an (e.g., at least one) zwitterion provided herein is an amino acid, trimethylglycine, cocamidopropyl betaine, sulfamic acid, anthranilic acid, psilocybin, and/or phosphatidylcholine. In still more specific embodiments, an (e.g., at least one) zwitterion provided herein is psilocybin.

[0064] In certain embodiments, provided herein are reagents and reagent compositions with high purity and/or low levels of impurities (e.g., phosphorous, calcium, or the like). In some embodiments, high purity and low-content phosphorous allows for the use of a reagent or reagent composition that produces high-yield fluorination relative to other reagent or reagent compositions having low purity and/or higher phosphorous content. In certain embodiments, high purity and low-content calcium allows for the use of a reagent or reagent composition that produces high-yield fluorination relative to other reagent or reagent compositions having low purity and/or higher

calcium content. In some instances, low-content calcium and/or phosphorous and high purity reagent or reagent compositions allow substantially improved fluorination capabilities.

[0065] In some embodiments, any reagent or reagent composition provided herein (and/or produced or used herein) comprises low-content phosphorus. In certain embodiments, any reagent or reagent composition provided herein (and/or produced or used herein) comprises phosphorous in an amount of about 0.015 % to about 12.5 % by weight (wt %) (w/w). In some embodiments, a reagent or reagent composition provided herein comprises phosphorous in an amount of about 0.015 % by weight (wt %) or more (e.g., about 0.05 wt % or more, about 0.1 wt % or more, about 0.5 wt % or more). In certain embodiments, a reagent or reagent composition provided herein comprises phosphorous in an amount of about 1 % by weight or less (e.g., about 1 wt % or less, about 0.5 wt% or less, about 0.1 wt% or less, about 0.05 wt % or less).

[0066] In some embodiments, a reagent or reagent composition provided herein comprises phosphorous in an amount of about 0.05 % to about 10 wt % (e.g., about 0.1 wt % to about 6 wt %, about 0.5 wt% to about 5 wt %, about 1 wt% to about 4 wt %). In certain embodiments, a reagent or reagent composition provided herein comprises phosphorous in an amount of about 5 wt % or less (e.g., about 3 wt % or less, about 2 wt % or less, about 1 wt % or less, about 0.5 wt % or less, about 0.1 wt % or less).

[0067] In specific embodiments, a reagent or reagent composition provided herein comprises phosphorous in an amount of about 0.05 wt % to about 0.2 wt %.

[0068] In some embodiments, any reagent or reagent composition provided herein (and/or produced or used herein) comprises low-content calcium. In certain embodiments, any reagent or reagent composition provided herein (and/or produced or used herein) comprises calcium in an amount of about 0.01 % to about 15 % by weight (wt %) (w/w). In some embodiments, a reagent or reagent composition provided herein comprises calcium in an amount of about 0.01 % by weight (wt %) or more (e.g., about 0.05 wt % or more, about 0.1 wt% or more, about 0.5 wt % or more, about 1 wt % or more). In certain embodiments, a reagent or reagent composition provided herein comprises calcium in an amount of about 2 % by weight or less (e.g., about 1 wt% or less, about 0.5 wt% or less, about 0.1 wt % or less, about 0.05 wt% or less).

[0069] In some embodiments, a reagent or reagent composition provided herein comprises calcium in an amount of about 0.05 wt % to about 12 wt % (e.g., about 0.1 wt % to about 8 wt %, about 0.5 wt % to about 4 wt %). In certain embodiments, a reagent or reagent composition provided herein comprises calcium in an amount of about 6 wt % or less (e.g., about 4 wt % or less, about 2 wt % or less, about 1 wt % or less, about 0.5 wt % or less, about 0.1 wt % or less, about 0.05 wt % or less).

[0070] In specific embodiments, a reagent or reagent composition provided herein comprises calcium in an amount of about 0.01 % to about 0.05 wt %.

[0071] In certain embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at about 28.1° . In some embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at 28.1° , 49.0° , and/or 52.3° . In some embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at 28.1° , 49.0° , 52.3° , 54.1° , 60.0° , and/or 69.7° . In specific embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at 28.1° , 49.0° , 52.3° , 54.1° , 60.0° , and 69.7° .

[0072] In certain embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at about 21.9° . In some embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at 21.9° , 30.3° , and/or 31.6° . In certain embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at 21.9° , 30.3° , 31.6° , and/or 43.4° . In specific embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at 21.9° , 30.3° , 31.6° , and 43.4° .

[0073] In certain embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at about 5.2° . In some embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at 5.2° , 31.5° , and/or 36.8° . In certain embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein may further comprise peaks corresponding $\pm 0.2^\circ 2\theta$ to one or more 2-theta values from Table 2. In specific embodiments, a powder x-ray diffraction spectrum of a reagent or reagent composition provided herein comprises characteristic 2θ reflections at 5.2° , 31.5° , and 36.8° .

[0074] In certain embodiments, any reagent or reagent composition provided herein comprises high-content fluorine. In some embodiments, fluorine conversion (or F conversion) refers to a relative proportion or percentage (%) of fluorine from a (e.g., first) salt or salt composition provided herein that is converted to a reagent or reagent composition provided herein. In some embodiments, about 10 % to about 80 % of fluorine from a (e.g., first) salt or salt composition provided herein is converted into a (e.g., fluorination) reagent or reagent composition provided herein. In specific embodiments, about 30% to about 60% of fluorine from a (e.g., first) salt or salt composition provided herein is converted into a (e.g., fluorination) reagent or reagent composition provided herein.

[0075] In certain embodiments, Fluorine wt% or F wt% refers to fluorine content by weight in a reagent or reagent composition provided herein. In certain embodiments, Fluorine wt% or F wt% is measured by any suitable method (e.g., quantitative ^{19}F NMR). In some embodiments, a weight % of fluorine (F wt%) in a (e.g., fluorination) reagent or reagent composition provided herein is about 8% to about 75% (e.g., about 10% to about 70%, about 20% to about 60%, about 30% to about 50%, about 45% to about 55%). In specific embodiments, a weight % of fluorine (F wt%) in a (e.g., fluorination) reagent or reagent composition provided herein is about 20% or more (e.g., about 30% or more, about 40% or more, about 50% or more, about 60% or more, about 70% or more). In still more specific embodiments, a weight % of fluorine (F wt%) in a (e.g., fluorination) reagent or reagent composition provided herein is about 75% or less.

[0076] In certain embodiments, a (e.g., fluorination) reagent or reagent composition provided herein can be characterized by X-ray powder diffraction (XRPD) using Cu K α 1 ($\lambda = 1.5406 \text{ \AA}$) and/or Cu K α 2 ($\lambda = 1.5444 \text{ \AA}$). Due to differences in instruments, samples, and sample preparation, peak values are often reported with the modifier " $\pm 0.2^\circ 2\theta$ ". This is common practice in the solid-state chemical arts because of the variation inherent in peak values.

[0077] In certain embodiments, a (e.g., fluorination) reagent or reagent composition provided herein can have an XRPD pattern comprising peaks corresponding $\pm 0.2^\circ 2\theta$ to at least 1, at least 2, at least 3, at least 5, at least 10, at least 20, at least 50, and/or at least 70 of the 2-theta values reported in Table 2 provided herein. In some embodiments, a (e.g., fluorination) reagent or reagent composition provided herein can have an XRPD pattern comprising peaks corresponding $\pm 0.2^\circ 2\theta$ to at least 10%, at least 30%, at least 50%, at least 70%, at least 90%, and/or 100% of the 2-theta values reported in Table 2 provided herein. In specific embodiments, a (e.g., fluorination) reagent or reagent composition provided herein can have an XRPD pattern comprising peaks corresponding $\pm 0.2^\circ 2\theta$ to at least 30% of the 50 2-theta values reported in Table 2 provided herein (the (e.g., fluorination) reagent or reagent composition may have an XRPD pattern comprising peaks corresponding to at least 15 of the 2-theta values, modified $\pm 0.2^\circ 2\theta$, in Table 2).

[0078] Table 2 illustrates X-ray powder diffraction using Cu K α 1 ($\lambda = 1.5406 \text{ \AA}$) and/or Cu K α 2 ($\lambda = 1.5444 \text{ \AA}$) for a (e.g., fluorination) reagent or reagent composition provided herein (e.g., fluorination reagent C as provided herein in Example 2).

[0079] In certain embodiments a method provided herein comprises combining a first salt and a second salt.

[0080] In certain embodiments, any of the methods or compositions provided herein comprise a first salt. In some embodiments, a first salt provided herein comprises fluoride. In specific embodiments, the first salt comprises calcium and fluoride. In some embodiments, the first salt

further comprises additional ions, such as cations and/or anions provided herein. In specific embodiments, the first salt comprises CaF_2 , $\text{Ca}_5(\text{PO}_4)_3\text{F}$, and/or combinations thereof.

[0081] In some embodiments, the first salt or composition comprising the first salt comprises fluoride. In specific embodiments, the first salt or composition comprising the first salt comprises calcium and fluoride. In some embodiments, the first salt or composition comprising the first salt further comprises additional ions, such as cations and/or anions provided herein. In specific embodiments, the first salt or composition comprising the first salt comprises CaF_2 , $\text{Ca}_5(\text{PO}_4)_3\text{F}$, and/or combinations thereof.

[0082] In some embodiments, a first salt (e.g., a first salt provided herein) or a composition comprising a first salt provided herein is sourced from a material with low-value, low-purity, such as a waste material. In specific embodiments, the first salt provided herein is sourced from a waste material (e.g., calcium fluoride). In yet more specific embodiments, a composition comprising the first salt provided herein is sourced from a waste material. In certain embodiments, provided herein are methods for manufacturing reagents or reagent composition with waste materials. In some embodiments, a waste material (e.g., a waste material provided herein) comprises a raw, processed, and/or treated waste material. In certain embodiments, a waste material provided herein is a (e.g., recovered) waste product (e.g., sourced from an industrial process). In some embodiments, a waste material herein is a (e.g., recovered) waste product from an industrial process such as semiconductor manufacturing, fluorochemical manufacturing, pharmaceutical manufacturing, or the like. In certain embodiments, a waste material provided herein comprises fluorine (or a fluorinated salt), fluorapatite, calcium fluoride (e.g., in low purity), CFC-12, per- and polyfluoroalkyl substances (PFAs), or the like. In specific embodiments, a waste material provided herein comprises fluorine, or a fluorinated salt (e.g., in low purity). In yet more specific embodiments, a waste material provided herein comprises fluorine and calcium (e.g., in low purity). In certain instances, a waste material provided herein may be used as a raw, processed, or treated waste material to provide reagent or reagent compositions provided herein.

[0083] In certain embodiments, the first salt or composition comprising the first salt comprises calcium and fluoride in a collective amount of about 20% or less. In some embodiments, the first salt or composition comprising the first salt comprises calcium and fluoride in a collective amount of about 30% or less. In certain embodiments, the first salt or composition comprising the first salt comprises calcium and fluoride in a collective amount of about 40% or less. In certain embodiments, the first salt or composition comprising the first salt comprises calcium and fluoride in a collective amount of about 50% or less. In some embodiments, the first salt or composition comprising the first salt comprises calcium and fluoride in a collective amount of about 60% or less. In some embodiments, the first salt or composition comprising the first salt comprises calcium

and fluoride in a collective amount of about 70% or less. In some embodiments, the first salt or composition comprising the first salt comprises calcium and fluoride in a collective amount of about 80% or less.

[0084] In certain embodiments, the first salt comprises calcium and fluoride in a collective amount of about 20% or less. In some embodiments, the first salt comprises calcium and fluoride in a collective amount of about 30% or less. In certain embodiments, the first salt comprises calcium and fluoride in a collective amount of about 40% or less. In certain embodiments, the first salt comprises calcium and fluoride in a collective amount of about 50% or less. In some embodiments, the first salt comprises calcium and fluoride in a collective amount of about 60% or less. In some embodiments, the first salt comprises calcium and fluoride in a collective amount of about 70% or less. In some embodiments, the first salt comprises calcium and fluoride in a collective amount of about 80% or less.

[0085] In certain embodiments, any of the methods or compositions provided herein comprise a second salt. In some embodiments, a second salt provided herein comprises a metal, such as an alkali metal or an alkaline earth metal. In certain embodiments, the second salt comprises a metal (e.g., an alkali metal or an alkaline earth metal) and an anion (e.g., a phosphate, hydroxide, sulphate, carbonate, and/or sulphite). In some embodiments, the second salt comprises sodium, lithium, cesium, potassium, and/or combinations thereof. In certain embodiments, the second salt further comprises phosphate (e.g., such as an inorganic phosphate or a pyrophosphate), hydroxide, carbonate, sulphite, and/or a sulphate. In specific embodiments, the second salt is NaOH, Na₂SO₃, K₂SO₃, KOH, KHSO₄, K₂HPO₄, KH₂PO₄, K₃PO₄, Na₃PO₄, Li₃PO₄, K₂CO₃, Na₂CO₃, NaHCO₃, Cs₂CO₃, K₂SO₄, KPO₃, K₅P₃O₁₀, K₄P₂O₇, Na₄P₂O₇, titanium phosphate, aluminum phosphate, uranium phosphate, and/or combinations of one or more thereof. In some embodiments, the second salt further comprises additional ions, such as cations and/or anions provided herein.

[0086] In some embodiments, the second salt or composition comprising the second salt comprises a metal, such as an alkali metal or an alkaline earth metal. In certain embodiments, the second salt or composition comprising the second salt comprises a metal (e.g., an alkali metal or an alkaline earth metal) and an anion (e.g., such as a phosphate, hydroxide, sulphate, carbonate, and/or sulphite). In some embodiments, the second salt or composition comprising the second salt comprises sodium, lithium, cesium, potassium, and/or combinations thereof. In certain embodiments, the second salt further comprises phosphate (e.g., such as an inorganic phosphate or a pyrophosphate), hydroxide, carbonate, sulphite, and/or a sulphate. In specific embodiments, the second salt or composition comprising the second salt is NaOH, Na₂SO₃, K₂SO₃, KOH, KHSO₄, K₂HPO₄, KH₂PO₄, K₃PO₄, Na₃PO₄, Li₃PO₄, K₂CO₃, Na₂CO₃, NaHCO₃, Cs₂CO₃, K₂SO₄, KPO₃, K₅P₃O₁₀, K₄P₂O₇, Na₄P₂O₇, titanium phosphate, aluminum phosphate, uranium phosphate, and/or

combinations of one or more thereof. In some embodiments, the second salt or composition comprising the second salt further comprises additional ions, such as cations and/or anions provided herein.

[0087] In certain embodiments, any of the methods or compositions provided herein comprise a third salt. In some embodiments, a third salt provided herein comprises calcium. In certain embodiments, a third salt provided herein further comprises an anion provided herein. In specific embodiments, a third salt provided herein comprises calcium and an anion provided herein (e.g., a phosphate, hydroxide, sulphate, carbonate, and/or sulphite).

[0088] In some embodiments, a combination of a first salt (or a composition comprising the first salt) and a second salt (or a composition comprising the second salt) of any method provided herein provides a third salt. In some embodiments, a third salt provided herein comprises any cation (e.g., Ca^{2+}) of a first salt or composition comprising a first salt provided herein and any anion of a second salt or composition comprising a second salt provided herein.

[0089] In certain embodiments, the third salt provided herein or a composition comprising the third salt comprises calcium. In certain embodiments, the third salt or composition comprising the third salt further comprises an anion provided herein. In specific embodiments, the third salt or a composition comprising the third salt comprises calcium and an anion provided herein (e.g., a phosphate, hydroxide, sulphate, carbonate, and/or sulphite).

[0090] In some embodiments, a combination of a first salt (or a composition comprising the first salt) and a second salt (or a composition comprising the second salt) of any method provided herein provides a third salt or composition comprising a third salt. In some embodiments, a third salt or a composition comprising the third salt comprises any cation (e.g., Ca^{2+}) of a first salt or composition comprising a first salt provided herein and any anion of a second salt or composition comprising a second salt provided herein.

[0091] In certain embodiments, a third salt or composition comprising a third salt provided herein has a lattice energy of about 2400 kJ/mol or more (e.g., about 2600 kJ/mol or more, about 3000 kJ/mol or more). In specific embodiments, the third salt or composition comprising the third salt has a lattice energy of about 2450 kJ or more. In still more specific embodiments, the third salt or composition comprising the third salt has a lattice energy of about 2630 kJ/mol or more. In some embodiments, a lattice energy of a third salt or composition comprising the third salt provided herein is greater than a lattice energy of a first salt or composition comprising the first salt provided herein and/or a lattice energy of a second salt or composition comprising the second salt provided herein.

[0092] In certain instances, reactivity of a third salt or composition comprising the third salt provided herein with a high lattice energy (e.g., about 2500 kJ/mol or more) is low.

[0093] In some embodiments, provided herein is a composition or a method comprising combining a first salt (or a composition comprising the first salt) and a second salt (or a composition comprising the second salt) provided herein. In certain embodiments, a ratio of a first ion in a first salt (or a composition comprising the first salt) provided herein to a second ion in a second salt (or a composition comprising the second salt) provided herein is about 0.1:5 to about 5:0.1. In specific embodiments, a ratio of the first ion in the first salt (or a composition comprising the first salt) to the second ion in the second salt (or a composition comprising the second salt) is about 1:1. In yet more specific embodiments, a ratio of the first ion in the first salt (or a composition comprising the first salt) to the second ion in the second salt (or a composition comprising the second salt) is about 1:2.

[0094] In certain embodiments, the first salt (or a composition comprising the first salt) and the second salt (or a composition comprising the second salt) of any method provided herein are combined in any suitable manner (e.g., thereby providing a mixed composition described herein). In some embodiments, both the first salt (or a composition comprising the first salt) and the second salt (or a composition comprising the second salt) are combined as solids. In specific embodiments, the first salt (or a composition comprising the first salt) and the second salt (or a composition comprising the second salt) are combined to form a solid salt combination. In some embodiments, a method provided herein comprises applying a mechanical force to a mixed composition provided herein (e.g., comprising the first salt or a composition comprising the first salt and the second salt or a composition comprising the second salt). In specific embodiments, any suitable mechanical force provide herein is used.

[0095] In certain embodiments, the first salt (or a composition comprising the first salt) and the second salt (or a composition comprising the second salt) of any method provided herein are combined in any suitable manner to provide a third salt provided herein or a composition comprising the third salt.

[0096] In certain embodiments, a (e.g., mixed) composition provided herein comprises a first salt. In specific embodiments, the first salt comprises fluoride. In specific embodiments, the first salt comprises calcium and fluoride. In certain embodiments, a (e.g., mixed) composition provided herein comprises a second salt.

[0097] In some embodiments, a (e.g., mixed) composition provided herein comprises a reagent or reagent composition provided herein. In specific embodiments, a mixed composition provided herein is useful for directly fluorinating a compound (such as a starting reagent provided herein).

[0098] In certain embodiments, a mechanical force (e.g., a mechanical force provided herein) comprises any suitable mechanical force, such as by using a ball mill, a planetary mill, a mortar and pestle, a twin-screw-extruder, an attritor, a drum mill, an ultrasonic bath, a mechanical press,

and/or combinations of one or more thereof. In certain embodiments, a mechanical force is applied using a high-shear mixer, an in-line homogenizer, one or more bead mills, and/or combinations thereof. In certain embodiments, mechanical force provided herein is provided with a ball mill. In some embodiments, a ball mill provided herein comprises a jar and balls (e.g., with a weight of about 1 g to about 20 g). In certain embodiments, a first (e.g., salt) composition provided herein and a second (e.g., salt) composition provided herein are combined in a jar and balls are added. In some embodiments, mechanical force provided herein is provided with a twin screw-extruder, such as by extruding a combination of (e.g., salt) compositions provided herein at varying screw speeds, screw temperatures, residence times, or the like. A twin screw-extruder provided herein is fixed with a gravimetric single screw feeder (e.g., hopper) for programmed addition of (e.g., salt) compositions provided herein.

[0099] In specific embodiments, mechanical force is applied under any suitable condition, such as at a selected or varying frequency, time, temperature, cycles, or the like. In some embodiments, a mechanical force provided herein is applied at a frequency of about 0.5 Hz to about 60 kHz (e.g., about 10 Hz to about 20 kHz). In certain embodiments, a mechanical force provided herein is applied at a frequency of about 5 Hz or more (e.g., about 10 Hz or more, about 20 Hz or more, about 30 Hz or more). In specific embodiments, a mechanical force provided herein is applied at about 35 Hz. In certain embodiments, a mechanical force provided herein is applied for about 1 cycle to about 50 cycles (e.g., about 5 to about 40 cycles, about 10 to about 30 cycles). In some embodiments, a mechanical force provided herein is applied for 1 cycle or more. In specific embodiments, a mechanical force provided herein is applied for 10 cycles. In some embodiments, mechanical force is applied to one or more compositions in solution-phase. In some embodiments, mechanical force is applied to one or more compositions in solid-phase.

[0100] In certain embodiments, mechanical force provided herein is applied at a temperature of about 20 to about 300 ° C (e.g., about 50 to about 250 ° C, about 100 to about 200 ° C). In some embodiments, mechanical force provided herein is applied at a temperature of about 20 ° C or more (e.g., about 50 ° C or more, about 100 ° C or more, about 150 ° C or more). In some embodiments, the reaction mixture is refluxed at a reaction temperature. In some embodiments, the reaction temperature and/or a reflux temperature is about 100 to about 175 °C. In some embodiments, the reaction mixture is stirred in a pressure vessel. In some embodiments, the reaction is performed in a heated twin-screw extruder. In specific embodiments, mechanical force provided herein is applied at a temperature of about 25 ° C (e.g., at room temperature).

[0101] In certain embodiments, a mechanical force provided herein is applied for about 5 minutes to about 3 hours (e.g., about 10 minutes to about 2.5 hours, about 20 minutes to about 2 hours, about 30 minutes to about 1.5 hours). In some embodiments, a mechanical force provided herein

is applied for about 5 minutes or more (e.g., about 15 minutes or more, about 30 minutes or more, about 45 minutes or more, about 1 hour or more, about 2 hours or more). In specific embodiments, mechanical force provided herein is applied for about 45 minutes.

[0102] In some instances, varying time, frequency, temperature, and/or the like provides high yields of a reagent (e.g., fluorination reagent, such as a purified fluorination reagent, or crude fluorination reagent) or composition (e.g., any reagent or mixed composition, such as used in making of a reagent) provided herein.

[0103] In certain embodiments, a method provided herein comprises combining a first composition and a second composition, the first composition comprising a first salt and the second composition comprising a second salt. In more specific embodiments, the first and/or the second composition is a waste material provided herein (e.g., raw, processed, or treated waste material).

[0104] In some embodiments, provided herein is a composition or a method comprising subjecting a (e.g., mixed) composition to a (e.g., fluid) composition. In certain embodiments, the (e.g., mixed) composition of any method provided herein is subjected to a (e.g., fluid) composition (e.g., thereby forming a reagent or reagent composition, such as described herein). In some embodiments, the (e.g., mixed) composition is subjected to a (e.g., fluid) composition under any suitable conditions, such as at any selected temperature, with any selected volume of fluid composition, with stirring or other agitation, at any selected pH (e.g., using a buffer), for any selected period of time, or the like.

[0105] In certain embodiments, an (e.g., fluid) composition provided herein comprises any suitable solvent. In specific embodiments, a fluid composition provided herein comprises a solvent (e.g., a solvent provided herein). In certain embodiments, a fluid composition provided herein comprises any suitable solvent (e.g., water or an organic solvent). In specific embodiments, a fluid composition provided herein comprises a solvent (e.g., water).

[0106] In some embodiments, a solvent provided herein is any suitable solvent, such as a polar aprotic solvent, water, an alcohol, an alkyl carbonate solvent, a halocarbon and/or a combination thereof. In certain embodiments, a solvent provided herein is acetonitrile, propionitrile, butyronitrile, toluene, 1,2-dichlorobenzene, chlorobenzene, fluorobenzene, 1,2-difluorobenzene, dichloroethane, trifluorotoluene, chloroform, sulfolane, DMF, DMSO, tert-butanol, dichloromethane (DCM), tert-amyl alcohol, water, dimethyl carbonate, diethyl carbonate, dipropyl carbonate, methyl ethyl carbonate, methyl propyl carbonate, ethyl propyl carbonate, trifluoroethyl carbonate, bis(trifluoroethyl) carbonate, trifluoroethyl methyl carbonate, THF, MeTHF, NMP, butyl acetate, dioxane, and/or combinations thereof.

[0107] In some embodiments, a solvent provided herein is selected according to its characteristics, such as boiling point, ability to solubilize a composition provided herein, polarity, pH, or the like.

[0108] In certain embodiments, a solvent (e.g., a solvent provided herein) has a boiling point of about 30 °C or more. In some embodiments, a solvent provided herein has a boiling point of about 70 °C or more. In certain embodiments, a solvent or (e.g., fluid) composition provided herein has a boiling point of about 120 °C or more. In some embodiments, a solvent or (e.g., fluid) composition provided herein has a boiling point of about 240 °C or less.

[0109] In some embodiments, a (e.g., mixed) composition provided herein is subjected to a (e.g., fluid) composition provided herein for about 0 to about 8 hours. In certain embodiments, a (e.g., mixed) composition is subjected to a (e.g., fluid) composition for about 1 hour or more. In some embodiments, a (e.g., mixed) composition is subjected to a (e.g., fluid) composition for about 6 hours or less. In specific embodiments, a (e.g., mixed) composition is subjected to a (e.g., fluid) composition for about 2 hours.

[0110] In certain embodiments, a combination of a (e.g., mixed) composition provided herein and a (e.g., fluid) composition provided herein is at a temperature of about 0 to about 120 °C. In some embodiments, a combination of a (e.g., mixed) composition and a (e.g., fluid) composition is at a temperature of about 80 °C or more. In certain embodiments, a combination of a (e.g., mixed) composition and a (e.g., fluid) composition is at a temperature of about 110 °C or less. In certain instances, the selected temperature of a combination of a (e.g., mixed) composition and (e.g., fluid) composition provided herein increases a yield of a reagent or reagent composition provided herein.

[0111] In certain embodiments, a (e.g., mixed) composition provided herein is subjected to a (e.g., fluid) composition at a selected pH of about 3 to about 12. In some embodiments, pH of a (e.g., fluid) composition provided herein can be modified in any suitable manner (e.g., by using a buffer). In certain embodiments, the selected pH is about 4 or more. In some embodiments, the selected pH is about 7 or more. In certain embodiments, the selected pH is about 10 or more.

[0112] In certain embodiments, a (e.g., mixed) composition of any method provided herein is subjected to a (e.g., fluid) composition provided herein, thereby forming a resultant fluid (e.g., comprising a (e.g., crude) reagent or reagent composition that can be further purified to provide a (e.g., purified reagent or reagent composition) and a washed (e.g., mixed) composition. In some embodiments, the washed (e.g., mixed) composition is a solid. In certain embodiments, the resultant fluid comprises a reagent or reagent composition, such as described herein. In specific embodiments, the resultant fluid comprises a crude reagent or reagent composition provided herein.

[0113] In certain embodiments, a method provided herein comprises adjusting pH (e.g., by any suitable means) of a (e.g., resultant) fluid described herein. In some embodiments, the pH of a resultant fluid of any method provided herein is adjusted (e.g., using an acid, base, and/or buffer).

[0114] In certain embodiments, pH of a resultant fluid provided herein is adjusted to a pH of about 5 to about 10. In some embodiments, pH of a resultant fluid is adjusted to a pH of about 6 to about 9. In certain embodiments, pH of a resultant fluid is adjusted to a pH of about 6 to about 8 (e.g., thereby neutralizing the resultant fluid). In certain embodiments, a pH of a resultant fluid is adjusted based on a presence of an alkaline impurity in the first salt (e.g., to a pH of about 6). In certain embodiments, a pH of the resultant fluid is adjusted to a pH compatible with one or more downstream processes of a method described herein (e.g., a pH may be adjusted to about 7, about 8, or about 9 for a process requiring neutral or mildly basic solutions, such as when using a pH sensitive filtration media). In some cases, the pH of the resultant fluid is adjusted for compatibility with and/or separation on one or more ion exchange columns.

[0115] In some embodiments, pH of a resultant fluid provided herein is adjusted to a pH of about 8 to about 14. In certain embodiments, pH of a resultant fluid is adjusted to a pH of about 12 to about 13.

[0116] In certain embodiments, pH of a (e.g., resultant) fluid provided herein is adjusted with any suitable acid or base. In some embodiments, pH of a resultant fluid is adjusted (e.g., neutralized) with a (e.g., polyprotic) acid. In specific embodiments, a resultant fluid described herein is neutralized.

[0117] In certain embodiments, an acid (e.g., an acid provided herein) is any suitable acid, such as a strong acid, a weak acid, a polyprotic acid, and/or a combination thereof. In some embodiments, an acid provided herein is phosphoric acid, hydrochloric acid, formic acid, acetic acid, sulfuric acid, sulfurous acid, carbonic acid, benzoic acid, boric acid, silicic acid, oxalic acid, and/or a combination thereof. In certain embodiments, use of polyprotic acids (e.g., phosphoric acid) provided herein avoids releasing additional anions.

[0118] In certain embodiments, a base (e.g., a base provided herein) is any suitable base, such as a strong base, a weak base, an organic base, or the like. In some embodiments, a base provided herein comprises a hydroxide, an amine, ammonia, a pyridine, and/or a combination thereof. In certain embodiments, a base provided herein is NaOH, KOH, or LiOH. In specific embodiments, a base provided herein is KOH.

[0119] In certain embodiments, the washed (e.g., mixed) composition comprises a salt (e.g., a first salt as described herein). In some embodiments, a method provided herein comprises combining the washed (e.g., mixed) composition and a second salt as provided herein (e.g., thereby forming a mixed composition described herein). In specific embodiments, a mixed composition provided herein comprises the washed (e.g., mixed) composition. In certain embodiments, a washed (e.g., mixed) composition described herein is provided as a first salt provided herein (e.g., thereby providing for sustainable manufacturing of a reagent or reagent composition described herein). In

some instances, providing a washed (e.g., mixed) composition described herein as the first salt in methods and compositions described herein provides for sustainable manufacturing of reagents or reagent compositions. In certain instances, providing a washed (e.g., mixed) composition described herein as the first salt in methods and compositions described herein reduces the cost of waste disposal and/or the cost manufacturing a reagent or reagent composition provided herein.

[0120] In certain embodiments, provided herein is a composition or a method comprising concentrating a resultant fluid described herein (e.g., thereby forming a reagent or reagent composition, such as described herein). In some embodiments, a resultant fluid provided herein is concentrated by any suitable method and/or to any suitable endpoint provided herein. In certain embodiments, concentrating a resultant fluid described herein provides a (e.g., crude) reagent or reagent composition (e.g., a reagent concentrate or precipitate). In specific embodiments, concentrating a resultant fluid described herein provides a crude reagent or reagent composition (e.g., a reagent concentrate or precipitate). In yet more specific embodiments, the (e.g., crude) reagent or reagent composition is useful for directly fluorinating an organic compound provided herein (e.g., a starting reagent).

[0121] In certain embodiments, any suitable concentration method is used, such as by drying, lyophilizing, evaporating (e.g., using a rotary evaporator), distilling, or the like. In some embodiments, any fluid or wash provided herein is concentrated to any suitable endpoint (e.g., by about 10% or more). In specific embodiments, a resultant fluid described herein is concentrated by drying, evaporation, and/or a combination thereof. In still more specific embodiments, a resultant fluid described herein is concentrated under reduced pressure. In yet more specific embodiments, a resultant fluid described herein is concentrated under reduced pressure thereby providing a (e.g., crude) reagent or reagent composition provided herein.

[0122] In some embodiments, alternate concentration methods may be performed prior to, during, after, or in place of drying, lyophilizing, evaporating, distilling or the like. In some embodiments, alternate concentration methods comprise reverse osmosis, ultra-high pressure reverse osmosis, falling film evaporation, agitated thin film evaporation, spray-drying, and/or any combination of two or more thereof (e.g., up to, and including, a combination of all methods thereof).

[0123] In some embodiments, provided herein is a composition or a method comprising washing a (e.g., crude) reagent or reagent composition with a (e.g., solvent) composition. In certain embodiments, a (e.g., solvent) composition is any suitable solvent. In some embodiments, a (e.g., solvent) composition is any (e.g., organic) solvent (e.g., a solvent provided herein). In certain embodiments, the (e.g., crude) reagent or reagent composition of any method provided herein is washed with a (e.g., organic) solvent (e.g., thereby forming a reagent or reagent composition, such as described herein). In some embodiments, a (e.g., crude) reagent or reagent composition provided

herein is washed with a (e.g., organic) solvent under any suitable conditions, such as at a targeted temperature, with any selected volume of fluid composition, with stirring or other agitation, at any selected pH (e.g., using a buffer), at any selected temperature, for any selected period of time, or the like. In specific embodiments, a (e.g., crude) reagent or reagent composition provided herein is washed with an organic solvent (e.g., an alcohol).

[0124] In some embodiments, the reaction mixture is refluxed at a reaction temperature. In some embodiments, the reaction temperature and/or a reflux temperature is about 100 to about 175 °C. In some embodiments, the reaction mixture is stirred in a pressure vessel. In some embodiments, the reaction is performed in a heated twin-screw extruder.

[0125] In some embodiments, a (e.g., crude) reagent or reagent composition provided herein is washed with a (e.g., organic) solvent for about 4 hours to about 48 hours. In certain embodiments, a (e.g., crude) reagent or reagent composition provided herein is washed with a (e.g., organic) solvent for about 8 hours to about 36 hours. In some embodiments, a (e.g., crude) reagent or reagent composition provided herein is washed with a (e.g., organic) solvent for about 10 hours to about 28 hours. In certain embodiments, a (e.g., crude) reagent or reagent composition provided herein is washed with a (e.g., organic) solvent for 8 hours or more. In some embodiments, a (e.g., crude) reagent or reagent composition provided herein is washed with a (e.g., organic) solvent for 36 hours or less. In specific embodiments, a (e.g., crude) reagent or reagent composition provided herein is washed with a (e.g., organic) solvent for about 18 hours.

[0126] In certain embodiments, a combination of a (e.g., crude) reagent or reagent composition provided and a (e.g., organic) solvent is at a temperature of about -20 to about 240 °C. In some embodiments, a combination of the (e.g., crude) reagent or reagent composition and the (e.g., organic) solvent is at a temperature of about 80 °C or more. In certain embodiments, a combination of the (e.g., crude) reagent or reagent composition and the (e.g., organic) solvent is at a temperature of about 235 °C or less.

[0127] In some embodiments, a (e.g., crude) reagent or reagent composition of any method provided herein is washed with a (e.g., organic) solvent described herein, thereby providing a reagent wash (e.g., a fluid reagent wash) and a washed (e.g., reagent) composition. In certain embodiments, a (e.g., crude) reagent or reagent composition of any method provided herein is washed with a (e.g., organic) solvent, thereby providing a reagent wash (e.g., comprising a (e.g., purified) reagent or reagent composition). In some embodiments, the (e.g., fluid) reagent wash comprises a reagent or reagent composition, such as described herein. In specific embodiments, the (e.g., fluid) reagent wash comprises a purified reagent or reagent composition provided herein.

[0128] In certain embodiments, provided herein is a composition or a method comprising concentrating a (e.g., fluid) reagent wash described herein (e.g., thereby forming a reagent or

reagent composition, such as described herein). In some embodiments, a (e.g., fluid) reagent wash provided herein is concentrated by any suitable method and/or to any suitable endpoint provided herein. In certain embodiments, concentrating (e.g., fluid) reagent wash provided herein provides and/or produces a (e.g., purified) reagent or reagent composition (e.g., a reagent wash concentrate or reagent precipitate). In specific embodiments, the (e.g., purified) reagent or reagent composition is useful for directly fluorinating an organic compound provided herein (e.g., a starting reagent).

[0129] In certain embodiments, a reagent or reagent composition provided herein is activated, whereby the reagent or reagent composition comprises an (e.g., fluorination) reagent or reagent composition that can be used to fluorinate a starting reagent (e.g., organic compound) in that form. In some embodiments, any reagent or reagent composition provided herein comprises a fluorination reagent or reagent composition. In specific embodiments, an (e.g., crude) reagent or reagent composition provided herein comprises a fluorination reagent or reagent composition provided herein. In yet more specific embodiments, a (e.g., purified) reagent or reagent composition provided herein comprises a fluorination reagent or reagent composition provided herein. In still more specific embodiments, a reagent or reagent composition provided herein comprises a fluorination reagent or reagent composition provided herein (e.g., a mixed composition provided herein and/or a first salt provided herein).

[0130] In some embodiments, provided herein is a method for fluorinating a starting reagent comprising PCl_3 , PCl_5 , PF_5 , LiPCl_6 , P_4O_6 , P_2O_5 or a salt thereof. In certain embodiments, starting reagents provided herein comprise a leaving group (e.g., chlorine, iodine, bromine). In specific embodiments, a leaving group of a starting reagent provided herein is chlorine. In certain embodiments, provided herein are methods for fluorinating a starting reagent (e.g., a starting reagent provided herein) or a salt thereof to provide a battery electrolyte precursor (e.g., the product of any of the reactions illustrated in FIGs. 3-9). In some embodiments, provided herein are methods for fluorinating a starting reagent (e.g., a starting reagent provided herein) or a salt thereof to provide a battery electrolyte precursor (e.g., KPF_6). In specific embodiments, provided herein are methods for fluorinating PCl_3 . In yet more specific embodiments, provided herein are methods for fluorinating PCl_5 . In still more specific embodiments, provided herein are methods for fluorinating P_4O_6 . In yet more specific embodiments, provided herein are methods for fluorinating LiPCl_6 . In still more specific embodiments, provided herein are methods for fluorinating P_2O_5 . In yet more specific embodiments, the starting reagent is PCl_5 .

[0131] In some instances, a reagent or reagent composition provided herein is used to fluorinate a starting reagent (e.g., a starting reagent provided herein) to provide a high value, high yield battery electrolyte or battery electrolyte precursor without the use of toxic chemicals such as HF.

[0132] In certain embodiments, provided herein are methods for isolating a battery electrolyte precursor provided herein (e.g., phosphorous hexafluoride or a salt thereof). In some embodiments, a battery electrolyte precursor provided herein is isolated and/or concentrated (e.g., purified) by any suitable method such as by distillation, crystallization, recrystallization, sublimation, any suitable chromatography method (e.g., column, HPLC, or the like), trituration or the like. In certain embodiments, an isolation and/or concentration method (e.g., purification) comprises recrystallizing a battery electrolyte precursor provided herein in any suitable solvent (e.g., a solvent provided herein). In certain embodiments, an isolation and/or concentration method (e.g., an isolation or concentration method provided herein) further comprises triturating a battery electrolyte precursor provided herein using any suitable method. In some embodiments, a battery electrolyte precursor provided herein is triturated with any suitable solvent (e.g., a solvent provided herein) to provide a (e.g., isolated) battery electrolyte precursor provided herein. In some embodiments, the triturating comprises crushing a solid in a solvent selected to remove impurities. In certain embodiments, triturating comprises evaporating the solvent from the crushed solid.

[0133] In certain embodiments, provided herein are methods for providing a battery electrolyte provided herein (e.g., lithium phosphorous hexafluoride). In some embodiments, a battery electrolyte comprises lithium phosphorous hexafluoride or a salt thereof. In certain embodiments, provided herein is a method comprising contacting a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) with an electrolyte agent (e.g., thereby providing a battery electrolyte). In some embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) provides a battery electrolyte (e.g., a battery electrolyte provided herein). In certain embodiments, an electrolyte agent provided herein is any suitable electrolyte salt such as a lithium salt (e.g., lithium perchlorate, lithium sulphate, lithium chloride, lithium bromide, lithium tetrafluoroborate). In some embodiments, an electrolyte agent provided herein is any suitable electrolyte salt such as a lithium salt or a sodium salt (e.g., NaCl, NaClO₄). In specific embodiments, an electrolyte agent is lithium sulphate. In still more specific embodiments, the electrolyte agent is lithium perchlorate. In yet more specific embodiments, the electrolyte agent is lithium chloride. In still more specific embodiments, the electrolyte agent is lithium bromide. In yet more specific embodiments, the electrolyte salt is lithium tetrafluoroborate. In still more specific embodiments, the electrolyte salt is NaCl. In yet more specific embodiments, the electrolyte salt is NaClO₄.

[0134] In certain instances, characteristics of the starting reagent (e.g., a starting reagent provided herein), such as reactivity, (e.g., low) solubility provide difficulties for obtaining high yields of a battery electrolyte precursor or battery electrolyte.

[0135] In some embodiments, the battery electrolyte precursor is contacted with the electrolyte agent under any suitable conditions, such as at any selected temperature, with stirring or other agitation, at any selected pH, for any selected period of time, or the like.

[0136] In certain embodiments, an amount of an electrolyte agent (e.g., an electrolyte agent provided herein) is about 0 equivalents to about 5 equivalents of a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) (e.g., about 0.5 to about 4 equivalents, about 1 to about 3 equivalents). In some embodiments, the amount of the electrolyte agent is about 0.5 or more (e.g., about 1 or more, about 2 or more, about 4) equivalents of the battery electrolyte precursor. In certain embodiments, the amount of the electrolyte agent is about 4 or less (e.g., about 2 or less, about 1 or less, about 0.5 or less) equivalents of the battery electrolyte precursor. In specific embodiments, the amount of the electrolyte agent is about 1.1 equivalents of the battery electrolyte precursor. In yet more specific embodiments, the amount of the electrolyte agent is about 2 equivalents of the battery electrolyte precursor. In still more embodiments, the amount of the electrolyte agent is about 1.5 equivalents of the battery electrolyte precursor. In yet more embodiments, the amount of the electrolyte agent is about 1.16 equivalents of the battery electrolyte precursor.

[0137] In certain embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) in any suitable solvent (e.g., a solvent provided herein). In certain embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) in a selected solvent (e.g., a solvent provided herein). In certain embodiments, a (e.g., selected) solvent is any suitable solvent (e.g., as provided herein). In some embodiments, the selected solvent is an organic solvent. In certain embodiments, the selected solvent is acetonitrile, acetone, tetrahydrofuran (THF), an alcohol (e.g., ethanol, methanol), dioxane, methyl *t*-butyl ether, diethyl ether, methyl ethyl ketone (MEK), an alkyl carbonate (e.g., dimethyl carbonate, propylene carbonate), and/or combinations of one or more thereof. In specific embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) in acetonitrile. In yet more specific embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) in acetone. In still more specific embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) in propylene carbonate and dimethyl carbonate. In yet more specific embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte

agent provided herein) in methanol. In still more specific embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) in ethanol. In yet more specific embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) in dioxane.

[0138] In certain embodiments, a combination of a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) and an electrolyte agent (e.g., an electrolyte agent provided herein) is at a temperature of about 10 to about 80 °C (e.g., about 20 to about 70 °C, about 30 to about 60 °C). In some embodiments, a combination of the battery electrolyte precursor and the electrolyte agent is at a temperature of about 10 °C or more (e.g., about 15 °C or more, about 20 °C or more). In some embodiments, a combination of the battery electrolyte precursor and the electrolyte agent is at a temperature of about 60 °C or less (e.g., about 50°C or less, about 40°C or less, about 30°C or less). In specific embodiments, a combination of the battery electrolyte precursor and the electrolyte agent is at a temperature of about 25 °C. In yet more specific embodiments, a combination of the battery electrolyte precursor and the electrolyte agent is at room temperature.

[0139] In some instances, lowering the temperature (e.g., to about 20 °C or less) of a combination of the battery electrolyte precursor and electrolyte agent after a predetermined amount of time (e.g., about 6 hours to about 48 hours) provides an increased yield of the battery electrolyte.

[0140] In some embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) for about 1 hour to about 84 hours (e.g., about 2 hours to about 76 hours, about 3 hours to about 24 hours, about 4 hours to about 12 hours, about 5 hours to about 10 hours). In some embodiments, the battery electrolyte precursor is contacted with the electrolyte agent for about 30 hours or less (e.g., about 24 hours or less, about 18 hours or less, about 12 hours or less, about 8 hours or less). In certain embodiments, the battery electrolyte precursor is contacted with the electrolyte agent for about 84 hours or less (e.g., about 72 hours or less). In some certain embodiments, the battery electrolyte precursor is contacted with the electrolyte agent for about 2 hours or more (e.g., about 4 hours or more, about 8 hours or more, about 16 hours or more, about 32 hours or more). In certain embodiments, a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) is contacted with an electrolyte agent (e.g., an electrolyte agent provided herein) for about 0.5 hours to about 10 hours (e.g., about 1 hours to about 8 hours, about 2 hours to about 6 hours). In some embodiments, the battery electrolyte precursor is contacted with the electrolyte agent for about 8 hours or less (e.g., about 6 hours or less, about 4 hours or less, about 3 hours or less, about 1 hour or less). In certain embodiments, the battery electrolyte precursor is contacted with the electrolyte

agent for about 1 hour or more (e.g., about 2 hours or more, about 4 hours or more, about 8 hours or more). In specific embodiments, the battery electrolyte precursor is contacted with the electrolyte agent for about 2 hours. In yet more specific embodiments, the battery electrolyte precursor is contacted with the electrolyte agent for about 18 hours. In still more specific embodiments, the battery electrolyte precursor is contacted with the electrolyte agent for about 1 hour. In yet more specific embodiments, the battery electrolyte precursor is contacted with the electrolyte agent for about 8 hours. In certain embodiments, the temperature of the combination is changed (e.g., lowered to 0 °C or less) after a period of time.

[0141] In certain embodiments, a drying agent is added to a combination of a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) and an electrolyte agent (e.g., an electrolyte agent provided herein). In some embodiments, the drying agent can be any suitable drying agent. In certain embodiments, the drying agent is an organolithium compound (e.g., an alkyl lithium). In specific embodiments, the drying agent is methyl lithium, tert-butyl lithium, lithium hydride, or the like. In yet more specific embodiments, the drying agent is methyl lithium (MeLi). In certain instances, addition of a drying agent improves yield of a battery electrolyte.

[0142] In certain embodiments, a drying agent (e.g., as provided herein) is added to a combination of a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) and an electrolyte agent (e.g., an electrolyte agent provided herein) after the temperature of the combination of the battery electrolyte precursor and the electrolyte agent (e.g., the combination comprising the battery electrolyte) is lowered to about 20 °C or less (e.g., about 15 °C or less, about 10 °C or less, about 3 °C or less). In specific embodiments, the combination of the battery electrolyte precursor and the electrolyte agent is lowered to about 0 °C prior to addition of the drying agent. In yet more specific embodiments, the combination of the battery electrolyte precursor and the electrolyte agent is lowered to about 5 °C prior to addition of the drying agent.

[0143] In some embodiments, an amount of a drying agent provided herein is about 0.001 equivalents to about 5 equivalents (e.g., about 0.005 to about 1, about 0.01 to about 0.1) of a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein). In certain embodiments, the amount of the drying agent is about 0.005 or more (e.g., about 0.01 or more, about 0.05 or more) equivalents of the battery electrolyte precursor. In some embodiments, the amount of the drying agent is about 5 or less (e.g., about 1 or less, about 0.5 or less, about 0.1 or less, about 0.05 or less) equivalents of the battery electrolyte precursor. In specific embodiments, the amount of the drying agent is about 0.01 to about 0.1 equivalents of the battery electrolyte precursor. In yet more specific embodiments, the amount of the drying agent is about 0.024 equivalents of the battery electrolyte precursor. In certain embodiments, a concentration of a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) in a combination

provided herein (e.g., a combination of a battery electrolyte precursor and an electrolyte agent in a selected solvent provided herein) is about 0.05 M to about 5 M (e.g., about 0.1 M to about 4 M, about 0.2 M to about 3 M, about 0.5 M to about 1.5 M). In some embodiments, the concentration of the battery electrolyte precursor in the combination is about 0.05 M or more (e.g., about 0.1 M or more, about 0.2 M or more, about 0.8 M or more). In certain embodiments, the concentration of the battery electrolyte precursor in the combination is about 0.4 M or less (e.g., about 3 M or less, about 1.5 M or less). In specific embodiments, the concentration of the battery electrolyte precursor in the combination is about 1 M. In yet more specific embodiments, the concentration of the battery electrolyte precursor in the combination is about 1.5 M.

[0144] In certain embodiments, provided herein is a composition or a method comprising contacting a starting reagent (e.g., a starting reagent provided herein) with a reagent or reagent composition described herein (e.g., thereby fluorinating the starting reagent and providing a fluorinated product). In some embodiments, a starting reagent provided herein contacted with a reagent or reagent composition provided herein provides a battery electrolyte precursor provided herein. In some embodiments, the starting reagent is contacted with the reagent or reagent composition under any suitable conditions, such as at any selected temperature, with stirring or other agitation, at any selected pH, for any selected period of time, or the like.

[0145] In certain embodiments, a combination of a (e.g., fluorination) reagent or reagent composition provided herein and a starting reagent provided herein is at a temperature of about -10 to about 150 °C (e.g., about -5 to about 120 °C, about 0 to about 90 °C). In certain embodiments, a combination of a (e.g., fluorination) reagent or reagent composition provided herein and a starting reagent provided herein is at a temperature of about 20 to about 200 °C (e.g., about 60 to about 160 °C, about 70 to about 120 °C). In some embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 20 °C or more. In certain embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 40 °C or more. In some embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 60 °C or more. In certain embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 80 °C or more. In some embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 100 °C or more. In certain embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 120 °C or more. In some embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 140 °C or more. In specific embodiments, a combination of the (e.g., fluorination) reagent

or reagent composition and the starting at a temperature of about 0 °C. In yet more specific embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at room temperature. In still more specific embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at temperature of about 25 °C. In specific embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 75 °C. In still more specific embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 80 °C. In yet more specific embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 90 °C. In still more specific embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 110 °C. In yet more specific embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 115 °C. In still more specific embodiments, a combination of the (e.g., fluorination) reagent or reagent composition and the starting reagent is at a temperature of about 150 °C.

[0146] In certain embodiments, a starting reagent provided herein is contacted with a (e.g., fluorination) reagent or reagent composition provided herein for about 0.5 hour to about 50 hours (e.g., about 1 hours to about 40 hours, about 1.5 hours to about 20 hours, about 3 hours to about 10 hours). In certain embodiments, a starting reagent provided herein is contacted with a (e.g., fluorination) reagent or reagent composition provided herein for about 1 hour to about 84 hours (e.g., about 2 hours to about 76 hours, about 3 hours to about 24 hours, about 4 hours to about 12 hours, about 5 hours to about 10 hours). In some embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 30 hours or less (e.g., about 24 hours or less, about 18 hours or less, about 12 hours or less, about 8 hours or less). In certain embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 84 hours or less (e.g., about 72 hours or less). In some certain embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 1 hour or more (e.g., about 4 hours or more, about 16 hours or more, about 56 hours or more). In specific embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 6 hours. In yet more specific embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 8 hours. In still more specific embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 18 hours. In yet more specific embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 66 hours. In yet more specific embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent

or reagent composition for about 2 hours. In still more specific embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 3 hours. In yet more specific embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 5 hours. In still more specific embodiments, the starting reagent is contacted with the (e.g., fluorination) reagent or reagent composition for about 12 hours.

[0147] In certain instances, contacting a starting reagent with a (e.g., fluorination) reagent or reagent composition provided herein under inert conditions provides a greater yield of a battery electrolyte precursor (e.g., a battery electrolyte precursor provided herein) than under conditions without an inert atmosphere. In some instances, contacting a starting reagent with a (e.g., fluorination) reagent or reagent composition provided herein under non-inert conditions provides a substantial yield of a battery electrolyte precursor (e.g., about 50% yield or more).

[0148] In some embodiments, an amount of a (e.g., fluorination) reagent or reagent composition provided herein is about 0.1 equivalents to about 20 equivalents of a starting reagent provided herein. In certain embodiments, an amount of a (e.g., fluorination) reagent or reagent composition provided herein is about 0.1 equivalents to about 10 equivalents of a starting reagent provided herein. In some embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 1 or more (e.g., about 2 or more, about 3 or more, about 4 or more, about 5 or more, about 6 or more, about 7 or more, about 8 or more, about 9 or more) equivalents of the starting reagent. In certain embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 20 or less (e.g., about 18 or less, about 15 or less, about 12 or less, about 10 or less) equivalents of the starting reagent. In certain embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 10 or less (e.g., about 8 or less, about 6 or less, about 4 or less, about 2 or less) equivalents of the starting reagent. In specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 1 equivalent of the starting reagent. In still more specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 2 equivalents of the starting reagent. In yet more specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 3 equivalents of the starting reagent. In still more specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 4 equivalents of the starting reagent. In yet more specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 5 equivalents of the starting reagent. In still more specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 6 equivalents of the starting reagent. In yet more specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 7 equivalents of the starting reagent. In still more specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 8 equivalents of the starting reagent. In

yet more specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 9 equivalents of the starting reagent. In still more specific embodiments, the amount of the (e.g., fluorination) reagent or reagent composition is about 10 equivalents of the starting reagent.

[0149] In some instances, contacting greater equivalents of an (e.g., fluorination) reagent or reagent composition relative to the starting reagent results in high yields of a battery electrolyte precursor provided herein.

[0150] In some embodiments, a starting reagent provided herein is contacted with a (e.g., fluorination) reagent or reagent composition provided herein under mechanochemical conditions to provide a battery electrolyte precursor provided herein. In specific embodiments, any suitable mechanical force is used as provided herein and under any suitable conditions (e.g., as provided herein). In still more specific embodiments, a starting reagent provided herein is combined with a (e.g., fluorination) reagent or reagent composition provided herein in a laboratory mixer mill (e.g., and milled for 2 hours at 35 Hz) thereby providing a battery electrolyte precursor provided herein.

[0151] In certain embodiments, a starting reagent provided herein is contacted with a (e.g., fluorination) reagent or reagent composition provided herein in a reaction mixture. In some embodiments, a reaction mixture provided herein comprises a starting reagent, a (e.g., fluorination) reagent or reagent composition, and a (e.g., reaction) solvent. In certain embodiments, a reaction mixture provided herein comprises a starting reagent, a (e.g., fluorination) reagent or reagent composition, and a reaction solvent. In some embodiments, an (e.g., reaction) solvent is any suitable solvent (e.g., as provided herein). In certain embodiments, a reaction solvent is any suitable solvent (e.g., organic solvent) provided herein. In specific embodiments, the reaction solvent is acetonitrile, propionitrile, dimethyl carbonate (DMC), sulfolane, MeTHF, butyl acetate, dioxane, pyridine, butyronitrile, diethyl carbonate, NMP, and/or DMSO, and/or combinations of one or more thereof. In certain embodiments, the reaction solvent is acetonitrile, propionitrile, pyridine, butyronitrile, toluene, 1,2-dichlorobenzene, chlorobenzene, fluorobenzene, 1,2-difluorobenzene, dichloroethane, trifluorotoluene, chloroform, sulfolane, tetrahydrofuran (THF), n-methyl-2-pyrrolidone (NMP), DMF, DMSO, an alcohol (e.g., tert-butanol, tert-amyl alcohol), water, and/or combinations thereof. In specific embodiments, the reaction solvent is acetonitrile, chloroform, sulfolane, tetrahydrofuran (THF), n-methyl-2-pyrrolidone (NMP), and/or combinations thereof. In yet more specific, embodiments, the reaction solvent is acetonitrile, propionitrile, pyridine, and/or dimethylcarbonate, and/or combinations of one or more thereof. In still more specific embodiments, the (e.g., reaction) solvent is an alkyl carbonate solvent provided herein. In yet more specific, embodiments, the reaction solvent is acetonitrile. In still more specific embodiments, the (e.g., reaction) solvent is sulfolane. In yet more specific, embodiments, the reaction solvent is

chloroform. In still more specific embodiments, the (e.g., reaction) solvent is THF. In yet more specific, embodiments, the reaction solvent is NMP.

[0152] In certain embodiments, a reaction mixture provided herein comprises a starting reagent provided herein, a (e.g., fluorination) reagent or reagent composition provided herein, a reaction solvent (e.g., a reaction solvent provided herein), a reaction base (e.g., a reaction base provided herein), water (e.g., deionized water), and/or an alcohol (e.g., an alcohol provided herein), and/or combinations of two or more thereof.

[0153] In certain embodiments, a reaction mixture provided herein further comprises a phase transfer agent. In specific embodiments, a reaction mixture provided herein comprises a starting reagent provided herein, a (e.g., fluorination) reagent or reagent composition provided herein, a reaction solvent (e.g., a reaction solvent provided herein), and a phase transfer reagent (e.g., a phase transfer agent provided herein). In certain embodiments, a phase transfer agent (e.g., a phase transfer agent provided herein) is any suitable phase transfer agent, such as a crown ether, a cryptand, an ionic transfer agent (e.g., an ammonium salt), a hydrogen-bonding phase transfer agent, and/or a combination thereof. In some embodiments, a phase transfer agent provided herein is Kryptofix 221, Kryptofix 222, 18-crown-6, (Dibenzo)18-crown-6, (dicyclo)18-crown-6, 12-crown-4, 15-crown-5, 21-crown-7, cryptand-222, 30-crown-10, (dibenzo)30-crown-10, Schreiner's urea, ammonium sulfate, ammonium bicarbonate, ammonium chloride (e.g., tetramethyl ammonium chloride (TMAC)), ammonium iodide, ammonium benzoate, benzyltrimethyl, ammonium hydroxide, ammonium carbonate, ammonium dichromate, ammonium acetate, ammonium bromide, sodium tetradecyl sulfate, ammonium iodate and/or combinations thereof. In specific embodiments, a phase transfer agent provided herein is 18-crown-6, ammonium chloride, TMAC, and/or combinations thereof.

[0154] In some instances, addition of a phase transfer agent (e.g., a phase transfer agent provided herein) to a reaction mixture provided herein results in high yields of a battery electrolyte precursor provided herein.

[0155] In certain embodiments, an amount of a phase transfer agent (e.g., a phase transfer agent provided herein) is about 0 equivalents to about 8 equivalents of a starting reagent provided herein (e.g., about 0.05 to about 5 equivalents, about 0.1 to about 4 equivalents, about 0.5 to about 3 equivalents). In some embodiments, the amount of the phase transfer agent is about 0.05 or more (e.g., about 0.1 or more, about 0.5 or more, about 1 or more, about 2 or more) equivalents of the starting reagent. In certain embodiments, the amount of phase transfer agent is about 5 or less (e.g., about 3 or less, about 2 or less, about 1 or less, about 0.5 or less, about 0.1 or less) equivalents of the starting reagent. In specific embodiments, the amount of the phase transfer agent is about 1 equivalent of the starting reagent. In still more specific embodiments, the amount of the phase

transfer agent is about 2 equivalents of the starting reagent. In yet more specific embodiments, the amount of the phase transfer agent is about 0.2 equivalents of the starting reagent. In still more specific embodiments, the amount of the phase transfer agent is about 0.5 equivalents of the starting reagent. In yet more specific embodiments, the amount of the phase transfer agent is about 0.1 equivalents of the starting reagent. In still more specific embodiments, the amount of the phase transfer agent is about 0.08 equivalents of the starting reagent.

[0156] In certain embodiments, a reaction mixture provided herein further comprises a reaction base (e.g., a reaction base provided herein). In specific embodiments, a reaction mixture provided herein comprises a starting reagent provided herein, a (e.g., fluorination) reagent or reagent composition provided herein, a reaction solvent (e.g., a reaction solvent provided herein), and a reaction base (e.g., a reaction base provided herein). In yet more specific embodiments, a reaction mixture provided herein comprises a starting reagent provided herein, a (e.g., fluorination) reagent or reagent composition provided herein, a reaction solvent (e.g., a reaction solvent provided herein), a phase transfer agent (e.g., a phase transfer agent provided herein), and a reaction base (e.g., a reaction base provided herein). In still more specific embodiments, a reaction mixture provided herein comprises a starting reagent provided herein, a (e.g., fluorination) reagent or reagent composition provided herein, a reaction solvent (e.g., a reaction solvent provided herein), a phase transfer agent (e.g., a phase transfer agent provided herein), and one or more reaction bases (e.g., a reaction base provided herein). In certain embodiments, a reaction base (e.g., a reaction base provided herein) is any suitable base, such as an organic base, an amine, a pyridine, and/or a pyridine derivative. In some embodiments, a reaction base provided herein is 4-dimethylaminopyridine (DMAP), diisopropylethylamine (DIPEA), and/or pyridine. In specific embodiments, a reaction base provided herein is DMAP. In yet more specific embodiments, a reaction base provided herein is DIPEA. In still more specific embodiments, a reaction base provided herein is pyridine.

[0157] In some instances, addition of a reaction base provided herein to a reaction mixture provided herein results in high yields of a battery electrolyte precursor provided herein.

[0158] In certain embodiments, an amount of a reaction base (e.g., a reaction base provided herein) is about 0 equivalents to about 5 equivalents of a starting reagent provided herein (e.g., about 0.1 to about 4 equivalents, about 0.2 to about 3 equivalents, about 0.5 to about 2 equivalents). In some embodiments, the amount of the reaction base is about 0.05 or more (e.g., about 0.1 or more, about 0.5 or more, about 1 or more, about 2 or more) equivalents of the starting reagent. In certain embodiments, the amount of the reaction base is about 3 or less (e.g., about 2 or less, about 1.5 or less, about 1 or less, about 0.5 or less) equivalents of the starting reagent. In specific embodiments, the amount of the reaction base is about 0.2 equivalent of the starting reagent. In still more specific

embodiments, the amount of the reaction base is about 1.2 equivalents of the starting reagent. In yet more specific embodiments, the amount of the reaction base is about 1 equivalents of the starting reagent. In still more specific embodiments, the amount of the reaction base is about 2 equivalents of the starting reagent. In yet more specific embodiments, the amount of the reaction base is about 0.5 equivalents of the starting reagent.

[0159] In certain embodiments, a reaction mixture provided herein further comprises (e.g., deionized) water. In specific embodiments, a reaction mixture provided herein comprises a starting reagent provided herein, a (e.g., fluorination) reagent or reagent composition provided herein, a reaction solvent (e.g., a reaction solvent provided herein), a reaction base (e.g., a reaction base provided herein), and water.

[0160] In some instances, addition of water to a reaction mixture provided herein results in high yields of a battery electrolyte precursor provided herein.

[0161] In certain embodiments, an amount of water is about 0 equivalents to about 15 equivalents of a starting reagent provided herein (e.g., about 1 to about 12 equivalents, about 2 to about 9 equivalents, about 3 to about 7 equivalents). In some embodiments, the amount of water is about 1 or more (e.g., about 2 or more, about 4 or more, about 6 or more, about 8 or more) equivalents of the starting reagent. In certain embodiments, the amount of water is about 12 or less (e.g., about 10 or less, about 5 or less, about 2 or less) equivalents of the starting reagent. In specific embodiments, the amount of water is about 10 equivalents of the starting reagent. In still more specific embodiments, the amount of water is about 5 equivalents of the starting reagent. In yet more specific embodiments, the amount of water is about 7.5 equivalents of the starting reagent.

[0162] In certain embodiments, a reaction mixture provided herein further comprises an alcohol (e.g., t-amyl alcohol). In specific embodiments, a reaction mixture provided herein comprises a starting reagent provided herein, a (e.g., fluorination) reagent or reagent composition provided herein, a reaction solvent (e.g., a reaction solvent provided herein), a reaction base (e.g., a reaction base provided herein), and an alcohol (e.g., an alcohol provided herein). In certain embodiments, an alcohol (e.g., an alcohol provided herein) is any suitable alcohol, such as an alkyl alcohol, a diol, and/or a combination thereof. In some embodiments, an alcohol provided herein is ethyl alcohol, methanol, isopropyl alcohol, t-amyl alcohol, butanol, ethylene glycol, propylene glycol, and/or a combination thereof. In specific embodiments, an alcohol provided herein is isopropyl alcohol, t-amyl alcohol, and/or ethylene glycol.

[0163] In certain embodiments, an amount of alcohol (e.g., an alcohol provided herein) is about 0 equivalents to about 10 equivalents of a starting reagent provided herein (e.g., about 1 to about 9 equivalents, about 2 to about 8 equivalents, about 3 to about 7 equivalents). In some embodiments, the amount of alcohol is about 1 or more (e.g., about 2 or more, about 3 or more, about 4 or more,

about 8 or more) equivalents of the starting reagent. In certain embodiments, the amount of alcohol is about 9 or less (e.g., about 8 or less, about 7 or less, about 3 or less) equivalents of the starting reagent. In specific embodiments, the amount of alcohol is about 5 equivalents of the starting reagent.

[0164] In some embodiments, a starting reagent provided herein is contacted with a (e.g., fluorination) reagent or reagent composition provided herein with any selected volume of an (e.g., reaction) solvent.

[0165] In certain embodiments, a concentration of a starting reagent provided herein in a reaction mixture provided herein is about 0.05 M to about 1 M (e.g., about 0.1 M to about 0.75 M, about 0.2 M to about 0.5 M). In some embodiments, a concentration of the starting reagent in the reaction mixture is about 0.08 M or more (e.g., about 0.1 M or more, about 0.2 M or more, about 0.4 M or more). In certain embodiments, a concentration of the starting reagent in the reaction mixture is about 0.75 M or less (e.g., about 0.5 M or less, about 0.25 M or less). In specific embodiments, a concentration of the starting reagent in the reaction mixture is about 0.25 M. In yet more specific embodiments, a concentration of the starting reagent in the reaction mixture is about 0.33 M. In still more specific embodiments, a concentration of the starting reagent in the reaction mixture is about 0.5 M. In yet more specific embodiments, a concentration of the starting reagent in the reaction mixture is about 0.125 M.

[0166] In some instances, increasing a concentration of a starting reagent provided herein in a reaction mixture provided herein results in high yields of a battery electrolyte precursor provided herein.

[0167] In certain embodiments, a starting reagent provided herein contacted with a (e.g., fluorination) reagent or reagent composition provided herein provides a battery electrolyte precursor provided herein. In some embodiments, a leaving group (e.g., chlorine, iodine, bromine) of a starting reagent provided herein is replaced with fluorine. In certain embodiments, contacting a starting reagent provided herein with a (e.g., fluorination) reagent or reagent composition provided herein provides a battery electrolyte precursor provided herein in a yield of about 10% or more (e.g., about 20% or more, about 30% or more, about 40% or more, about 50% or more, about 60% or more, about 70% or more, about 80% or more, about 90% or more). In certain embodiments, a yield of a battery electrolyte precursor provided herein is about 10% to about 95% (e.g., about 20% to about 80%, about 30% to about 70%, about 40% to about 60%). In certain embodiments, a yield of a battery electrolyte precursor provided herein is about 50% to about 98% (e.g., about 60% to about 97%, about 70% to about 96%, about 80% to about 95%). In some embodiments, a yield of a battery electrolyte precursor provided herein is about 75% to about 95%.

[0168] In some embodiments, a battery electrolyte precursor provided herein comprises PF_3 , PF_5 , KPF_6 or salts thereof. In some embodiments, a battery electrolyte precursor provided herein comprises PF_3 , PF_5 , KPF_6 , NaPF_6 or salts thereof. In specific embodiment, the battery electrolyte precursor is KPF_6 . In yet more specific embodiments, the battery electrolyte precursor is NaPF_6 .

[0169] In some embodiments, a battery electrolyte provided herein is LiPF_6 or NaPF_6 . In specific embodiments, the battery electrolyte is LiPF_6 . In yet more specific embodiments, the battery electrolyte is NaPF_6 .

[0170] In certain embodiments, any of the steps provided herein can comprise any of the methods provided herein.

[0171] While preferred embodiments of the present invention have been shown and described herein, it will be obvious to those skilled in the art that such embodiments are provided by way of example only. Numerous variations, changes, and substitutions will now occur to those skilled in the art without departing from the invention. It should be understood that various alternatives to the embodiments of the invention described herein may be employed in practicing the invention. It is intended that the following claims define the scope of the invention and that methods and structures within the scope of these claims and their equivalents be covered thereby.

EXAMPLES

[0172] Example 1: applying mechanical force to a combination of a first and second salt

[0173] To a 50 mL stainless steel milling jar was charged calcium fluoride (1 equiv.), and a second salt (e.g., K_2HPO_4 , K_3PO_4) (1 or 2 equiv.). The jar was sealed finger tight and fastened to the MM500 Vario before milling for 10 cycles (1 hour at 35 Hz followed by 45 min at 5 Hz). For fluorination reagent B only, additional K_2HPO_4 (1 eq) was added to the mixture in the stainless steel milling jar. The jar was sealed finger tight and fastened to the MM500 Vario a second milling for 10 cycles (1 hour at 35 Hz followed by 45 min at 5 Hz). The jar was removed from the mill and taken to a fume cupboard before opening. The solid residue was removed and collected. An exemplary scheme is provided in FIG. 1.

Table 1

Fluorination Reagent	Fluorine source - equivalents	Activator - equivalents
A	calcium fluoride - 1 equiv.	K_2HPO_4 1 equiv.
B	calcium fluoride - 1 equiv.	K_2HPO_4 2 equiv.

[0174] Example 2: manufacture and use of a fluorination reagent

[0175] To a 50 mL stainless steel milling jar was charged calcium fluoride (1 equiv), and an activator, K_3PO_4 (1 eq). The jar was sealed finger tight and fastened to the MM500 Vario before milling for 10 cycles (1 hour at 35 Hz followed by 45 min at 5 Hz). The jar was removed from the

mill and taken to a fume cupboard before opening. The solid residue, a mixed composition, was removed and collected. An exemplary scheme is provided in FIG. 2.

[0176] The mixed composition (49.0 g, 168mmol) was stirred in water (150 mL, pH = 12-13) and heated at 100 °C for 2 h. The mixture was allowed to cool to room temperature before adjusting to pH of 7 by addition of ~ 20 ml of H₃PO₄ 20% (aqueous). 300 ml of methanol were added, and the suspension stirred for 30 minutes. The mixture was filtered washing with methanol. The filtrate was concentrated under reduced pressure to a small volume. Methanol (100 mL) was added to the flask and the suspension stirred at 50 °C for 1 h, then cooled down to rt and filtered. The filtrate was collected evaporated under reduced pressure and dried to yield an off-white purified fluorination reagent solid (11.5 g, 56 % fluorine conversion, 32 F wt%), fluorination reagent C. An exemplary reaction scheme is provided in FIG. 2. Powder X-ray diffraction data for fluorination reagent C is found in Table 2 below.

[0177] Additional purified reagents were prepared using a scheme similar to that provided in **FIG. 13**. A reactor described herein was charged with tripotassium phosphate, and mechanical force was applied according to methods described herein. The reactor was charged with calcium fluoride, heated to reflux, and aged before cooling to room temperature whilst continuing to apply mechanical force.

[0178] A resultant suspension was charged into a benchtop centrifuge. The solids were then separated and were combined with water to form a slurry which was cycled through the centrifuge. The liquids were charged into a container equipped with an overhead stirrer and stirring commenced. The solution was charged with phosphoric acid until a pH of 6 was obtained and stirring was performed for 1 hour.

[0179] The liquids were charged to and concentrated using a rotary evaporator to yield a white crystalline powder. A reactor as described herein was charged with methanol, mechanical force was applied, and the reactor was charged the white crystalline powder. The stirred suspension was heated, aged, cooled to room temperature, and discharged from the reactor. The resulting suspension was siphoned onto a Buchner funnel and filtered. The filter cake was re-slurried in methanol and then re-filtered.

[0180] The subsequent combined filtrates were then charged to a rotary evaporator and concentrated in vacuo to afford a crystalline solid comprising a purified fluorination reagent.

[0181] Production of a purified fluorination reagent was also achieved using reactions at elevated temperatures (including 150 °C) in a pressure flask, as well as using high shear mixing and/or a homogenizer as a reactor, as described herein.

[0182] For example, production of a purified fluorination reagent using a reactor equipped with an in-line homogeniser comprised charging the reactor, sealing the reactor, and applying

mechanical force. The reaction mixture was and then cooled to room temperature. The resulting suspension was purified using similar methods to those described throughout this example.

Further, spray-drying was tested for purification and produced similar results.

[0183] Table 2: XRPD of fluorination reagent C

Angle ($^{\circ}2\theta$)	Intensity [a.u.]
17.1065	48.03
18.1243	56.65
19.8556	180.49
20.1077	443.85
21.7697	127.08
24.6649	55.53
26.0729	59.75
26.5044	765.05
27.8025	593.63
28.8975	1146.01
29.6196	1502.66
30.7473	181.75
33.5278	3742.12
34.1891	299.51
34.5311	619.91
34.5606	259.81
35.0389	711.86
36.0813	137.03
36.6838	132.76
40.8449	35.02
41.6134	472.23
42.5274	1030.97
44.3484	550.71
44.5738	159.05
46.2653	170.23
46.7412	525.11
48.0764	2597.43
49.0280	84.97
49.1401	118.82

50.1292	60.09
50.5421	71.06
52.0373	83.41
52.4004	213.73
54.0201	207.41
57.1244	590.72
57.7184	226.83
58.4393	49.82
59.8714	1179.03
61.9070	71.87
63.7016	161.22
64.6762	99.11
66.3163	159.72
66.7518	92.20
67.6036	93.35
70.4056	467.93
72.3758	149.53
72.5514	50.60
75.0914	61.76
75.2740	47.83
77.8145	372.80

[0184] Fluorination reagent C (1.5 eq), an optional additive (1 eq) (TMAC or 18-crown-6), and a starting reagent are combined and stirred in acetonitrile (80 °C, 3h) to provide a battery electrolyte precursor. All reaction parameters are provided in Table 3. An exemplary reaction scheme is provided in FIG. 3.

Table 3

Starting Reagent	Battery Electrolyte Precursor
PCl ₃	PF ₃
PCl ₅	PF ₅

Example 3: Mechanochemical conditions for providing a battery electrolyte precursor

[0185] Fluorination reagent C (1.5 eq) and a starting reagent are combined under mechanochemical conditions (milled for 2 hours at 35 Hz) to provide a battery electrolyte precursor. Reagents and reaction conditions are provided in Table 4. An exemplary reaction scheme is provided in FIG. 4.

Table 4

Starting Reagent	Battery Electrolyte Precursor
PCl ₃	PF ₃
PCl ₅	PF ₅

Example 4: Use of a Fluorination Reagent to Fluorinate PCl₅

[0186] To an oven dried flask with stirring device was added anhydrous acetonitrile under inert atmosphere and a fluorination reagent added under constant inert gas flow. The mixture was then cooled to 0 °C whilst stirring and phosphorus pentachloride was added slowly. Upon complete addition, the reaction was allowed to stir at 0 °C for 2h. The mixture was diluted with acetonitrile and then filtered. The filter cake was washed with acetonitrile and the filtrate concentrated, before allowing to dry under high vacuum to afford a metal hexafluorophosphate(V) which was analyzed by ¹⁹F-NMR. An exemplary reaction scheme is provided in FIG. 5.

Example 5: Fluorination Reagent Purity and Yield of Battery Electrolyte Precursor

[0187] The procedure as provided in Example 4 was reproduced with fluorination reagents of different levels of purity, the results of which are provided in Table 5. An exemplary reaction is provided in FIG. 6.

Table 5

Fluorination Reagent Purity	Yield (%)	Purity Battery Electrolyte Precursor (%)
93	80	92
86	88	96
86	83	96
98	91	98
78	73	93
50	1	3
87	63	77
87	77	93
98	85	96
67	76	92

Example 6: Formation of Battery Electrolyte Precursor Varying Reaction Solvent

[0188] A solvent screen was undertaken in the general procedure of Example 4. As shown in Table 6 below, acetonitrile proved to yield the most product. The full results of the solvent screen are provided in Table 6 below and an exemplary reaction scheme is provided in FIG. 7.

Table 6

Solvent	Temperature [°C]	yield % (MPF ₆)
Chloroform	Rt	5
Chloroform	50	0
Sulfolane	100	55
Sulfolane	150	27
Acetonitrile	Rt	80
Acetonitrile	0	88
THF	Rt	3
NMP	100	1

Example 7: Formation of Battery Electrolyte Precursor Varying Fluorination Reagent amount

[0189] An exemplary reaction scheme is provided in FIG. 8 and results of varied equivalents is provided in Table 7 below.

[0190] The reaction does also occur with sub-stoichiometric quantities of the fluorination reagent.

Table 7

Equivalents	Yield (%)	Purity (%)
3	6	-
6	70	88
7	88	98
10	80	92

Example 8: Formation of Battery Electrolyte Precursor varying reaction conditions

[0191] A test was undertaken to determine whether running the reaction without an inert atmosphere was possible under the general procedure of Example 4, as shown in Table 8 below. An exemplary reaction scheme is provided in FIG. 9.

Table 8

Atmosphere	Yield (%)	Purity (%)
Nitrogen	80	92
Air	63	77

Example 9: Preparation of battery electrolyte LiPF₆ (solvent screen)

[0192] In a 50 ml 2-necked flask oven dried under nitrogen with stirring bar, lithium chloride (1-2 equiv.) was weighed in and suspended in the appropriate solvent, the suspension was stirred for 40 minutes. Metal hexafluorophosphate (1 equiv.) was added as a solid in one portion and the suspension left stirring for 18 h at room temperature. The suspension was concentrated under nitrogen and filtered. The solid evaporated to dryness overnight. Lithium hexafluorophosphate was isolated. Purity was assessed by ¹⁹F and ⁷Li NMR. The tested solvents are provided in Table 9 below and an exemplary reaction scheme is provided in FIG. 10.

Table 9

Solvent	Yield %	Purity %
Acetonitrile	80	60
Acetone	80	80

Example 10A: Preparation of battery electrolyte LiPF₆ (variation of lithium salt)

[0193] In a 50 ml 2-necked flask oven dried under nitrogen with stirring bar, the appropriate lithium salt (1-2 equiv.) was weighed in and suspended in the appropriate solvent, the suspension was stirred for 40 minutes. Metal hexafluorophosphate (1 equiv.) was added as a solid in one portion and the suspension left stirring for 18 h at room temperature. The suspension was concentrated under nitrogen and filtered. The solid evaporated to dryness overnight. Lithium hexafluorophosphate was isolated. Purity was assessed by ¹⁹F and ⁷Li NMR. Lithium salts and the associated yield of lithium hexafluorophosphate is provided below in Table 10. An exemplary reaction scheme is provided in FIG. 10.

Table 10

Solvent	Lithium salt	Yield %	Purity %
Acetonitrile	LiCl	80	60
Acetonitrile	LiBr	0	-
Acetonitrile	LiClO ₄	5	50
PC/DMC	LiBF ₄	90	95

Example 10B: Preparation of battery electrolyte LiPF₆ (MeLi additive)

[0194] Reactions were run to assess whether the addition of alkyl lithium to the reaction for preparing battery electrolyte had an effect on yield. In a 2-necked flask oven dried under nitrogen with stirring bar, lithium chloride (1-2 equiv.) was weighed in with metal hexafluorophosphate and the solid mixture dried under vacuum. THF was added and the suspension stirred. After the required time, the mixture was cooled down to 0 °C and Methyl Lithium was added (0.024 equiv.) with strong stirring. The mixture was then filtered and evaporated under vacuum. The solid evaporated to dryness overnight. Lithium hexafluorophosphate was isolated. Reaction conditions are provided below in Table 11 and an exemplary reaction scheme is provided in FIG. 11.

[0195] As shown below, the addition of methyl lithium showed improved yields, but an otherwise equivalent reaction worked without addition of methyl lithium as showed in Example 10A and 10B.

Table 11

Solvent	Time [h]	Concentration [vol.]	Yield %	Purity %
THF	1	4	90	+98
THF	18	4	90	98
THF	18	10	75	95
THF	18	40	60	91
Dioxane	18	20	0	-

Example 11: Preparation of battery electrolyte NaPF₆

[0196] In a 50 ml 2-necked flask oven dried under nitrogen with stirring bar, sodium chloride was weighed in and suspended in the appropriate solvent, the suspension was stirred for 40 minutes. Metal hexafluorophosphate (1 equiv.) was added as a solid in one portion and the suspension left stirring for 18 h at room temperature. The suspension was concentrated under nitrogen and filtered. The solid evaporated to dryness overnight. Sodium hexafluorophosphate was isolated. Purity was assessed by ¹⁹F-NMR. Yield of an exemplary reaction is provided in Table 12 below and an exemplary reaction scheme is provided in FIG. 12.

Table 12

Yield %	Purity %
73	95

Example 12: Preparation of a Sodium Hexafluorophosphate Precursor

[0197] Calcium fluoride and sodium phosphate were stirred in water and heated at 100 °C for 18 h. The mixture was allowed to cool to room temperature and the solid were removed by

centrifugation. The solution was acidified with phosphoric acid to pH=6. After this time, the water was evaporated to leave a solid. The solid (from the workup) was suspended in Methanol (500 mL) and allowed to stir at 60 °C, for 2h whilst stirring. The mixture was cooled to room temperature and the solution filtered. The resulting filtrate was concentrated under vacuum to afford the product (Fluorination reagent D).

[0198] To an oven dried flask with stirring device was added anhydrous acetonitrile under inert atmosphere and Fluorinating Reagent D was added under constant inert gas flow (e.g., as illustrated in **FIG. 14**). The mixture was then cooled to 0 °C whilst stirring and phosphorus pentachloride (5 g) was added. Upon complete addition, the reaction was allowed to stir at 0 °C for 2h. The mixture was diluted with acetonitrile and then filtered to obtain sodium hexafluorophosphate (99% yield , 98% purity).

Example 13: Mechanochemical conditions for providing a battery electrolyte precursor

[0199] Fluorination reagent C (6-12 eq) and a starting reagent are combined under mechanochemical conditions (milled for 2 hours at 35 Hz) to provide a battery electrolyte precursor. Reagents and reaction conditions are provided in Table 4. An exemplary reaction scheme is provided in **FIG. 15**.

CLAIMS

WHAT IS CLAIMED IS:

1. A method of manufacturing a battery electrolyte precursor, the method comprising:
 - (a) providing a fluorination reagent, wherein the fluorination reagent comprises a first salt and a second salt, the first salt comprising calcium and fluorine;
 - (b) contacting a starting reagent with the fluorination reagent to provide a battery electrolyte precursor.
2. The method of any one of the preceding claims, wherein the second salt comprises an anion, wherein the anion, when combined with Ca^{2+} to form a third salt, has a lattice energy greater than 2450 KJ/mol.
3. The method of any one of the preceding claims, wherein a powder x-ray diffraction spectrum of the fluorination reagent comprises characteristic 2θ reflections at about 21.9° , 30.3° , 31.6° , 43.4° and/or combinations thereof.
4. The method of any one of the preceding claims, wherein a powder x-ray diffraction spectrum of the fluorination reagent comprises characteristic 2θ reflections at about 28.1° , 49.0° , 52.3° , 54.1° , 60.0° , 69.7° and/or combinations thereof.
5. A method of manufacturing a battery electrolyte precursor, the method comprising:
 - (a) combining a first salt with a second salt to form a mixed composition, the first salt comprising calcium and fluoride;
 - (b) subjecting the mixed composition with a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
 - (c) concentrating the resultant fluid to produce a crude fluorination reagent (e.g., a wash concentrate or precipitate);
 - (d) washing the crude fluorination reagent with a solvent (e.g., an alcohol) to produce a reagent wash (e.g., a second solid component and fluid reagent wash); and
 - (e) concentrating the reagent wash to form a purified fluorination reagent (e.g., the purified fluorination reagent having a higher concentration of fluorine compared to the crude fluorination reagent).
 - (f) contacting a starting reagent with the purified fluorination reagent to provide a battery electrolyte precursor.
6. A method of manufacturing a battery electrolyte precursor, the method comprising:
 - (a) combining a first salt with a second salt, the first salt comprising calcium and fluoride;
 - (b) applying mechanical force to the combination of the first salt and the second salt to form a mixed composition;

- (c) subjecting the mixed composition with a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
 - (d) concentrating the resultant fluid to produce a crude fluorination reagent (e.g., a wash concentrate or precipitate);
 - (e) washing the crude fluorination reagent with a solvent (e.g., an alcohol) to produce a reagent wash (a second solid component and fluid reagent wash); and
 - (f) concentrating the reagent wash to form a purified fluorination reagent (e.g., the purified fluorination reagent having a higher concentration of fluorine compared to the crude fluorination reagent).
 - (g) contacting a starting reagent with the purified fluorination reagent to provide a battery electrolyte precursor.
7. A method of manufacturing a purified fluorination reagent, the method comprising:
- (a) combining a first salt with a second salt to form a mixed composition, the first salt comprising calcium and fluoride;
 - (b) subjecting the mixed composition to a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
 - (c) concentrating the resultant fluid to produce a purified fluorination reagent (e.g., a reagent concentrate or precipitate); and
 - (d) contacting a starting reagent with the purified fluorination reagent to provide a battery electrolyte precursor.
8. A method of manufacturing a purified fluorination reagent, the method comprising:
- (a) combining a first salt with a second salt, the first salt comprising calcium and fluoride;
 - (b) applying mechanical force to the combination of the first salt and the second salt to form a mixed composition;
 - (c) subjecting the mixed composition to a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
 - (d) concentrating the resultant fluid to produce a purified fluorination reagent (e.g., a reagent concentrate or precipitate); and
 - (e) contacting a starting reagent with the purified fluorination reagent to provide a battery electrolyte precursor.
9. The method of any one of the preceding claims, wherein the fluorination reagent and/or purified fluorination reagent is contacted with the starting reagent in an alkyl carbonate solvent (e.g., dimethyl carbonate).

10. The method of any one of the preceding claims, wherein the alkyl carbonate solvent is dimethyl carbonate, diethyl carbonate, dipropyl carbonate, methyl ethyl carbonate, methyl propyl carbonate, ethyl propyl carbonate, and/or combinations thereof.
11. The method of any one of the preceding claims, wherein the alkyl carbonate solvent is a fluoroalkyl carbonate solvent (e.g., trifluoroethyl carbonate, bis(trifluoroethyl) carbonate, trifluoroethyl methyl carbonate, and/or combinations thereof).
12. The method of any one of the preceding claims, wherein a combination of the fluorination reagent and/or purified fluorination reagent, the alkyl carbonate solvent, and the starting reagent is at a temperature of about 50 to about 150 °C.
13. The method of any one of the preceding claims, wherein a combination of the fluorination reagent and/or purified fluorination reagent, the alkyl carbonate solvent, and a starting reagent is at a temperature of about 80 °C about or more (e.g., about 100 °C or more).
14. A method of manufacturing a battery electrolyte precursor, the method comprising:
 - (a) providing a purified fluorination reagent, wherein the purified fluorination reagent comprises an alkali metal comprising lithium, potassium, or sodium, fluoride, and at least one additional ion;
 - (b) contacting a starting reagent with the purified fluorination reagent to provide a battery electrolyte precursor.
15. A method of manufacturing a purified fluorination reagent, the method comprising:
 - (a) combining a first salt with a second salt to form a mixed composition, the first salt comprising calcium and fluoride;
 - (b) subjecting the mixed composition with a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
 - (c) concentrating the resultant fluid to produce a crude fluorination reagent (e.g., a wash concentrate or precipitate);
 - (d) washing the crude fluorination reagent with a solvent (e.g., an alcohol) to produce a reagent wash (a second solid component and fluid reagent wash); and
 - (e) concentrating the reagent wash to form a purified fluorination reagent (e.g., the purified fluorination reagent having a higher concentration of fluorine compared to the crude fluorination reagent).
16. A method of manufacturing a purified fluorination reagent, the method comprising:
 - (a) combining a first salt with a second salt, the first salt comprising calcium and fluoride;
 - (b) applying mechanical force to the combination of the first salt and the second salt to form a mixed composition;

- (c) subjecting the mixed composition with a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
 - (d) concentrating the resultant fluid to produce a crude fluorination reagent (e.g., a wash concentrate or precipitate);
 - (e) washing the crude fluorination reagent with a solvent (e.g., an alcohol) to produce a reagent wash (a second solid component and fluid reagent wash); and
 - (f) concentrating the reagent wash to form a purified fluorination reagent (e.g., the purified fluorination reagent having a higher concentration of fluorine compared to the crude fluorination reagent).
17. A method of manufacturing a purified fluorination reagent, the method comprising:
- (a) combining a first salt with a second salt to form a mixed composition, the first salt comprising calcium and fluoride;
 - (b) subjecting the mixed composition with a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
 - (c) concentrating the resultant fluid to produce a purified fluorination reagent (e.g., a wash concentrate or precipitate).
18. A method of manufacturing a purified fluorination reagent, the method comprising:
- (a) combining a first salt with a second salt, the first salt comprising calcium and fluoride;
 - (b) applying mechanical force to the combination of the first salt and the second salt to form a mixed composition;
 - (c) subjecting the mixed composition with a fluid composition (to produce a solid component and a resultant fluid) and collecting a resultant fluid thereof;
 - (d) concentrating the resultant fluid to produce a purified fluorination reagent (e.g., a wash concentrate or precipitate).
19. A method of manufacturing a battery electrolyte precursor, the method comprising:
- (1) providing a fluorination reagent, wherein the fluorination reagent comprises calcium and fluorine;
 - (2) contacting a starting reagent with the fluorination reagent to provide a battery electrolyte precursor.
20. The method of any one of the preceding claims, further comprising adjusting the pH of the resultant fluid (e.g., to a pH of about 6 to about 8) prior to concentrating the resultant fluid.
21. The method of any one of the preceding claims, wherein the pH of the resultant fluid is adjusted with an acid (e.g., strong acid, weak acid, polyprotic acid, and/or combinations thereof).

22. The method of any one of the preceding claims wherein the acid comprises phosphoric acid, hydrochloric acid, formic acid, acetic acid, benzoic acid, boric acid, silicic acid, oxalic acid, sulfuric acid, sulfurous acid, carbonic acid, and/or combinations thereof.
23. The method of any one of the preceding claims, wherein the acid comprises hydrochloric acid, phosphoric acid, sulfuric acid, and/or combinations thereof.
24. The method of any one of the preceding claims, wherein the pH of the resultant fluid is adjusted to a pH of about 5 to about 10 (e.g., about 6 to about 9).
25. The method of any one of the preceding claims, wherein the fluid composition has a pH of about 7 or more (e.g., about 10 or more).
26. The method of any one of the preceding claims, wherein the fluid composition has a pH of about 12 to about 13.
27. The method of any one of the preceding claims, wherein a combination of the fluid composition and the mixed composition is at a temperature of about 0 to about 120 °C.
28. The method of any one of the preceding claims, wherein a combination of the fluid composition and the mixed composition is at a temperature of 80 °C or more.
29. The method of any one of the preceding claims, wherein a combination of the fluid composition and the mixed composition is at a temperature of 110 °C or less.
30. The method of any one of the preceding claims, wherein the mixed composition is subjected to the fluid composition for about 0 hours to about 8 hours.
31. The method of any one of the preceding claims, wherein the mixed composition is subjected to the fluid composition for about 1 hour or more.
32. The method of any one of the preceding claims, wherein the mixed composition is subjected to the fluid composition for about 6 hours or less.
33. The method of any one of the preceding claims, wherein the mixed composition is subjected to the fluid composition for about 2 hours.
34. The method of any one of the preceding claims, wherein the fluid composition has a boiling point of about 30 °C or more (e.g., about 70 °C or more, about 120 °C or more).
35. The method of any one of the preceding claims, wherein the fluid composition has a boiling point of about 240 °C or less.
36. The method of any one of the preceding claims, wherein a combination of the solvent and the crude fluorination reagent is at a temperature of about -20 to about 240 °C.
37. The method of any one of the preceding claims, wherein a combination of the solvent and the crude fluorination reagent is at a temperature of about 80 °C or more.
38. The method of any one of the preceding claims, wherein a combination of the solvent and the crude fluorination reagent is at a temperature of about 60 °C.

39. The method of any one of the preceding claims, wherein a combination of the solvent and the crude fluorination reagent is at a temperature of about 235 °C or less.
40. The method of any one of the preceding claims, wherein the crude fluorination reagent is washed with the solvent for about 4 hours to about 48 hours (e.g., about 8 hours to about 36 hours, about 10 hours to about 28 hours).
41. The method of any one of the preceding claims, wherein the crude fluorination reagent is washed with the solvent for about 8 hours or more.
42. The method of any one of the preceding claims, wherein the crude fluorination reagent is washed with the solvent for about 36 hours or less.
43. The method of any one of the preceding claims, wherein the crude fluorination reagent is washed with the solvent for about 18 hours.
44. The method of any one of the preceding claims, wherein the solvent has a boiling point of about 30 °C or more (e.g., about 70 °C or more, about 120 °C or more).
45. The method of any one of the preceding claims, wherein the solvent has a boiling point of about 240 °C or less.
46. The method of any one of the preceding claims, wherein the solvent and/or the fluid composition is an organic solvent, water, an alcohol, a polar aprotic solvent, a halocarbon, and/or combinations thereof.
47. The method of any one of the preceding claims, wherein the solvent and/or the fluid composition is acetonitrile, propionitrile, butyronitrile, toluene, 1,2-dichlorobenzene, chlorobenzene, fluorobenzene, 1,2-difluorobenzene, dichloroethane, trifluorotoluene, chloroform, DMF, DMSO, sulfolane, MeTHF, THF, NMP, pyridine, butyl acetate, dioxane, an alcohol (e.g., tert-butanol, tert-amyl alcohol), water, and/or combinations thereof.
48. The method of any one of the preceding claims, wherein the solvent and/or the fluid composition is acetonitrile, propionitrile, butyronitrile, and/or combinations thereof.
49. The method of any one of the preceding claims, further comprising providing the mixed composition subjected to the fluid composition as the first salt.
50. The method of any one of the preceding claims, wherein the first salt is a recovered waste material.
51. The method of any one of the preceding claims wherein the first salt comprises low purity calcium and fluoride (e.g., less than 80 weight percent in total is calcium and fluorine).
52. The method of any one of the preceding claims, wherein the first salt is CaF_2 or $\text{Ca}_5(\text{PO}_4)_3\text{F}$.

53. The method of any one of the preceding claims, wherein the second salt is a metal hydroxide, a metal sulphite, a metal sulphate, a carbonate, or an inorganic phosphate (e.g., a pyrophosphate).
54. The method of any one of the preceding claims, wherein the second salt comprises NaOH, KOH, Na₂SO₃, K₂SO₃, KHSO₄, CaCO₃, H₂CO₃, K₂CO₃, Na₂CO₃, K₄P₂O₇, Na₄P₂O₇, Na₃PO₄, Li₃PO₄, KHCO₃, K₂CO₃, NaHCO₃, Cs₂CO₃, K₂HPO₄, KH₂PO₄, K₃PO₄, KPO₃, K₅P₃O₁₀, K₂SO₄, titanium phosphate, aluminum phosphate, uranium phosphate, and/or combinations thereof.
55. The method of any one of the preceding claims, wherein a combination of the fluorination reagent and/or purified fluorination reagent and the starting reagent comprises a reaction mixture.
56. The method of any one of the preceding claims, wherein the reaction mixture is at a temperature of about -5 to about 120 °C.
57. The method of any one of the preceding claims, wherein the reaction mixture is at a temperature of about 55 to about 150 °C.
58. The method of any one of the preceding claims, wherein the reaction mixture is at a temperature of about 100 °C or less.
59. The method of any one of the preceding claims, wherein the fluorination reagent and/or the purified fluorination reagent is contacted with starting reagent for about 0.5 hours to about 40 hours.
60. The method of any one of the preceding claims, wherein the fluorination reagent and/or the purified fluorination reagent is contacted with starting reagent for about 12 hours or more.
61. The method of any one of the preceding claims, wherein the fluorination reagent and/or the purified fluorination reagent is contacted with the starting reagent for about 14 hours to about 22 hours.
62. The method of any one of the preceding claims, wherein the fluorination reagent and/or the purified fluorination reagent is contacted with the starting reagent for about 84 hours or less.
63. The method of any one of the preceding claims, wherein the fluorination reagent and/or the purified fluorination reagent is contacted with the starting reagent for about 60 hours to about 80 hours.
64. The method of any one of the preceding claims, wherein the reaction mixture further comprises a phase transfer agent, a base, and/or combinations thereof.

65. The method of any one of the preceding claims, wherein the phase transfer agent is a crown ether (e.g., 18 crown 6), a cryptand, an ionic transfer agent (e.g., tetramethylammonium chloride), and/or a hydrogen-bonding phase transfer agent.
66. The method of any one of the preceding claims, wherein the phase transfer agent is a crown ether (e.g., 18 crown 6).
67. The method of any one of the preceding claims, wherein the base is a pyridine derivative (e.g., DMAP).
68. The method of any one of the preceding claims, wherein the reaction mixture further comprises a reaction solvent.
69. The method of any one of the preceding claims, wherein the reaction solvent is an organic solvent, water, an alcohol, a polar aprotic solvent, a halocarbon, and/or combinations thereof.
70. The method of any one of the preceding claims, wherein the reaction solvent is acetonitrile, propionitrile, pyridine, butyronitrile, toluene, 1,2-dichlorobenzene, chlorobenzene, fluorobenzene, 1,2-difluorobenzene, dichloroethane, trifluorotoluene, chloroform, DMF, DMSO, an alcohol (e.g., tert-butanol, tert-amyl alcohol), water, and/or combinations thereof.
71. The method of any one of the preceding claims, wherein the reaction solvent is acetonitrile, propionitrile, pyridine, butyronitrile, and/or combinations thereof.
72. The method of any one of the preceding claims, wherein the reaction solvent is acetonitrile, chloroform, sulfolane, THF, NMP, and/or combinations thereof.
73. The method of any one of the preceding claims, wherein the starting reagent comprises a leaving group.
74. The method of any one of the preceding claims, wherein the leaving group is chlorine, iodine, or bromine.
75. The method of any one of the preceding claims, wherein the starting reagent is PCl_3 , PCl_5 , PF_5 , PCl_6 , or LiPCl_6 .
76. The method of any one of the preceding claims, wherein the starting reagent is PCl_5 .
77. The method of any one of the preceding claims, wherein the battery electrolyte precursor comprises at least one additional fluorine (e.g., at least two additional fluorine) compared to the starting reagent.
78. The method of any one of the preceding claims, wherein the battery electrolyte precursor is PF_3 , PF_5 , KPF_6 , or salts thereof.
79. The method of any one of the preceding claims, wherein the battery electrolyte precursor is NaPF_6 , PF_3 , PF_5 , KPF_6 , or salts thereof.

80. The method of any one of the preceding claims, wherein an amount of phosphorous in the purified fluorination reagent is about 0.015 % to about 12.5 % by weight (wt %).
81. The method of any one of the preceding claims, wherein an amount of calcium in the purified fluorination reagent is about 0.01 % to about 15 % by weight (wt %).
82. The method of any one of the preceding claims, wherein an amount of phosphorous in the purified fluorination reagent is about 0.02 % to about 10 % by weight (wt %) (e.g., about 0.05 wt % to about 8 wt %, about 0.1 wt % to about 6 wt %, about 0.5 wt % to about 5 wt %, about 1 wt % to about 4 wt %), or wherein an amount phosphorous in the purified fluorination reagent is about 1 ppm to about 25 ppm (e.g., about 1 ppm, about 10 ppm, about 20 ppm, or about 25 ppm).
83. The method of any one of the preceding claims, wherein an amount of phosphorous in the purified fluorination reagent is about 0.015 % by weight (wt %) or more (e.g., about 0.05 wt % or more, about 0.1 wt % or more, about 0.5 wt % or more).
84. The method of any one of the preceding claims, wherein an amount of phosphorous in the purified fluorination reagent is about 5 % by weight (wt %) or less (e.g., about 3 wt % or less, about 2 wt % or less, about 1 wt % or less, about 0.5 wt % or less, about 0.1 wt % or less, about 0.05 wt % or less).
85. The method of any one of the preceding claims, wherein a powder x-ray diffraction spectrum of the crude fluorination reagent comprises characteristic 2θ reflections at about 5.2° , 31.5° , 36.8° and/or combinations thereof.
86. The method of any one of the preceding claims, wherein the at least one additional ion of the purified fluorination reagent comprises (i) at least one cation and at least one anion; or (ii) at least one zwitterion (e.g., psilocybin).
87. The method of any one of the preceding claims, wherein the at least one cation comprises K^+ , Na^+ , Ca^{2+} , Li^+ , or Cs^+ .
88. The method of any one of the preceding claims, wherein the at least one anion comprises a hydroxide, a sulphate, a carbonate, a phosphate, a pyrophosphate.
89. The method of any one of the preceding claims, wherein a molar ratio of the phase transfer agent to the starting reagent is about 0 to about 4.
90. The method of any one of the preceding claims, wherein a molar ratio of the base to the starting reagent is about 0 to about 2.
91. The method of any one of the preceding claims, wherein a molar ratio of a fluorine equivalent content in the fluorination reagent and/or purified fluorination reagent to the starting reagent is about 0.1 or more.

92. The method of any one of the preceding claims, wherein a yield of the battery electrolyte precursor is about 10% or more.
93. The method of any one of the preceding claims, wherein a yield of the battery electrolyte precursor is about 20% to about 80%.
94. The method of any one of the preceding claims, wherein a yield of the battery electrolyte precursor is about 75% to about 95%.
95. The method of any one of the preceding claims, wherein a concentration of the starting reagent in the reaction solvent and/or alkyl carbonate solvent is about 0.01 M to about 3 M.
96. The method of any one of the preceding claims, wherein a concentration of the starting reagent in the reaction solvent and/or alkyl carbonate solvent is about 1 M or less.
97. The method of any one of the preceding claims, wherein the fluorination reagent and/or purified fluorination reagent is contacted with the starting reagent under mechanochemical conditions (e.g., ball mill).
98. The method of any one of the preceding claims, wherein the battery electrolyte precursor is contacted with an electrolyte agent (e.g., lithium salt) to provide a battery electrolyte.
99. The method of any one of the preceding claims, wherein the electrolyte agent is a lithium salt or a sodium salt.
100. The method of any one of the preceding claims, wherein the electrolyte agent is lithium perchlorate, lithium sulphate, lithium chloride, lithium bromide, lithium tetrafluoroborate, or a combination of two or more thereof.
101. The method of any one of the preceding claims, wherein the electrolyte agent is NaCl or NaClO₄.
102. The method of any one of the preceding claims, wherein the starting reagent is PCl₃, PCl₅, PF₅, LiPCl₆, P₄O₆, P₂O₅.
103. The method of any one of the preceding claims, wherein the starting reagent is PCl₅.
104. The method of any one of the preceding claims, wherein the battery electrolyte is LiPF₆.
105. The method of any one of the preceding claims, wherein the battery electrolyte is NaPF₆.
106. The method of any one of the preceding claims, wherein the battery electrolyte precursor is contacted with the electrolyte agent in a selected solvent selected from acetonitrile, acetone, THF, ethanol, methanol, dioxane, methyl t-butyl ether, diethyl ether, MEK, dimethyl carbonate, and propylene carbonate, and/or combinations of one or more thereof.
107. The method of any one of the preceding claims, wherein the battery electrolyte precursor is contacted with the electrolyte agent for about 1 hour to about 84 hours (e.g., about 2 hours to about 76 hours, about 3 hours to about 24 hours, about 4 hours to about 12 hours, about 5 hours to about 10 hours).

108. The method of any one of the preceding claims, wherein the battery electrolyte precursor is contacted with the electrolyte agent for about 30 hours or less (e.g., about 24 hours or less, about 18 hours or less, about 12 hours or less, about 8 hours or less).
109. The method of any one of the preceding claims, wherein the battery electrolyte precursor is contacted with the electrolyte agent for about 2 hours or more (e.g., about 4 hours or more, about 8 hours or more, about 16 hours or more).
110. The method of any one of the preceding claims, wherein the battery electrolyte precursor is contacted with the electrolyte agent for about 18 hours.
111. The method of any one of the preceding claims, wherein a combination of the battery electrolyte precursor and the electrolyte agent is at a temperature of about 10 to about 80 °C (e.g., about 20 to about 70 °C, about 30 to about 60 °C).
112. The method of any one of the preceding claims, wherein a combination of the battery electrolyte precursor and the electrolyte agent is at a temperature of about 10 °C or more (e.g., about 15 °C or more, about 20 °C or more).
113. The method of any one of the preceding claims, wherein a combination of the battery electrolyte precursor and the electrolyte agent is at a temperature of about 60 °C or less (e.g., about 50°C or less, about 40°C or less, about 30°C or less).
114. The method of any one of the preceding claims, wherein a combination of the battery electrolyte precursor and the electrolyte agent is at room temperature.

FIG. 1

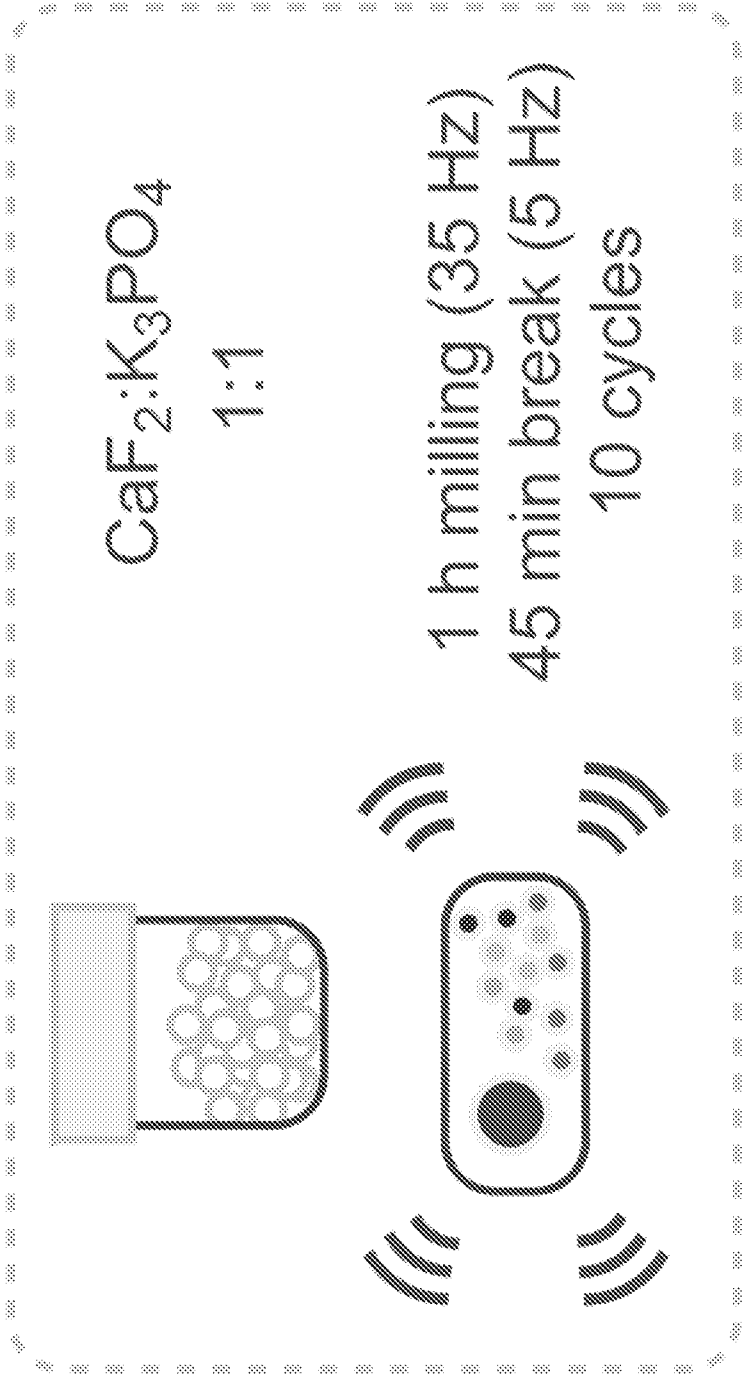


FIG. 2

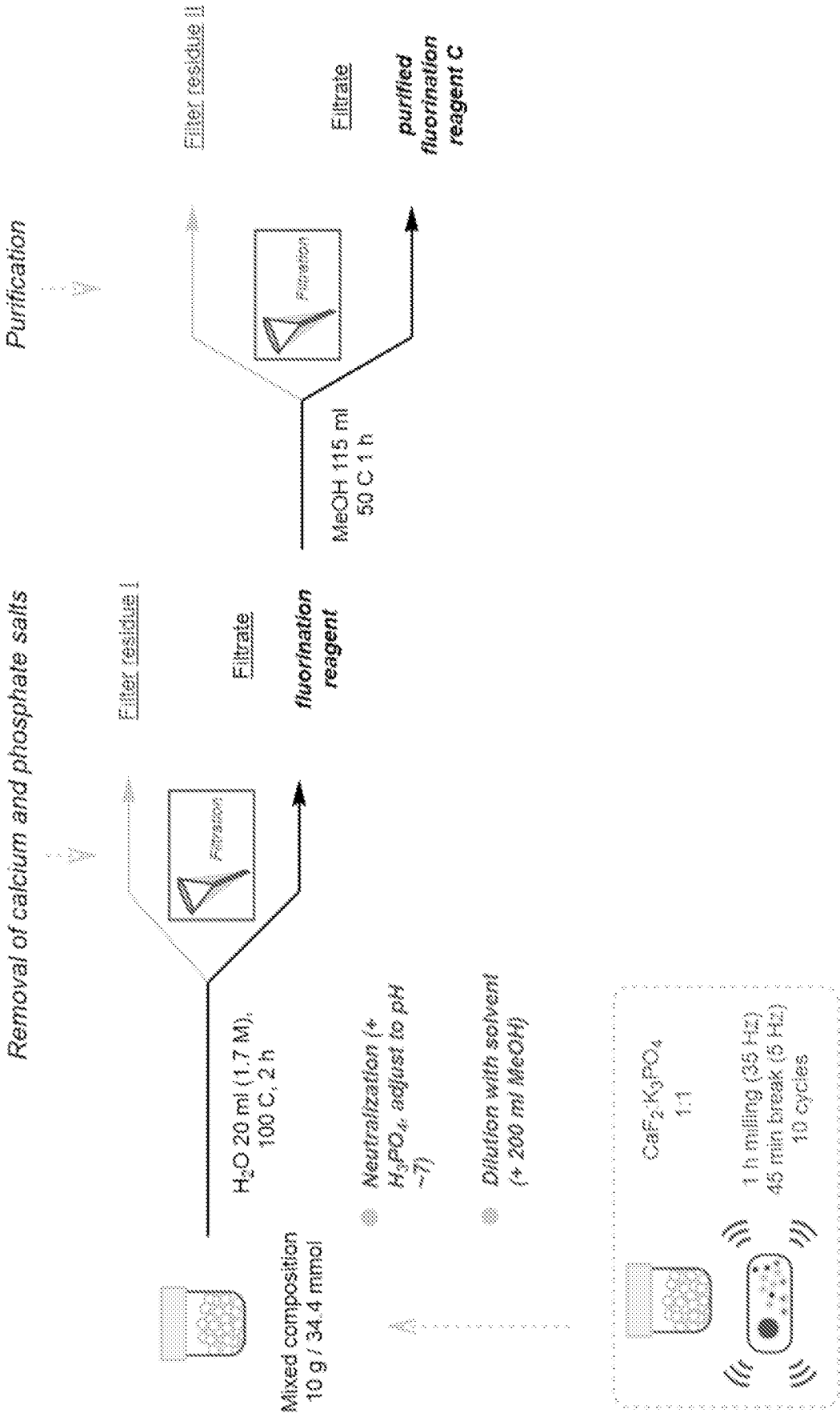


FIG. 3

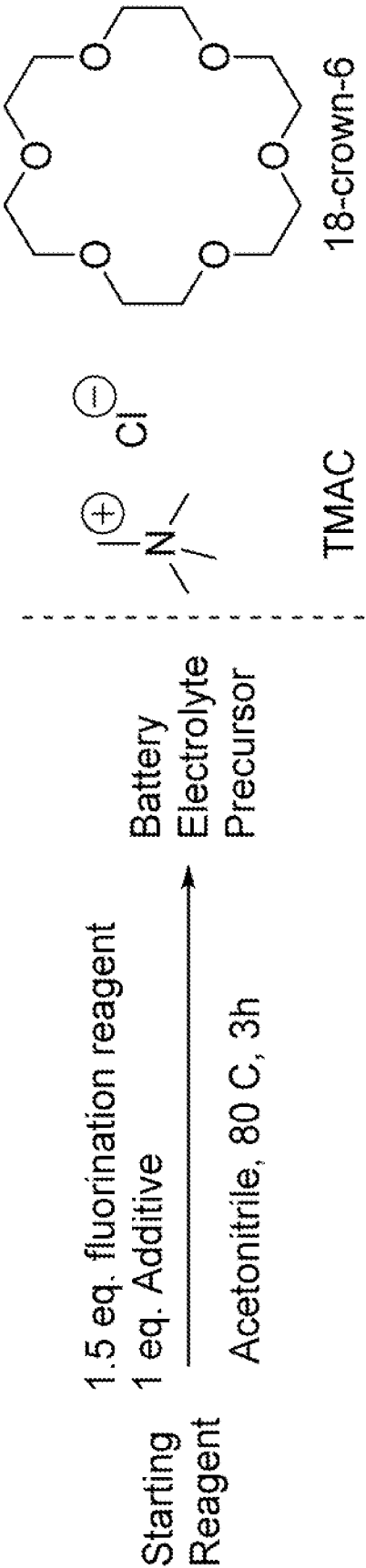


FIG. 4

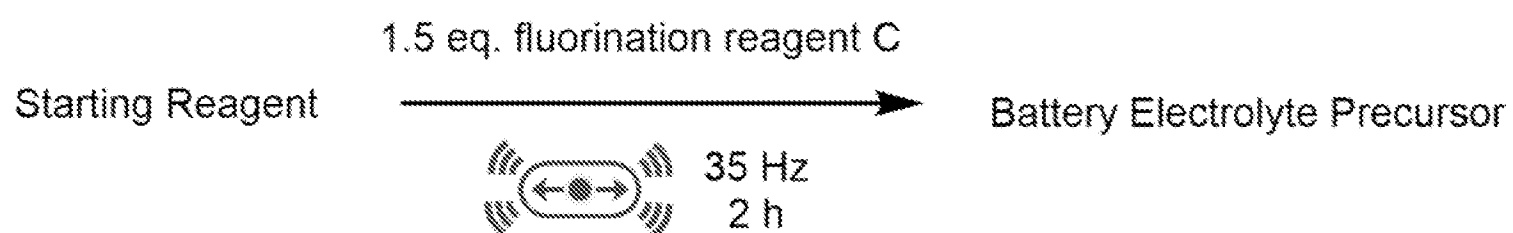


FIG. 5

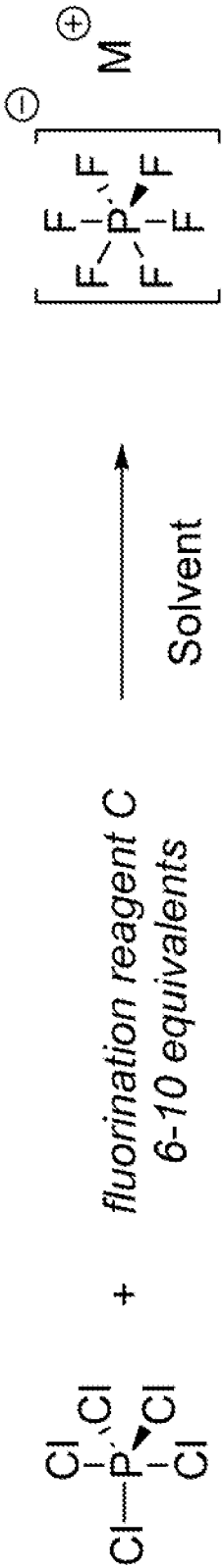


FIG. 6

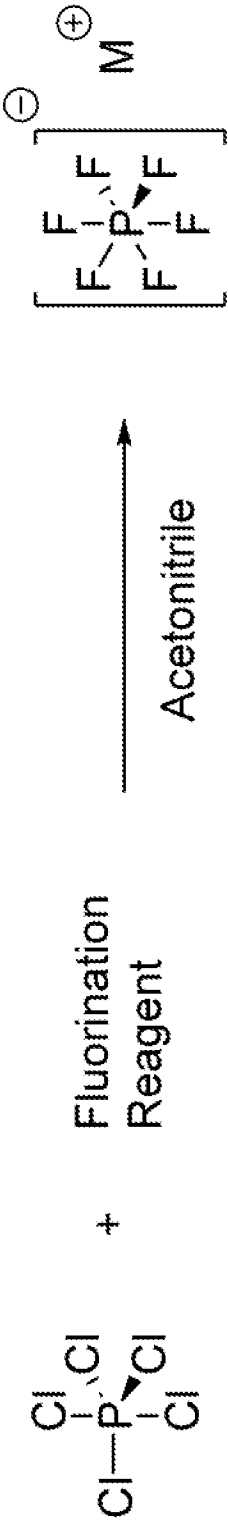


FIG. 7

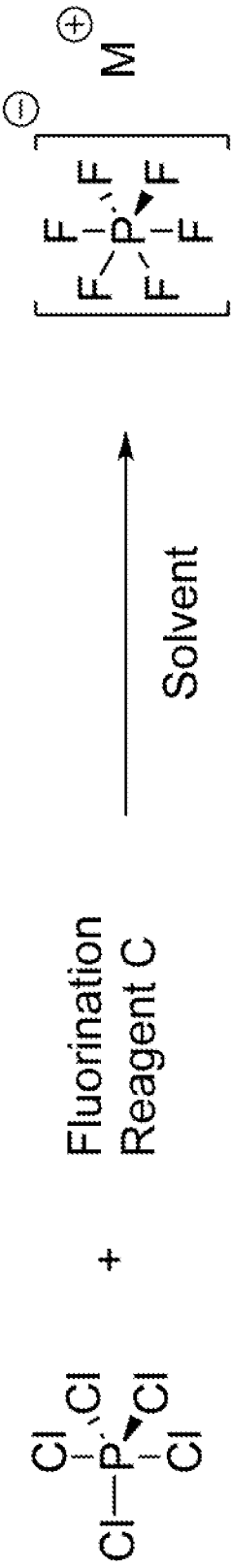


FIG. 8



FIG. 9

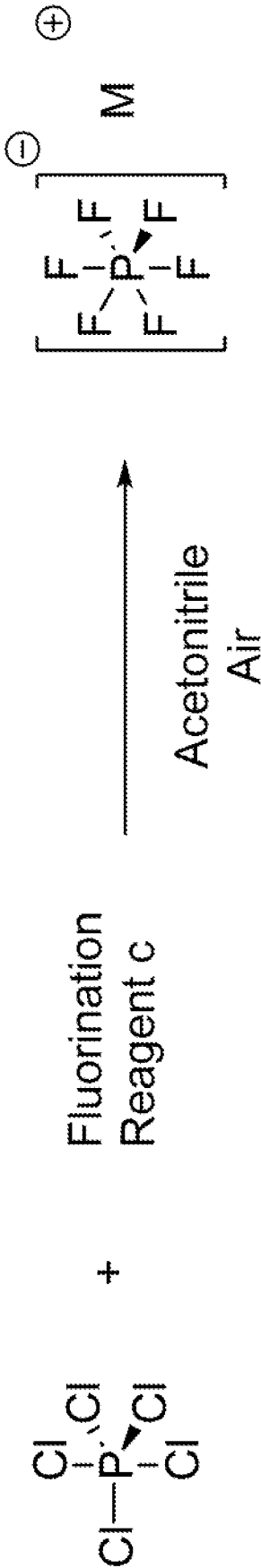


FIG. 10

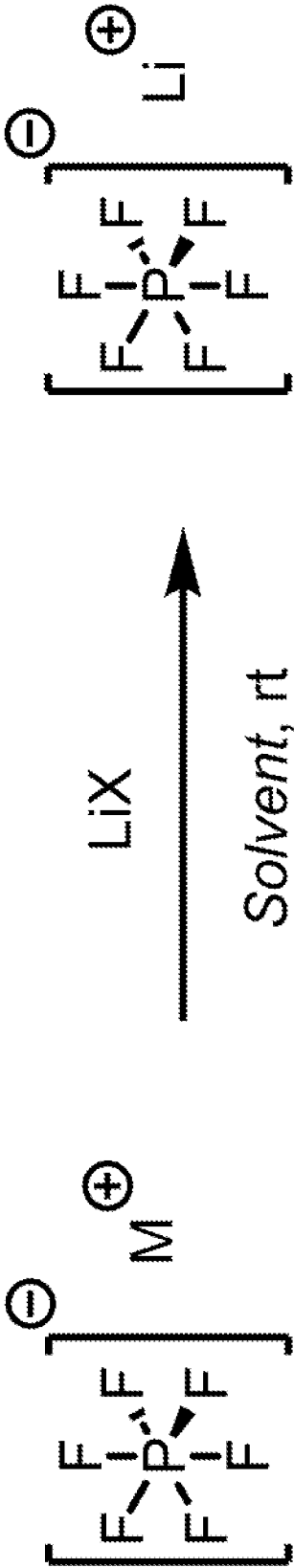


FIG. 11

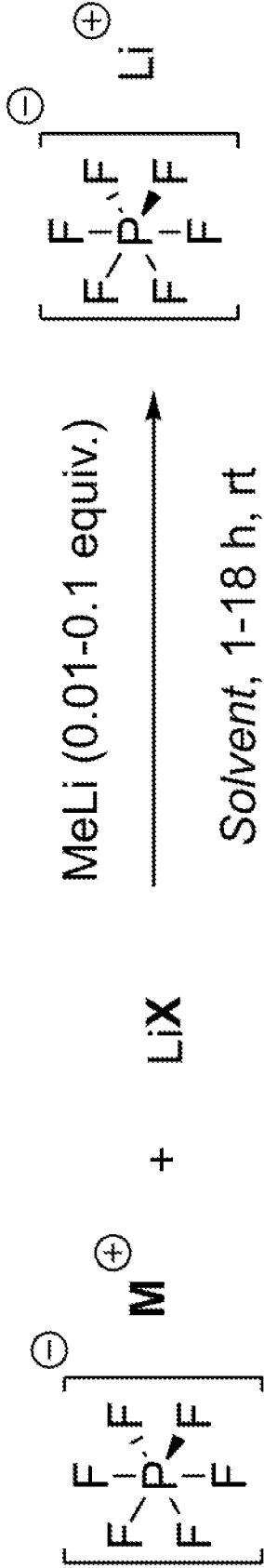


FIG. 12

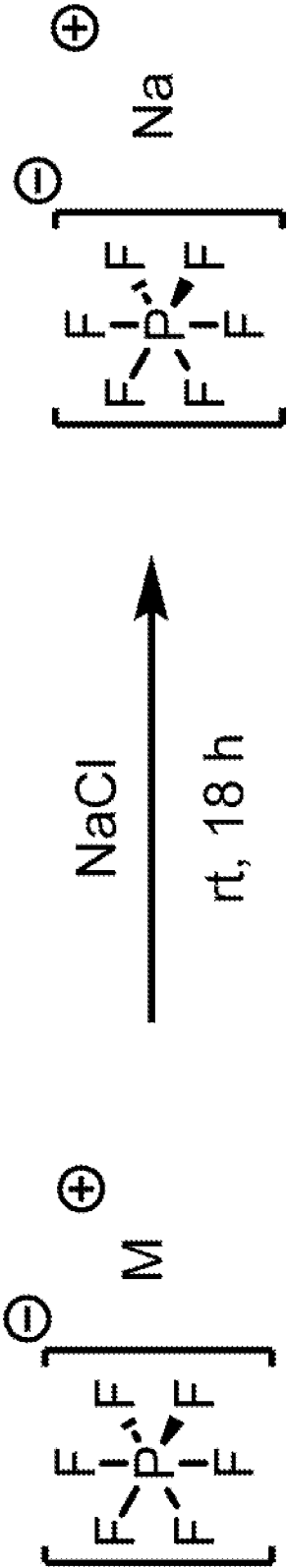


FIG. 13

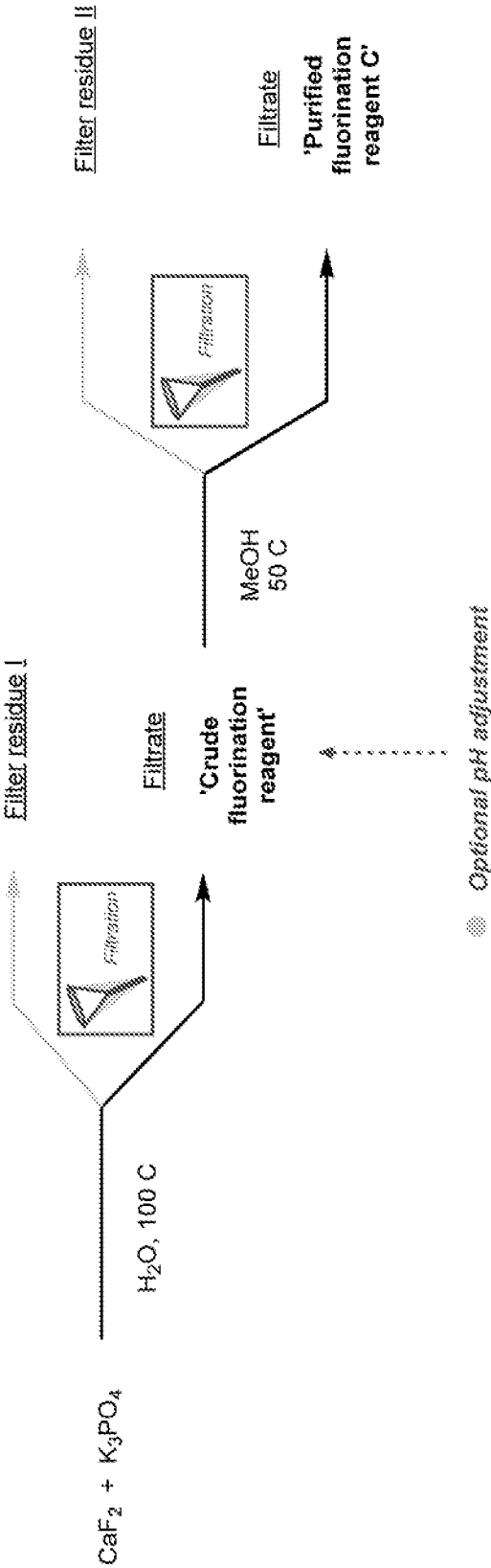


FIG. 14

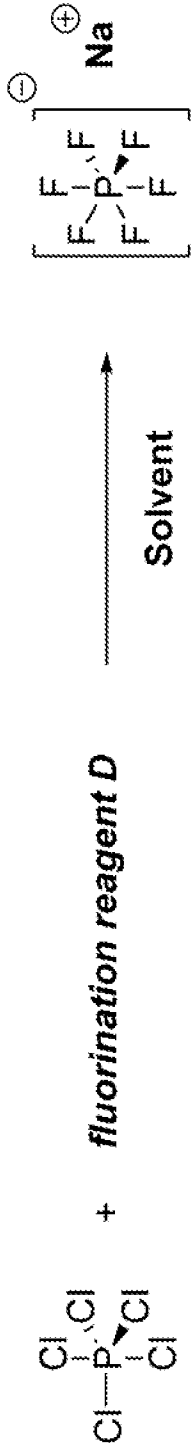


FIG. 15

