Chapter 1
Nonaqueous Electrolytes: Advances in Lithium Salts

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Abstract The synthesis and characterization of new lithium salts has been a core component of electrolyte research for the past three decades. Upon the commercialization of Li-ion batteries with a graphite anode, $LiPF_6$ became the dominant salt for lithium battery electrolytes. But the advent of new electrodes/cell chemistries (e.g., Si alloy anodes, high-voltage cathodes, Li-air, Li-S), as well as the need for exceptional battery safety, higher/lower temperature operation, improved durability/longer lifetimes, etc., has resulted in the pressing need for new electrolyte formulations. Lithium salts, either as a substitute for $LiPF_6$ or as an additive, are one central focus for this electrolyte transformation.

Keywords Li-ion batteries • Electrolyte • Salts • Solvation • Ionic association

1.1 Introduction

Early lithium battery electrolyte research in the 1970s used available lithium salts—i.e., principally $LiClO_4$, $LiAlCl_4$, $LiBF_4$, $LiPF_6$, and $LiAsF_6$. Efforts at the time were devoted to stabilizing the stripping/plating of Li metal [1–3], as well as the use of intercalation electrodes [4]. $LiPF_6$ was reported to not provide the best Li metal stripping/plating efficiency amongst the salts studied [5, 6] and $LiPF_6$ was also found initially to be problematic for the cycling of carbon electrodes [7, 8]. $LiPF_6$ did not become the paramount salt for lithium battery electrolytes until carbon coke replaced Li metal (later to be replaced by graphite) and the solvents were optimized for the electrolyte utilized in the Li-ion batteries initially commercialized by Sony in 1991 [9–19].

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Many/most of the anions used for electrolyte lithium salts were initially developed in efforts to generate stronger superacids. The term superacid generally refers to acids which are more acidic than mineral Brønsted acids [20–27]. In particular, a widely used definition for superacids was given by Gillespie who defined this term to be applicable to acids which are more acidic than sulfuric acid [28–30]. The stronger the acidity, the weaker the coordination of the anions is with the associated protons (H^+ cations). The gas-phase acidity is given by:

$$\mathrm{HA} \overset{K_\mathrm{a}}{\rightleftarrows} \mathrm{A}^- + \mathrm{H}^+$$

and

$$\Delta G_\mathrm{acid} = -RT \ln K_\mathrm{a}$$

In practice, however, the determination of the acidity of superacid anions in solution is not a trivial matter as the acidity of a given acid (HA) is a function of the solvent (S) used:

$$\mathrm{HA} + \mathrm{S} \overset{K_\mathrm{a}}{\rightleftarrows} \mathrm{A}^- + \mathrm{SH}^+$$

which is influenced by factors such as the steric bulk of the anions and solvent, ion pairing, etc. Three major substitution effects contribute to increasing the acidity of a neutral acid functional group (C–H, O–H, N–H, S–H, B–H, etc.) [20, 26]:

1. field/inductive effects
2. π-electron acceptor (resonance) and negative hyperconjugation effects
3. substituent polarization effects

The influence of these factors is evident from the acidity variations noted for different substituents (Figs. 1.1 and 1.2) [20, 26]. No simple patterns in acidity behavior are found for these effects, however, due to the interplay between them (i.e., these effects do not operate independently of one another) [20, 26]. For $=\mathrm{Z(X)}_n$ substituents (replacing an =O: $\mathrm{M{=}O} \rightarrow \mathrm{M{=}Z(X)}_n$, where M=O might be $CH_3C(=O)H$, $HC(=O)NH_2$, HC(=O)OH, etc.), the acidification for the same X increases with increasing *n* [22]:

$$= \mathrm{N(CN)} <= \mathrm{C(CN)_2} <= \mathrm{P(CN)_3} <= \mathrm{S(CN)_4}$$
$$= \mathrm{NSO_2F} <= \mathrm{C(SO_2F)_2} <= \mathrm{P(SO_2F)_3}$$

In addition, the acidifying effect of fluorosulfonyl groups is greater than that of the corresponding cyano groups (Fig. 1.2) [22].

Classical strong mineral acids include:

$$\mathrm{HF}(365.7) < \mathrm{HNO_2}(330.5) < \mathrm{HCl}(328.0) < \mathrm{HBr}(318.2) < \mathrm{HNO_3}(317.8)$$
$$< \mathrm{HI}(309.3) < \mathrm{H_2SO_4}(302.2) < \mathrm{HSO_3F}(299.8) < \mathrm{HClO_4}(285 \pm 8)$$

(experimental gas-phase acidity (ΔG_acid) values in kcal mol^{-1}) [31]. Interestingly, the gas-phase acidity may be increased somewhat by the replacement of fluorine

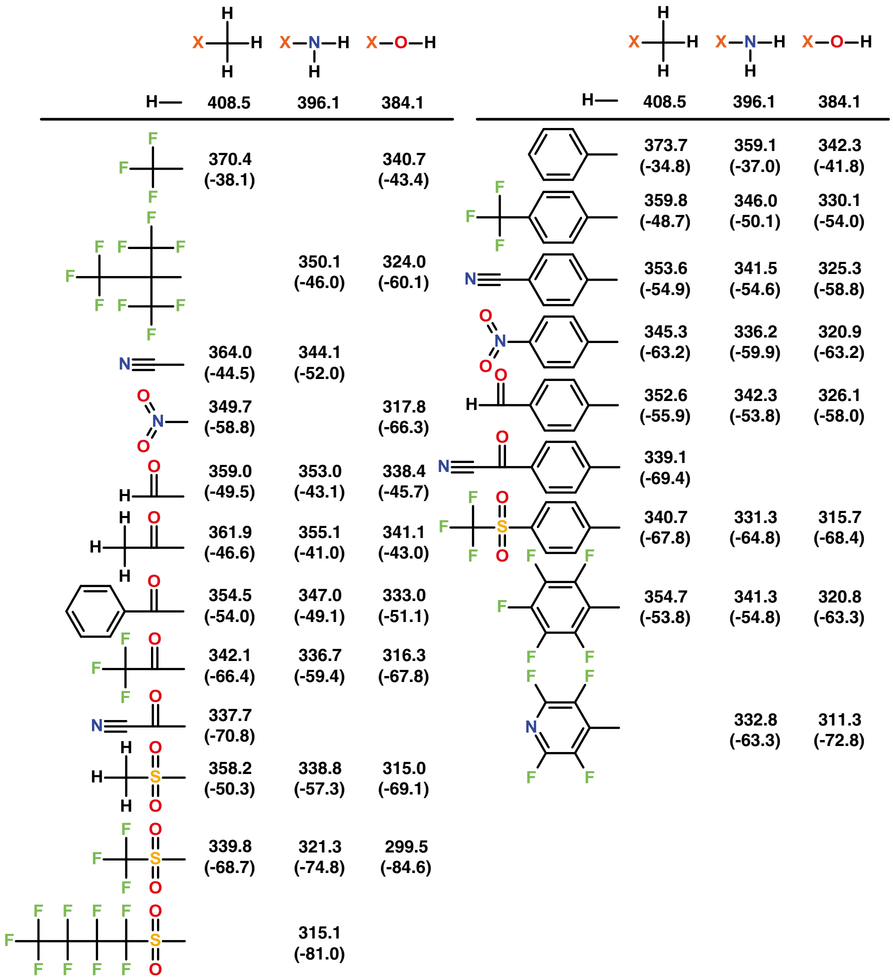


Fig. 1.1 Experimental gas-phase acidity values (kcal mol^{-1}) for various monosubstituents [20]

atoms with chlorine [32, 33]. The values noted above for HF and HCl are one example. The corresponding values for HCO_2CH_2F, HCO_2CH_2Cl, HCO_2CHF_2, and HCO_2CHCl_2 are 337.6, 335.4, 330.0, and 328.4 kcal mol^{-1}, respectively [32]. This effect, which is opposite to that expected from electronegativity alone, is attributed to the greater delocalization of the charge on the chlorine atom relative to fluorine. Thus, HSO_3Cl is expected to be a stronger acid than HSO_3F [26, 32, 33].

Replacement of the acid protons with Li$^+$ cations results in the corresponding lithium salts. Due to differences between the gas and condensed liquid-phase interactions, as well as the varying factors which determine proton (H$^+$) and Li$^+$ cation coordination/solvation, the exact trends noted for anion acidity do not always hold for the relative strength of lithium salt interactions. But, overall, the ionic association tendency of anions to coordinate Li$^+$ cations is governed by the same effects noted

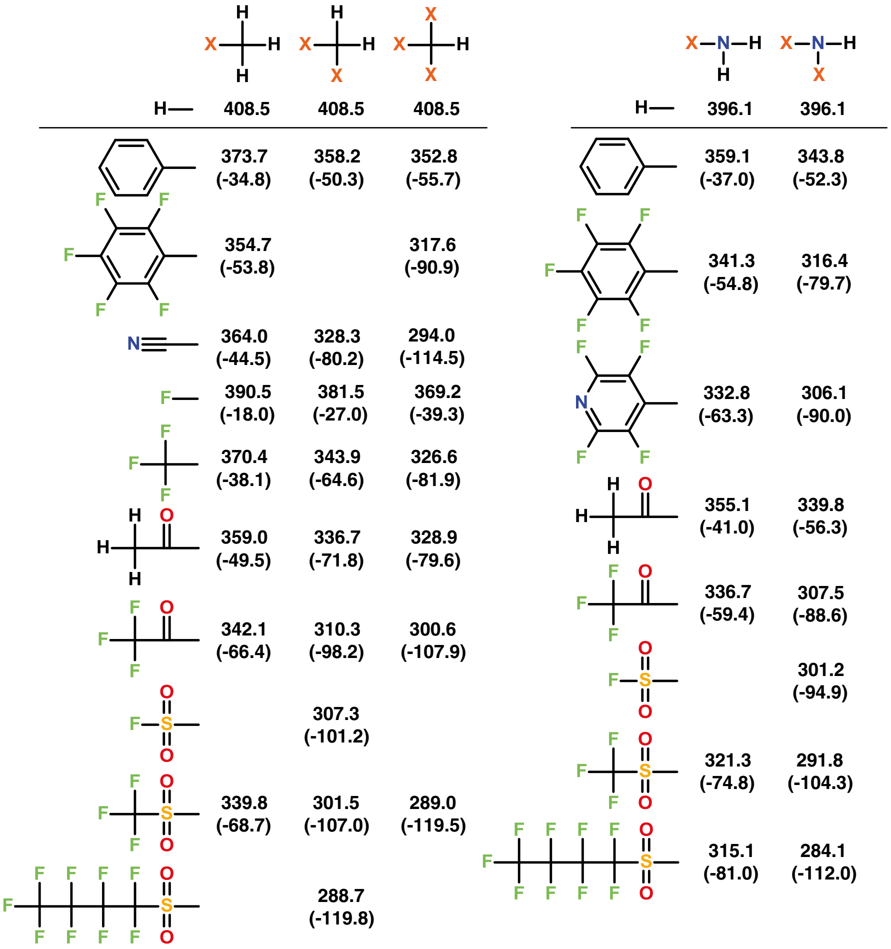


Fig. 1.2 Experimental gas-phase acidity values (kcal mol^{-1}) for various mono-, di-, and trisubstituents [20, 26]

above for acidity, as exemplified by DMSO electrolytes with lithium acetate salts with increasing anion fluorination which have the following increasing ionic association trend [34]:

$$CO_2CH_3^- > CO_2CH_2F^- > CO_2CHF_2^- > CO_2CF_3^-$$

Efforts to develop new lithium salts began in the late 1970s and 1980s with the use of perfluoroalkylsulfate (e.g., $SO_3CF_3^-$) and bis(perfluoroalkanesulfonyl)imide (e.g., $N(SO_2CF_3)_2^-$) anions. New salt development efforts gained momentum throughout the next three decades, but none of the developed salts have offered significant advantages over—while retaining the benefits of—the $LiPF_6$ salt, which serves as the standard for industry. Thus, $LiPF_6$ continues to be the predominant salt used for most commercial Li-ion batteries.

Some of the new lithium salts, however, have been found to be very effective as electrolyte additives for the modification of electrolyte–electrode interfaces. Other lithium salts hold promise as future primary salts for lithium batteries (i.e., replacements for some or all of the $LiPF_6$), especially for demanding battery electrolyte requirements such as low/high-temperature operation and superb safety characteristics, which cannot be met by the sole use of $LiPF_6$ [35–38]. Thus, there is a continued need for rapid advances in lithium salts for the diverse range of lithium battery chemistries which are now the focus of worldwide efforts to greatly improve vehicular and stationary energy storage technologies.

1.2 Electrolyte Salt Properties

Electrolyte salts must meet a broad and demanding range of properties—some of these include [12, 37, 39, 40] the following:

1. *Ionic Conductivity*: A high Li^+ cation transport rate is necessary to achieve high power (i.e., a high rate for the overall battery reaction) as the Li^+ cation mobility within the bulk electrolyte is often one of the main sources of impedance for the battery [41]. The choice of a lithium salt's anion dramatically influences the electrolyte's conductivity due to the variations in the Li^+ cation solvation and ionic association interactions resulting from the differences in anion structure and coordination strength. Although electrolyte conductivity is the parameter most frequently considered, the Li^+ cation mobility is actually obtained from the product of the conductivity and Li^+ cation transference (or transport) number (i.e., t_{Li+}) (fraction of the current carried by the Li^+ cations). This latter parameter is frequently not reported in the literature for a given electrolyte composition. The most common t_{Li+} measurement method is that reported by Bruce and Vincent [42–44]. Caution should be exercised in interpreting results from such measurements, however, as the data can be skewed by the reaction of the electrolyte with Li metal (active electrodes) resulting in erroneous t_{Li+} values, especially for liquid electrolytes.
2. *Salt Solubility/Crystalline Solvates*: Achieving a reasonably high lithium salt solubility in the electrolyte solvent(s) is necessary to provide sufficient charge carriers for rapid ionic conduction, as well as to prevent salting out of the salt (i.e., precipitation). It is important to distinguish between salt solubility and crystalline solvates. A salt may be highly soluble, but also may readily form a crystalline solvate phase with a high melting point (T_m) resulting in the formation of solids in the electrolyte which effectively extracts the salt from the electrolyte solution (causing the conductivity to plummet). This is a particular concern for low-temperature cell operation.

3. *Stability*: Electrolytes must, in general, be robust (nonreactive with other cell components) within the electrochemical potential window utilized for the battery charge/discharge reactions [45], as well as at elevated temperature, to achieve thousands of charge/discharge cycles with low capacity loss (fade). The temperature and voltage(s) at which oxidation and/or reduction of the electrolyte components occur are not independent of one another. As for chemical stability, the electrochemical stability of electrolytes (i.e., potential stability window) is strongly temperature dependent with a modest increase in temperature, in some cases, resulting in a substantial decrease in stability [46]. The potential window is also a strong function of the materials in contact with the electrolyte [46], as well as the presence of impurities. Thus, the potential window, as measured on an inert glassy carbon electrode, is not a clear indicator of the stability of an electrolyte in contact with active electrode materials (although a poor stability on this electrode likely also indicates a poor stability with more active electrodes). Stability is a complicated factor, however, as it may also be necessary for the salt's anions to selectively degrade—e.g., to form a solid-electrolyte interface (SEI) layer on the anode and/or cathode [12] and to stabilize the Al current collector [47].
4. *SEI Formation*: The SEI is a layer formed between the electrode surface and the electrolyte through the degradation/reaction of both electrolyte components and electrode material(s) [12]. Ideally, only a limited amount of materials react, with the resulting SEI layer preventing further electrode–electrolyte reactions and enabling the facile transport of only Li^+ cations between the electrode and electrolyte (resulting in a low impedance). The lithium salt(s) present in the electrolyte, whether as a bulk salt or an additive, can dramatically influence the SEI's composition, properties, and stability [48].
5. *Al Corrosion*: The use of Al as a cathode current collector in commercial Li-ion batteries is nearly ubiquitous [47]. A given electrolyte must passivate the electrolyte–Al interface to prevent corrosive pitting of the current collector during cell cycling to high potential (>3.6 V vs. Li/Li^+).
6. *Hydrolysis Stability*: Many anions hydrolyze when exposed to water, especially at elevated temperature—often resulting in the formation of HF. This results in additional costs associated with the salt's preparation, storage, and handling. This also strongly influences the cycling behavior and lifetime of batteries, especially when cycled to high temperature and/or high potential (>4.8 V vs. Li/Li^+) [49–54]. HF formation may also result from the reaction of the anions with solvent molecules (abstraction of a hydrogen) [55].

Other desirable features include low cost and low toxicity. Failure to meet one or more of these criterions prevents the practical use of a salt in lithium and Li-ion batteries. It is important to note, however, that many of these properties are strongly dependent upon the electrolyte formulation (e.g., solvents used, salt concentration, additives). Thus, electrolyte compositions need to be tailored to specific battery applications/demands.

1.3 Established Salts

A number of lithium salts are well established as salts which are used or have been previously used for lithium battery electrolytes, although many of these do not meet the necessary criterion for commercial battery electrolytes. Frequently studied lithium salts include (Fig. 1.3) [12, 40] the following:

1. *$LiClO_4$*: Lithium perchlorate was widely used for battery electrolyte research in the 1970s and 1980s due to its high ionic conductivity, high solubility in aprotic solvents, high thermal/electrochemical stability, and favorable SEI-forming properties [12, 56]. Electrolytes with the $LiClO_4$ salt, however, typically do not passivate the Al current collector as well as those with $LiPF_6$ [38, 57–60]. The high oxidation state of the Cl^{VII} atom also makes the anion a strong oxidant and thus the salt a potential explosive [61–63]. This has largely precluded the use of $LiClO_4$ for commercial batteries.
2. *$LiAsF_6$*: Like $LiClO_4$, lithium hexafluoroarsenate was widely used for electrolyte research in the 1970s and 1980s. In particular, it was found to improve the efficiency of Li metal plating/stripping relative to electrolytes with $LiClO_4$ [12]. $LiAsF_6$ has many properties in common with $LiPF_6$ [12, 40, 64], but the potential hazards associated with the salt have largely prevented its commercial usage. Although the As^V oxidation state is not toxic, the As^{III} and As^0 states, which might be formed from electrochemical reduction, are highly toxic.
3. *$LiPF_6$*: Lithium hexafluorophosphate is used almost exclusively in commercial Li-ion batteries. This salt has thus far demonstrated the best balance of essential properties necessary for a primary Li-ion electrolyte salt [12, 40]. In aprotic solvents, the resulting electrolytes have some of the highest conductivity values measured. $LiPF_6$-based electrolytes react to form a stable interface with the Al

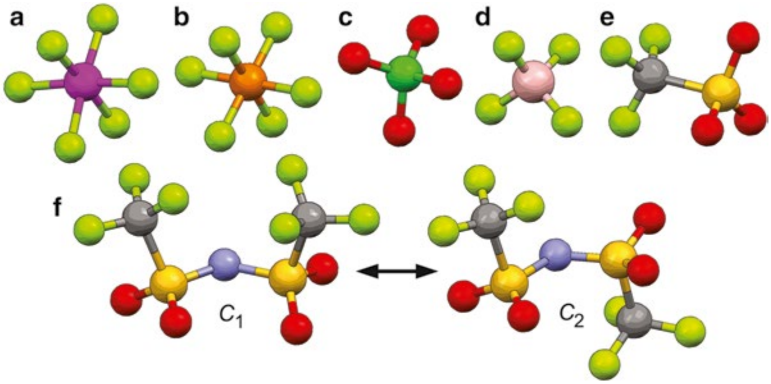


Fig. 1.3 Widely used lithium salt anions: (**a**) AsF_6^-, (**b**) PF_6^-, (**c**) ClO_4^-, (**d**) BF_4^-, (**e**) $SO_3CF_3^-$, and (**f**) $N(SO_2CF_3)_2^-$ ($TFSI^-$) (*C_1* (*cis*) and *C_2* (*trans*) conformations) (B—*tan*, C—*gray*, N—*blue*, O—*red*, F—*light green*, P—*orange*, S—*yellow*, Cl—*dark green*, As—*purple*)

current collector at high potential [58–60, 65–68] and a stable SEI with graphite electrodes when used with carbonate solvents [12, 40]. The P–F bond is labile, however, and the salt thus readily undergoes hydrolysis [69–73] and has a relatively low thermal stability [56, 71, 74–78]. The presence of HF in $LiPF_6$-based electrolytes, and its impact on cell performance, is one of the principal concerns associated with this salt's usage [49–54].

4. *$LiBF_4$*: Electrolytes with lithium tetrafluoroborate tend to have a significantly lower conductivity, relative to those with $LiPF_6$ [79–81], which has been a major impediment to its use in commercial Li-ion cells. The B–F bond is less labile than the P–F bond. Thus, the $LiBF_4$ salt is less susceptible to hydrolysis and more thermally stable than $LiPF_6$ [56, 82–85], but electrolytes with this salt do passivate Al well at high potential [59, 67]. Despite the lower conductivity, electrolytes with $LiBF_4$ have been shown to have improved cell cycling performance at low/high temperature, relative to cells with $LiPF_6$-based electrolytes, due to the formation of a less resistive SEI layer and improved thermal stability [81, 82, 86–89]. $LiBF_4$ may also serve as a useful additive to electrolytes with $LiPF_6$ [90] and it enables the use of γ-butyrolactone (GBL) as an electrolyte solvent (which is unstable with $LiPF_6$) [91–96].
5. *$LiSO_3CF_3$*: Lithium trifluoromethanesulfonate (triflate—most commonly abbreviated as "$CF_3SO_3^-$" in the scientific literature) was at one time widely used for electrolytes, especially for polymer electrolytes [97–107]. This salt has a high thermal stability [56] and is not susceptible to hydrolysis due to the stability of the C–F bond. Electrolytes with this salt, however, are found to be notably less conductive than those with $LiPF_6$ [5, 108–110], and $LiSO_3CF_3$-based electrolytes corrode the Al current collector at high potential [59, 65–67]. Thus, while this salt has been extensively used for research purposes, it is not used in commercial Li-ion batteries.
6. *LiTFSI* (i.e., *$LiN(SO_2CF_3)_2$*): Many acronyms are used for the lithium bis(trifluoromethanesulfonyl)imide salt: LiTFSI, LiTFSA, $LiNTf_2$, $LiTf_2N$, etc. The terms "imide" and "amide" are frequently interchanged. Much of the initial interest in the LiTFSI salt was due to its tendency to form amorphous mixtures with poly(ethylene oxide), rather than crystalline phases, when mixed to form polymer electrolytes [97, 111–113]. The bis(perfluoroalkanesulfonyl)imide anions, such as $TFSI^-$, are highly flexible with two low-energy conformations in which the $–CF_3$ groups are either *cis* (C_1) or *trans* (C_2) to one another (Fig. 1.3f) [114–117]. The combination of the strongly electron-withdrawing fluorine atoms and resonance structures due to the sulfonyl groups results in extensive negative-charge delocalization across the $–SO_2–N–SO_2–$ backbone of the anion [108, 118]. Thus, $TFSI^-\ldots Li^+$ cation coordination occurs predominantly through oxygen atom coordination to the Li^+ cations (rather than $N\ldots Li^+$ or $F\ldots Li^+$ cation coordination) [119–124]. Electrolytes with this salt generally are somewhat less conductive than the corresponding $LiPF_6$ electrolytes [5, 11, 125]. The $TFSI^-$ anion has a high thermal stability and is not susceptible to hydrolysis due to the very stable C–F bonds [56, 126], but dilute aprotic solvent-base electrolytes with LiTFSI are known to strongly corrode the Al current collector at high potential [59, 60, 65–68]. This observation should be qualified with the fact that electrolytes

with ionic liquids with the $TFSI^-$ anion (with or without LiTFSI) do not corrode the Al current collector [127–132], nor do electrolytes with very high concentrations of LiTFSI [133]. Thus, simple generalizations about electrolyte salt properties (e.g., "the LiTFSI salt corrodes Al") may be flawed.

1.4 Electrolyte Characterization Tools

Rigorous electrolyte characterization requires a thorough understanding of not only the properties of the electrolyte, but also the solution structure. Anion solvation in protic solvents (e.g., water, methanol, ammonia) occurs through hydrogen-bonding interactions. Protic solvents, however, have poor electrochemical stability due to the acidic protons (i.e., O–H, N–H, S–H). The aprotic solvents useful for electrolyte applications do not have acidic protons. Thus, in general, the anions remain unsolvated (naked) in electrolytes with such solvents. Dissolution of a lithium salt therefore occurs through the solvation of the Li^+ cations by the formation of coordination bonds between the Li^+ cations and electron lone-pairs of the solvent donor atoms. Anions also form coordination bonds to the Li^+ cations using the electron lone-pairs on donor atoms (F, O, N, etc.). The competition between the solvent and anions for Li^+ cation coordination—the occupancy of the Li^+ cation's coordination shell—determines the solvate species which are present in the electrolyte. Thus, the ion solvation (solvent–Li^+ cation interactions) and ionic association tendency of the anions (anion–Li^+ cation interactions) are important features of electrolytes which are governed by the solvent/anion structure: steric factors which influence the coordination bond formation and packing around the cation, polarizability, charge delocalization, etc. The following discussion provides a short overview of a methodology which may be used to identify the electrolyte interactions and how these are determinants for electrolyte properties [134–136]:

1. *Phase Diagrams and Solvate Crystal Structures*: Solvent-lithium salt phase diagrams are an underutilized, but highly informative tool for examining electrolyte interactions. Acetonitrile (AN) is a particularly useful model solvent for comparing a salt's phase behavior as this solvent has only a single electron lone-pair and thus is either uncoordinated or coordinated to a single Li^+ cation. Figure 1.4 compares the phase diagrams of $(AN)_n$–LiX mixtures with $LiPF_6$, LiTFSI, $LiClO_4$, $LiBF_4$, and $LiCO_2CF_3$ [134, 135]. Figure 1.4 also shows the solvent/ion coordination in the solvate crystal structures determined for some of the indicated phases in the phase diagrams: $(AN)_6$:$LiPF_6$ [137], $(AN)_5$:$LiPF_6$ [138], $(AN)_1$:LiTFSI [139], $(AN)_4$:$LiClO_4$ [140], $(AN)_2$:$LiBF_4$ [141], and $(AN)_1$:$LiBF_4$ [142]. Notable features include the tendency of the $(AN)_n$–$LiPF_6$ and $(AN)_n$–$LiClO_4$ mixtures to form crystalline solvates with a high T_m. This is attributed to the small, symmetric, and relatively weakly coordinating anions which readily pack well within the solvate crystalline lattices. In contrast, dilute $(AN)_n$–LiTFSI mixtures form low T_m solvates and a crystallinity gap exists for more concentrated mixtures for which it is difficult or impossible to crystallize the

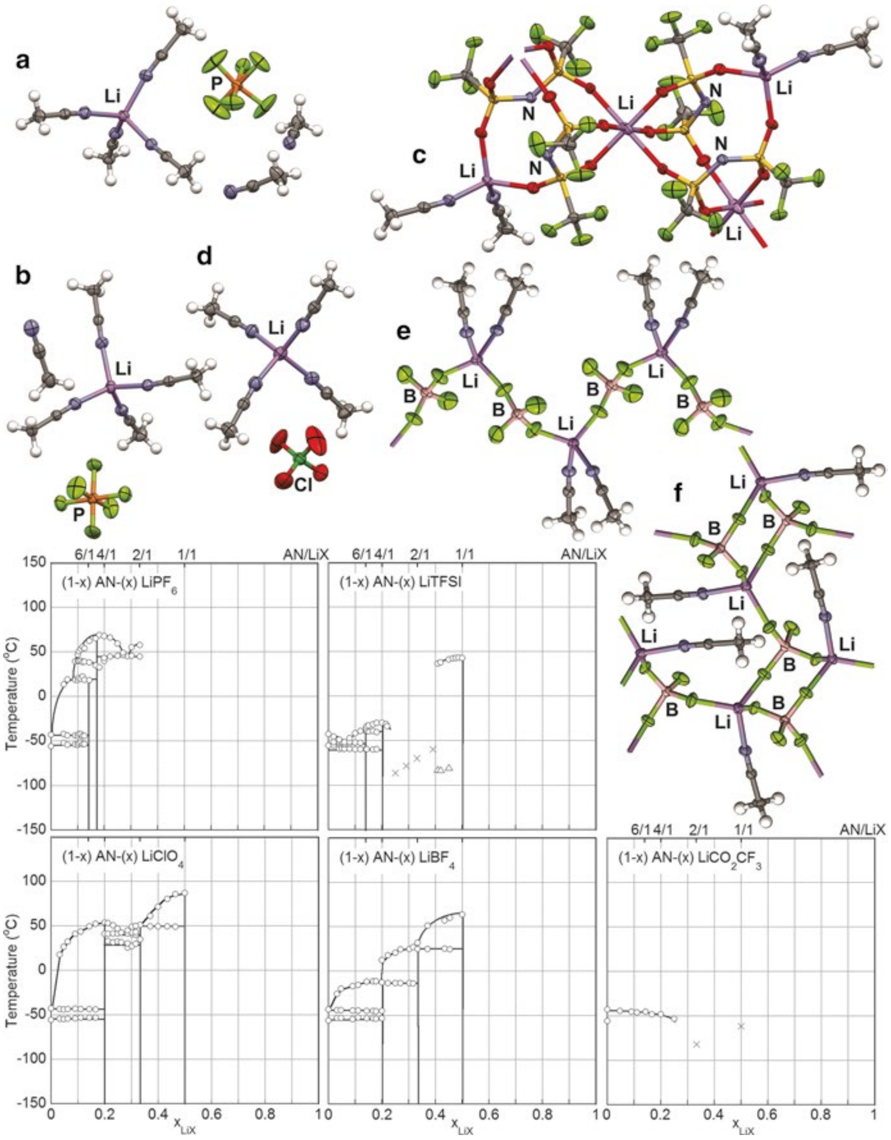


Fig. 1.4 Phase diagrams of $(AN)_n$–LiX mixtures with $LiPF_6$, LiTFSI, $LiClO_4$, $LiBF_4$, and $LiCO_2CF_3$ [134, 135] and ion/solvent coordination within the solvate crystal structures: (**a**) $(AN)_6$:$LiPF_6$ [137], (**b**) $(AN)_5$:$LiPF_6$ [138], (**c**) $(AN)_1$:LiTFSI [139], (**d**) $(AN)_4$:$LiClO_4$ [140], (**e**) $(AN)_2$:$LiBF_4$ [141], and (**f**) $(AN)_1$:$LiBF_4$ [142] (the sample T_g values are indicated by an "x" for fully amorphous samples and by a triangle for partially crystalline samples)

electrolytes—these features may be attributable to the bulky anion with lower symmetry and its influence on solvate formation. The ClO_4^- and BF_4^- anions are both tetrahedral and nearly of the same size (Fig. 1.3c, d). Interestingly, although the $(AN)_n$–$LiClO_4$ and $(AN)_n$–$LiBF_4$ mixtures form the same solvate crystalline

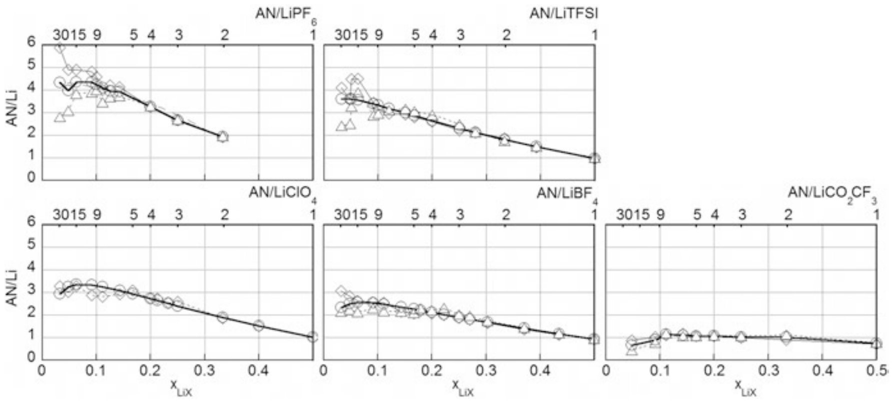


Fig. 1.5 Calculated Li$^+$ cation average solvation numbers (AN/Li) for (AN)$_n$–LiX electrolytes. The *dark solid line* corresponds to the average of the data obtained from the analysis of the C–C and C≡N Raman vibrational stretching bands (note that values for approximately $n > 9$ are unreliable due to a compilation of experimental errors associated with the Raman solvent band deconvolution) [134, 135]

phases (i.e., 4/1, 2/1, and 1/1 AN/LiX compositions), significant differences exist in the T_m of the 4/1 phases in which the Li$^+$ cations are fully solvated by four AN molecules and the anions are uncoordinated (Fig. 1.4d). This can be explained by the difference in the solvation/ionic association tendency of the two salts, as the BF$_4^-$ anions have a greater tendency to displace the solvent molecules in the Li$^+$ cation coordination shells (and thus a greater tendency to disrupt the solvate structure). The 6/1 and 5/1 solvates with LiPF$_6$ also form solvates with four-fold Li$^+$ cation coordination (Fig. 1.4a, b), but also include uncoordinated solvent molecules to facilitate the packing of the solvated Li$^+$ cations and PF$_6^-$ anions together. Finally, the (AN)$_n$–LiCO$_2$CF$_3$ mixtures do not form crystalline solvates. Only some of the excess AN is able to crystallize as a pure solvent phase.

2. *Li$^+$ Cation Solvation*: Electrolyte (average) solvation numbers are most commonly determined using vibrational spectroscopy by examining the solvent's vibrational band(s). Upon coordination to a Li$^+$ cation through an electron lone-pair, the electron density of the solvent molecule (and thus bond lengths/angles) changes resulting in variations in the solvent band positions [134, 135]. Integration of the peak area of the bands associated with the uncoordinated and coordinated solvent enables the calculation of the fraction of coordination solvent molecules—this number multiplied times the total number of solvent molecules in the electrolytes gives the average solvation number (Fig. 1.5). This analysis can be confounded, however, by overlapping vibrational bands, as well as variations in the relative intensity (scaling) of the peaks. Failure to account for these factors can result in a highly misleading interpretation of the experimental data. The scientific literature related to the determination of solvation numbers is rife with these problems. From an analysis of the AN Raman C–C and C≡N stretching

vibrations [134, 135], ion solvation for $(AN)_n$–LiX mixtures increases in the order:

$$LiPF_6 > LiTFSI \geq LiClO_4 > LiBF_4 >> LiCO_2CF_3$$

The maximum ion solvation number (average number of solvent molecules coordinated to the Li^+ cations) for the AN mixtures is found to be about 4 [134, 135]. Wide differences are noted for the solvation numbers for the varying anions. For example, for highly concentrated (>3 M) $(AN)_n$–LiX ($n=4$) liquid mixtures (i.e., four AN molecules present per Li^+ cation) at 60 °C, the solvation numbers are approximately 3.2 for $LiPF_6$, 2.8 for LiTFSI, 2.7 for $LiClO_4$, 2.1 for $LiBF_4$, and 1.0 for $LiCO_2CF_3$ [134, 135]. Note that these numbers are not the coordination numbers for the Li^+ cations. Rather, they represent the average number of coordinated solvent molecules with the anions making up the difference in the Li^+ cation coordination shells. Thus, the Li^+ cations in the $LiPF_6$ electrolytes are expected to be well solvated, whereas those for the $LiCO_2CF_3$ electrolytes are instead expected to be highly associated to the anions over the entire concentration range. Despite this, the solubility of $LiCO_2CF_3$ in AN is exceptionally high (>5 M). Thus, salt solubility and ionic association are not necessarily directly correlated with one another.

3. *Ionic Association*: The ionic association present in electrolytes is most commonly also determined using vibrational spectroscopy by examining one or more anion vibrational bands which shift upon coordination to the Li^+ cations [103–105, 121, 134, 135, 143, 144]. Often, the assignment of the bands to specific modes of coordination is somewhat ambiguous and the analysis of the data is simply based upon guesswork. This can be very misleading. To resolve this problem, the study of the vibrational bands for model crystalline (solid) solvates with known structure is particularly useful for assigning the vibrational band positions to particular anion…Li^+ cation coordination modes. This evaluation has been done for the salts $LiClO_4$ [145, 146], $LiBF_4$ [141], and LiDFOB [147]. Work is currently in progress to provide similar analyses for $LiPF_6$, LiTFSI, and $LiSO_3CF_3$. An example of the modes of coordination for the BF_4^- anion and the corresponding anion band positions obtained from crystalline solvates (such as AGG-I $(AN)_2$:$LiBF_4$ and AGG-II $(AN)_1$:$LiBF_4$—Fig. 1.4e, f) is shown in Fig. 1.6 [141]. This characterization "tool" is quite useful for the evaluation (deconvolution) of the Raman band data for the liquid electrolytes shown in Fig. 1.7b. This form of analysis is greatly complemented by molecular dynamics (MD) simulations (validated by the experimental data) which provide a visual representation of the solvates present in solution (Fig. 1.8) [134–136]. As an example of the utility of this marriage of methods, the solvates shown in Fig. 1.8c, d represent contact ion pairs (CIP-I and CIP-II, respectively). But the solvates in Fig. 1.8b, e, g–j also contain BF_4^- anions coordinated to a single-Li^+ cation. Thus the spectral signature of these anions would be that of CIP-I anion coordination (Fig. 1.6b). The experimental spectroscopic data, therefore, does not provide direct information about the solvates present. Instead, it indicates the fraction of the anions present

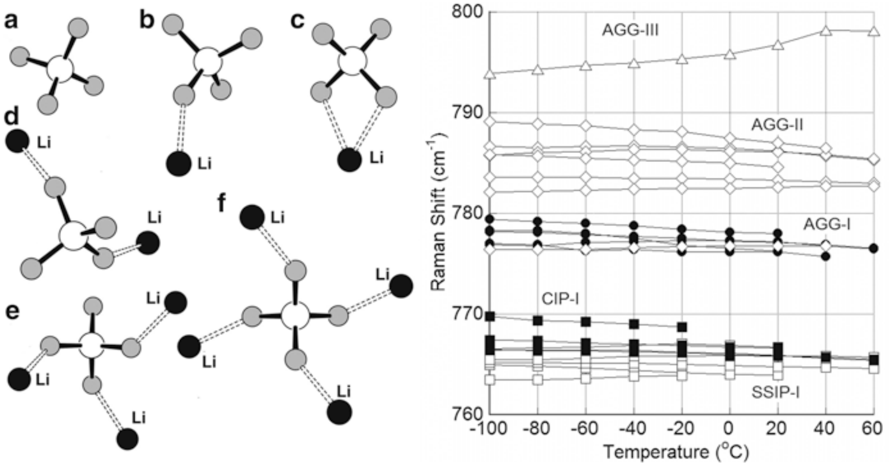


Fig. 1.6 Varying modes of BF_4^-…Li^+ cation coordination: (**a**) SSIP, (**b**) CIP-I, (**c**) CIP-II, (**d**) AGG-I, (**e**) AGG-II, and (**f**) AGG-III and Raman band peak positions for the BF_4^- anion v_1 vibrational band for different crystalline solvates (each line corresponds to a different crystalline solvate) (no crystalline solvates with CIP-II coordination were available for analysis) [141]

with different modes of anion…Li^+ cation coordination. This is an important distinction which is generally not made throughout the published scientific literature on electrolyte characterization. The ionic association tendency of lithium salts is found to increase in the order [134–136, 148]:

$$LiAsF_6, LiPF_6 < LiTFSI \le LiClO_4 < LiBF_4 < LiSO_3CF_3 < LiCO_2CF_3$$

A comparison of this with the Li^+ cation solvation data (Fig. 1.5) indicates that this increasing ionic association tendency is opposite to that noted for the Li^+ cation solvation. This is due to the competitive coordination of the solvent molecules and anions to the Li^+ cations. Quantum chemical (QC) calculations and MD simulations find that $(AN)_n$–LiX mixtures consist predominantly of Li^+ cations with four-fold coordination to anions and/or AN solvent molecules (very little five-fold Li^+ cation coordination is found for AN-based electrolytes) [134–136]. As noted above, aprotic solvent molecules, such as AN, have only weak interactions with anions. Thus, the Li^+ cation coordination shell in solution consists of anions and/or solvent molecules. The competitive coordination between these determines the solvate distribution present in solution (Fig. 1.8) [134–136, 141].

4. *Transport Properties*: The transport properties (e.g., viscosity and ionic conductivity) of electrolytes are one of the key metrics used to gauge electrolytes. The viscosity may be correlated with the wettability of an electrolyte with the porous separator and electrodes. Figure 1.9 shows the variation in the viscosity of $(AN)_n$–LiX mixtures at 60 °C with different lithium salts [136]. Perhaps contrary to expectations, the most dissociated salts result in the highest viscosity for the more dilute mixtures. The differences in the viscosity for the different salts and

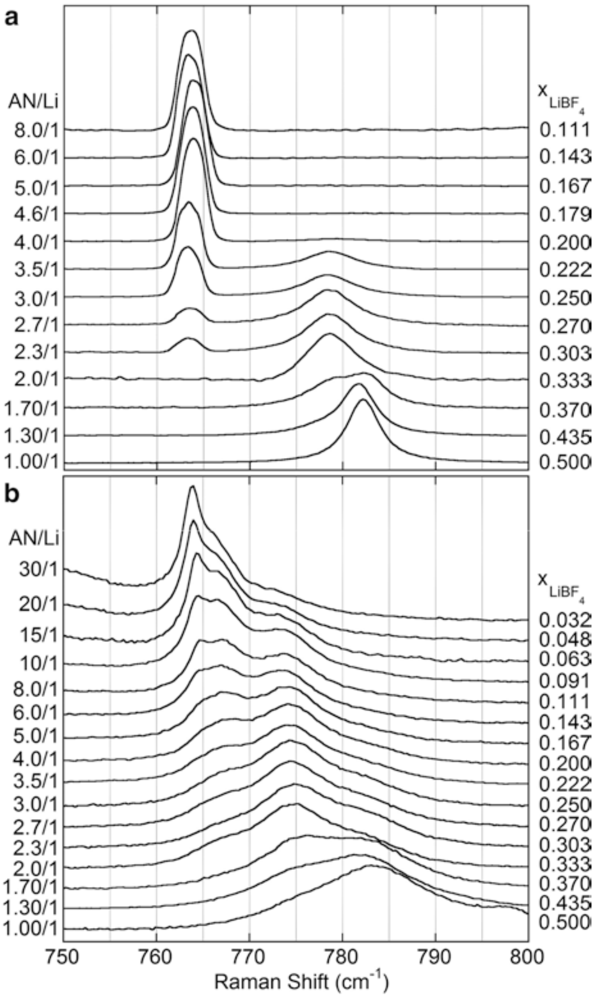


Fig. 1.7 BF_4^- anion band variation with concentration for the $(AN)_n$–$LiBF_4$ mixtures at (**a**) −80 °C and (**b**) 60 °C [134]

concentrations can be explained by the differences in solution structure (amount of uncoordinated solvent and number/distribution of solvates) (Figs. 1.8 and 1.10) [136]. Figure 1.11 shows the variation in the conductivity of the $(AN)_n$–LiX mixtures at 60 °C with the different lithium salts [136]. The differences noted in terms of both salt concentration and anion structure can once again be explained by the differences in electrolyte solution structure (types and distribution of solvates and uncoordinated anions, as well as the solvate formation/evolution dynamics) [136]. It is important to note that the AN electrolytes with $LiPF_6$ have the highest conductivity, but also the highest viscosity (Fig. 1.9). Similarly, the AN electrolytes with $LiCO_2CF_3$ have the lowest conductivity and the lowest viscosity.

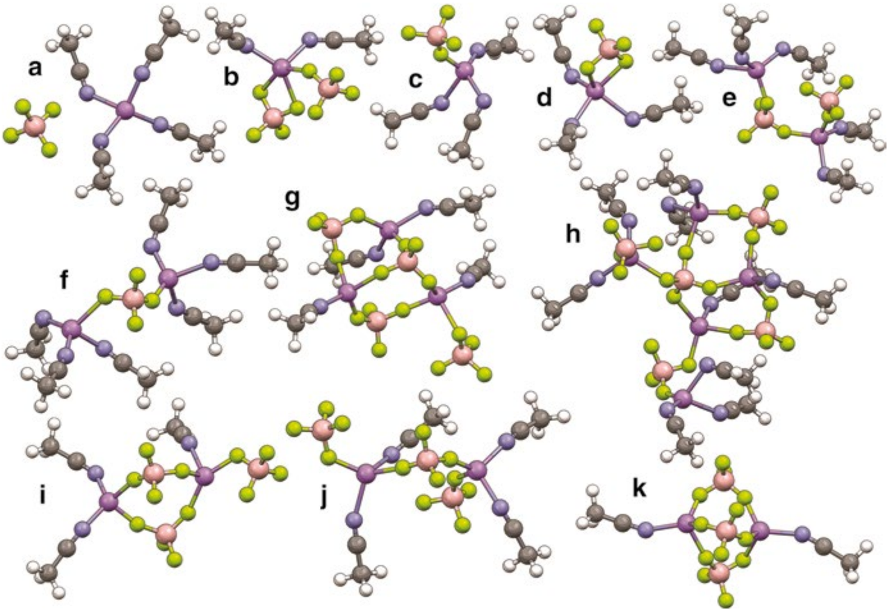


Fig. 1.8 Representative Li^+ cation solvate species (i.e., coordination shells) extracted from the MD simulations for the $(AN)_n$–$LiBF_4$ mixtures ($n=30$, 20, and 10) at 60 °C with BF_4^- coordination: (**a**) SSIP; (**b**) CIP-I, CIP-II; (**c**) CIP-I; (**d**) CIP-II; (**e**) CIP-I, AGG-I; (**f**) AGG-I; (**g**) CIP-I, AGG-I (×3); (**h**) CIP-I, AGG-I (×3), AGG-III; (**i**) CIP-I, AGG-I (×2); (**j**) CIP-I (×2), AGG-I; and (**k**) AGG-I (×3). Only solvent and BF_4^- anions within 3.33 Å of a Li^+ cation are shown (Li—*purple*, B—*tan*, C—*gray*, N—*blue*, F—*light green*) [134]

This clearly shows that the conventional wisdom that conductivity is directly linked with viscosity—"a high conductivity is achieved for electrolytes with a low viscosity"—is inaccurate. Instead, both properties originate from the molecular-level interactions between the ions and solvent (i.e., solution structure). These properties are thus only indirectly correlated with one another. Frequently, it is found that a maximum in electrolyte conductivity is obtained near a 1 M salt concentration (Fig. 1.12) (for the AN electrolytes shown in Fig. 1.11, this corresponds to an AN/Li ratio of about 14 to 17—depending upon the salt's formula weight [136]). In addition, the choice of aprotic solvent or solvent mixtures used greatly impacts the conductivity of an electrolyte (Figs. 1.12 and 1.13) [5, 149–153], but importantly the trend in the conductivity for different salts remains largely the same irrespective of the solvent(s) used. For example, the conductivity with different lithium salts in an EC:DMC equimolar binary mixture with the solvents 3-methylsydnone (3-MSD) or 3-ethylsydnone (3-ESD) added is found to increase in the order [154]:

$$\mathrm{LiPF_6} > \mathrm{LiClO_4} > \mathrm{LiN\left(SO_2CF_3\right)_2\left(LiTFSI\right)} > \mathrm{LiN\left(SO_2C_2F_5\right)_2} > \mathrm{LiBF_4} > \mathrm{LiSO_3CF_3}$$

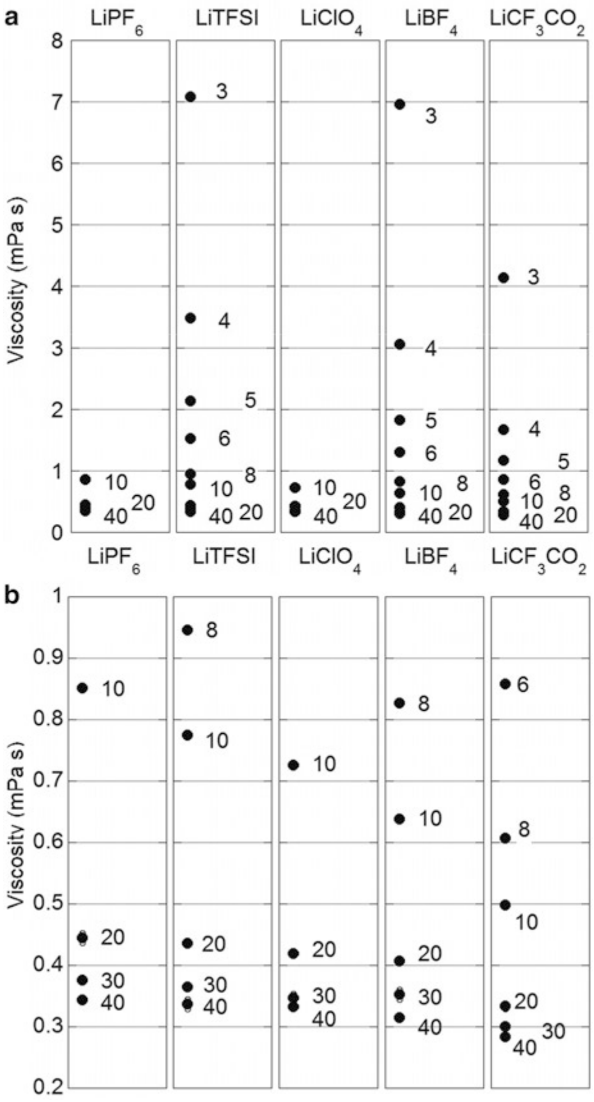


Fig. 1.9 (**a**) Viscosity of $(AN)_n$–LiX mixtures at 60 °C (AN/LiX (n) noted in plots) and (**b**) the same data for the dilute mixtures alone. Data for concentrated mixtures with $LiPF_6$ and $LiClO_4$ were not gathered as these samples crystallize during the measurements [136]

which is similar to the trend in Fig. 1.11 (if $LiClO_4$ and LiTFSI are interchanged). A similar order is also noted for the electrolytes in Table 1.1 and Fig. 1.14 [5, 108–110, 125, 150–153, 155, 156]. This latter figure indicates that the EC:PC electrolyte with $LiPF_6$ is slightly more conductive than the corresponding electrolyte with $LiAsF_6$, whereas the opposite is true for the 2-MeTHF:EC:PC electrolytes. Also, there is a crossover in the conductivity for the 2-MeTHF:EC:PC

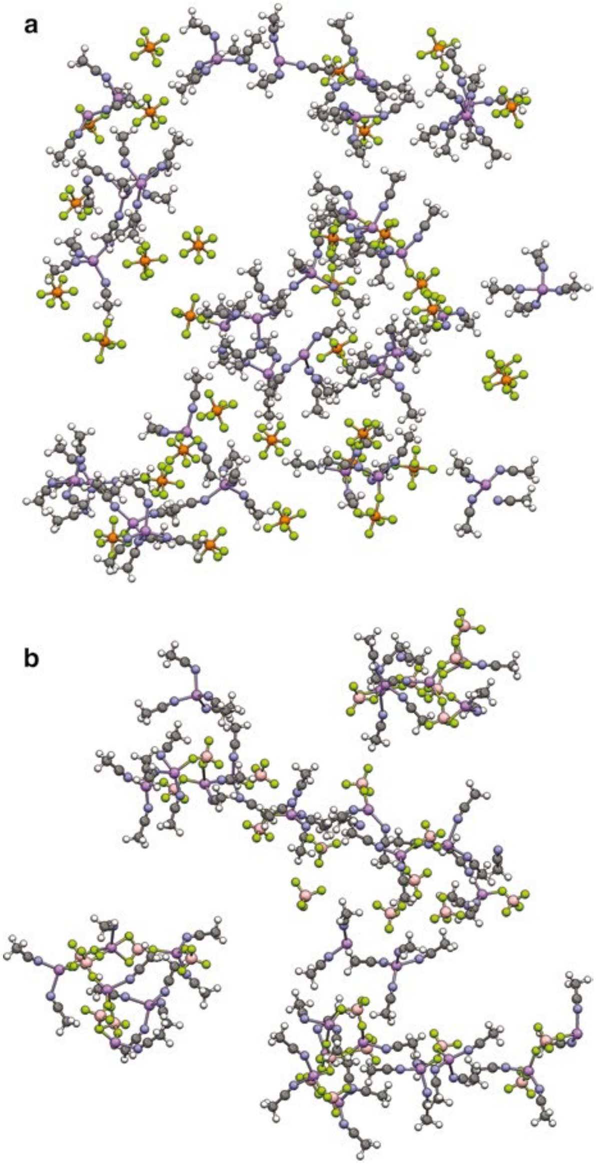


Fig. 1.10 Snapshot of the molecular simulations of $(AN)_n$–LiX mixtures ($n = 20$) with (**a**) $LiPF_6$ and (**b**) $LiBF_4$ (Li—*purple*, B—*tan*, C—*gray*, N—*blue*, F—*light green*, P—*orange*). Uncoordinated AN solvent molecules have been removed to aid in discerning the solvates present [136]

electrolytes with $LiBF_4$ or $LiSO_3CF_3$ with varying temperature. These points demonstrate that the ion solvation and ionic association interactions within the electrolytes are a function of numerous factors and generalizations about salt properties/behavior should be used with caution, as noted above.

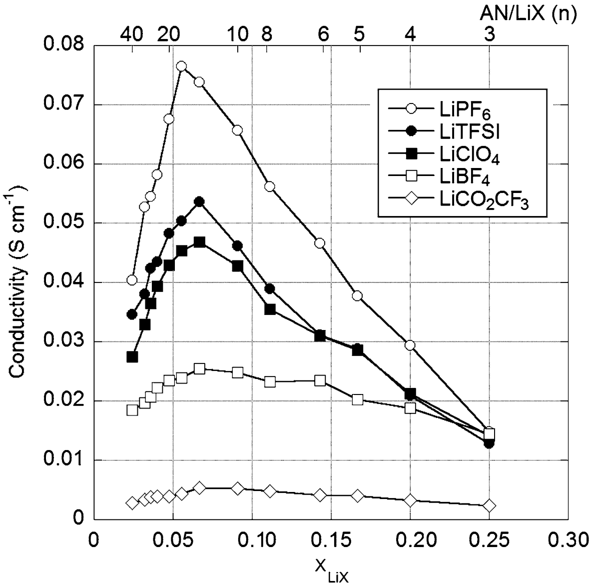


Fig. 1.11 Ionic conductivity of $(AN)_n$–LiX mixtures at 60 °C (solvent/LiX ratio (*n*) noted at the top of the plot) ($LiSO_3CF_3$ data not shown due to crystalline solvate formation) [136]

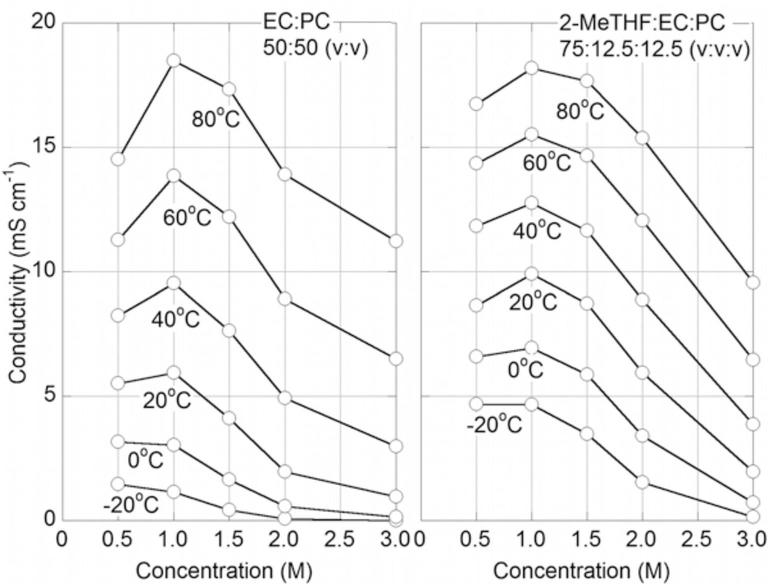


Fig. 1.12 Ionic conductivity of EC:PC (50:50 v:v) and 2-MeTHF:EC:PC (75:12.5:12.5 v:v:v) mixtures with $LiAsF_6$ for different temperatures and salt concentrations [5]

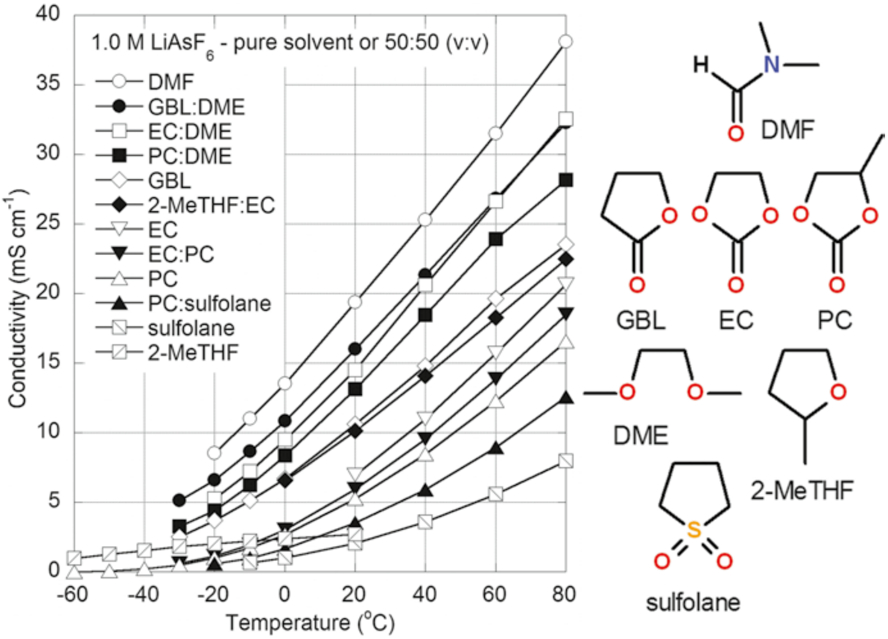


Fig. 1.13 Ionic conductivity of 1 M LiAsF$_6$ electrolytes with the indicated solvents (either a single solvent or a 50:50 v:v binary mixture) [5]

Table 1.1 Conductivity (mS cm^{-1}) of electrolytes with various lithium salts (1 M) at 25 °C (^{a}v:v; ^{b}w:w) [108–110, 125, 155, 156]

Lithium salt (anion)	PC:DME (50:50)^a	PC:DMC (50:50)^a	PC:DEC (50:50)^b	PC:EMC (50:50)^a	EC:DMC (50:50)^a	EC:THF (50:50)^a	GBL
$\mathrm{AsF_6^-}$	14.8	–	7.6	–	–	13.7	10.48
$\mathrm{PF_6^-}$	15.3	10.0	7.1	9.33	11.2	–	11.63
$\mathrm{ClO_4^-}$	13.5	6.8	4.5	6.26	10.1	13.5	–
$\mathrm{N(SO_2CF_3)_2^-}$	12.6	–	–	7.57	–	–	9.21
$\mathrm{BF_4^-}$	9.5	–	2.5	3.72	–	9.5	7.33
$\mathrm{SO_3CF_3^-}$	6.1	2.1	–	–	3.1	5.4	–

PC propylene carbonate, *EC* ethylene carbonate, *DME* 1,2-dimethoxyethane (or monoglyme), *DMC* dimethyl carbonate, *DEC* diethyl carbonate, *THF* tetrahydrofuran, *GBL* γ-butyrolactone

1.5 Advanced Salts—Fluoroborates and -Phosphates

Lithium salts with tetraalkylborate anions are highly soluble in dioxolane (up to 3 M) [157]. This is due to the poorly coordinating tetraalkylborate anions which lack donor atoms with electron lone-pairs for Li$^+$ cation coordination [158]. Thus, solvent molecules readily displace the anions in the Li$^+$ cation coordination shells. This also accounts for the poor chemical stability of the salts [159]: lithium

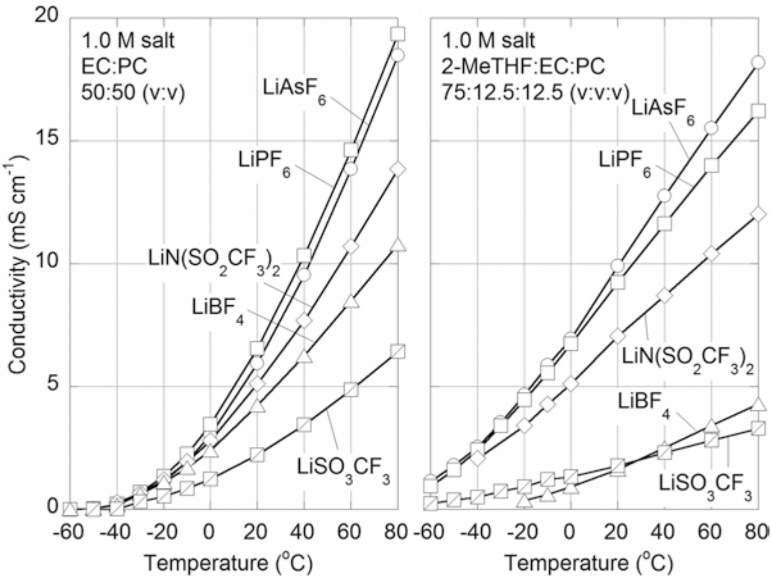


Fig. 1.14 Ionic conductivity of EC:PC (50:50 v:v) and 2-MeTHF:EC:PC (75:12.5:12.5 v:v:v) mixtures with the indicated lithium salts (1 M) [5]

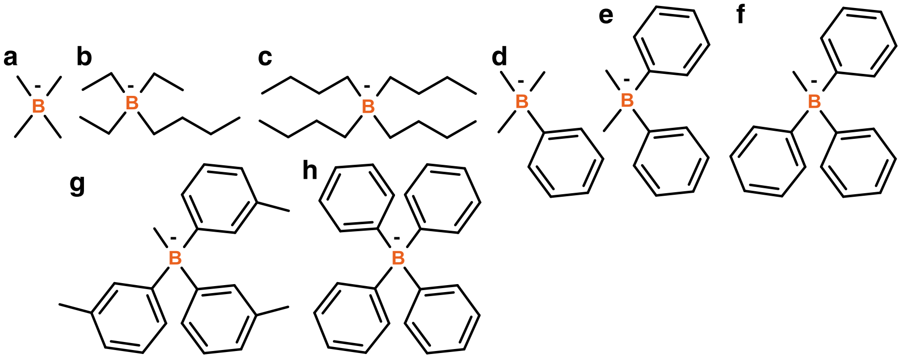


Fig. 1.15 Examples of organoborate anions: (**a**) $B(CH_3)_4^-$ [158, 160, 161], (**b**) $B(C_2H_5)_3(C_4H_9)^-$ [160, 161], (**c**) $B(C_4H_9)_4^-$ [160, 161], (**d**) $B(CH_3)_3(C_6H_5)^-$ [160, 162], (**e**) $B(CH_3)_2(C_6H_5)_2^-$ [160], (**f**) $B(CH_3)(C_6H_5)_3^-$ [160], (**g**) $B(CH_3)(C_6H_4(CH_3))_3^-$ [160], and (**h**) $B(C_6H_5)_4^-$ [160, 161]

tetramethylborate (i.e., $LiB(CH_3)_4$) (Figs. 1.15a and 1.16a) [158, 160–162] is stable in air [157], but lithium tetrabutylborate (i.e., $LiB(C_4H_9)_4$) (Fig. 1.15c) is pyrophoric [157]. In the former salt, the Li^+ cations are coordinated by the methyl hydrogens [158], but less favorable coordination may occur for anions with longer alkyl chains making the anions more reactive. Lithium tetraphenylborate (*LiBPh₄*) (i.e., $LiB(C_6H_5)_4$) (Figs. 1.15h and 1.16c) is stable only when the Li^+ cations are fully solvated—this

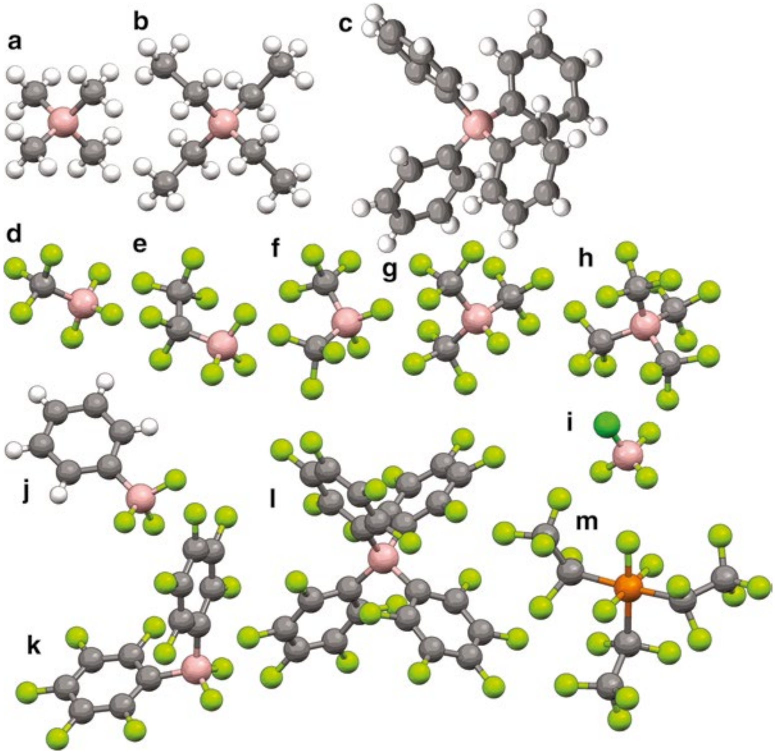


Fig. 1.16 Anion structures: (**a**) $B(CH_3)_4^-$, (**b**) $B(C_2H_5)_4^-$, (**c**) $B(C_6H_5)_4^-$ (BPh_4^-), (**d**) $BF_3(CF_3)^-$ (**e**) $BF_3(C_2F_5)^-$ (FAB^-), (**f**) $BF_2(CF_3)_2^-$, (**g**) $BF(CF_3)_3^-$, (**h**) $B(CF_3)_4^-$, (**i**) BF_3Cl^-, (**j**) $BF_3(C_6H_5)^-$, (**k**) $BF_2(C_6F_5)_2^-$, (**l**) $B(C_6F_5)_4^-$ ($BArF^-$), and (**m**) $PF_3(C_2F_5)_3^-$ (FAP^-) (B—*tan*, C—*gray*, F—*light green*, P—*orange*)

salt is typically sold commercially as the $(DME)_3$:$LiBPh_4$ solvate, while crystal structures have been reported for the $(H_2O)_4$:$LiBPh_4$ [163], $(H_2O)_2(DME)_1$:$LiBPh_4$ [164], $(H_2O)_2(THF)_2$:$LiBPh_4$ [165], $(THF)_1(12C4)_1$:$LiBPh_4$ [166], $(triglyme)_1$:$LiBPh_4$ [167], and $P(EO)_5$:$LiBPh_4$ [168] solvates—all with fully solvated Li^+ cations and uncoordinated BPh_4^- anions. The unsolvated $LiBPh_4$ salt slowly reacts with dry air. PC-based electrolytes with $LiBPh_4$ (and some DME) have a conductivity which is similar (slightly lower) to that of electrolytes with $LiBF_4$, but the conductivity of THF- and DME-based electrolytes with $LiBPh_4$ is higher than for those with $LiBF_4$ [169]. This may be attributable to variations in the ionic association interactions for the $LiBF_4$ salt with the different solvents, whereas the $LiBPh_4$ salt remains fully dissociated for all of the electrolyte formulations.

The anions from conjugate Brønsted–Lewis superacids represent the core lithium salts used for commercial lithium batteries (i.e., $LiPF_6$ and $LiBF_4$). The acidity order determined from QC calculations is as follows: HBF_4 (287.7) < HPF_6 (276.6) < $HTaF_6$ (268.3) < $HAlCl_4$ (257.4) < $HSbF_6$ (255.5) (DFT-calculated ΔG_{acid}

values in kcal mol^{-1}) [21]. $LiBF_4$ and $LiPF_6$ have both been extensively used for lithium battery research, and the latter is used in commercial Li-ion cells. The synthesis of $LiTaF_6$ and $LiNbF_6$ has been reported [170–175]. Although $LiTaF_6$ is highly soluble in THF, after the salt dissolution the electrolyte subsequently polymerized [175]. The conductivity of a 0.33 M $LiTaF_6$ electrolyte with sulfolane is 3.8×10^{-3} S cm^{-1} at 75 °C [175] (which is somewhat lower than a 0.33 M $LiPF_6$ electrolyte with sulfolane at this temperature [5]). The conductivity of PC-based electrolytes with either $LiTaF_6$ or $LiNbF_6$ was also found to be lower than the corresponding electrolytes with $LiPF_6$ [174]. Electrolytes with $LiTaF_6$ result in very poor Li metal cycling efficiency suggesting that the TaF_6^- anions may also have poor reductive stability [6]. The reductive stability of the MF_6^- anions is reported to follow the order $SbF_6^- < AsF_6^- < PF_6^-$ [176]. Electrolytes with $LiSbF_6$ have a similar conductivity to those with $LiPF_6$, but the $LiSbF_6$ salt may be corrosive to metals [6]. The Li_2SiF_6 and Li_3AlF_6 salts tend to be poorly soluble in aprotic solvents, and the resulting electrolytes have a low conductivity [6]. In contrast to these salts, $LiAsF_6$ is highly soluble and its use results in electrolytes with comparable and, in some cases, superior conductivity and properties to those with $LiPF_6$ (Table 1.1 and Fig. 1.14) [5]. But the potential to reduce the anion As^{V} oxidation state to the highly toxic As^{III} or As^{0} oxidation states, as noted above, has largely limited the commercial use of $LiAsF_6$ [177].

$LiAlCl_4$ has been widely used for Li/SO_2Cl_2 batteries [178] (as has $LiGaCl_4$ [179–186]), but $LiAlCl_4$ has also been studied for use with intercalation cathodes. For example, an electrolyte composed of $LiAlCl_4{\cdot}3SO_2$ was used for a $Li/LiCoO_2$ cell [187, 188]. This electrolyte has a very high conductivity (70–80 mS cm^{-1} at 0–20 °C), but the salt undergoes a degradation side reaction to produce Cl_2 which then reacts with Li metal to form LiCl [187, 188]:

$$\mathrm{LiAlCl_4 \rightarrow AlCl_3 + 1/2\,Cl_{2(dissolved)} + Li^+ + e^-}$$

$$\mathrm{1/2\,Cl_{2(dissolved)} + Li_{(s)} \rightarrow LiCl_{(s)}}$$

A diverse range of analogues of BF_4^- anions (Fig. 1.17) has been reported with the replacement of fluorine atoms with perfluoroalkyl chains. The conductivity of 1 M salt in EC:EMC electrolytes with lithium salts with these anions is given in Table 1.2. Perhaps contrary to expectations, it is interesting to note that the electrolyte conductivity increases with increasing size/mass of the anions. One might instead expect that bulkier anions would be less mobile, thus decreasing the conductivity. This increase in the conductivity may be due to decreased ionic association interactions with increasing perfluoroalkyl chain lengths and numbers (Figs. 1.1 and 1.2). A separate publication, however, indicated that an electrolyte with $LiBF_3C_2F_5$ (*LiFAB*) (Figs. 1.16e and 1.17a) has a lower conductivity than the corresponding $LiBF_4$ electrolyte (1 M salt in EC:EMC 30:70 v:v) above 0 °C, but a higher conductivity at < −20 °C [190]. Yet another publication indicates that LiFAB electrolytes (EC:EMC 30:70 v:v) are more conductive than $LiBF_4$ electrolytes (but less conductive than $LiPF_6$-based electrolytes) from −10 to 25 °C [192]. The LiFAB salt has

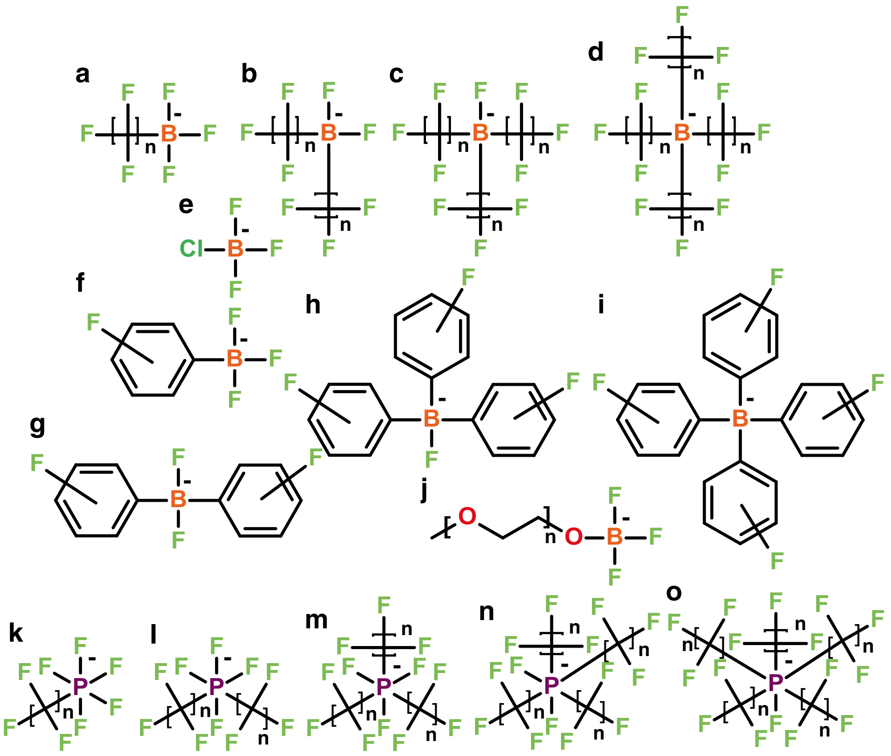


Fig. 1.17 Examples of analogues of BF_4^- and PF_6^- anions: (**a**) $BF_3(C_nF_{2n+1})^-$ [189–193], (**b**) $BF_2(C_nF_{2n+1})_2^-$ [189, 191], (**c**) $BF(C_nF_{2n+1})_3^-$ [189, 191], (**d**) $B(C_nF_{2n+1})_4^-$ [189, 194, 195], (**e**) BF_3Cl^- [196], (**f**) $BF_3(C_6H_nF_{5-n})^-$ [160, 197, 198], (**g**) $BF_2(C_6H_nF_{5-n})_2^-$ [160, 197, 198], (**h**) $BF(C_6H_nF_{5-n})_3^-$ [160, 197, 198], (**i**) $B(C_6H_nF_{5-n})_4^-$ [160, 198–201], (**j**) $BF_3O(C_2H_4O)_nCH_3^-$ [202], (**k**) $PF_5(C_nF_{2n+1})^-$ [203, 204], (**l**) $PF_4(C_nF_{2n+1})_2^-$ [203, 204], (**m**) $PF_3(C_nF_{2n+1})_3^-$ [70, 203–206], (**n**) $PF_2(C_nF_{2n+1})_4^-$ [63, 70, 203–209], and (**o**) $PF(C_nF_{2n+1})_5^-$ [204]

Table 1.2 Conductivity of electrolytes with various lithium salts at 20 °C and 1 M salt in EC:EMC (25:75 v:v) [189]

Lithium salt (anion)	Conductivity (mS cm^{-1})	Lithium salt (anion)	Conductivity (mS cm^{-1})
PF_6^-	8.52		
BF_4^-	2.91		
$BF_3(CF_3)^-$	3.21	$BF_3(C_2F_5)^-$	3.31
$BF_2(CF_3)_2^-$	4.02	$BF_2(C_2F_5)_2^-$	4.62
$BF(CF_3)_3^-$	5.11	$BF(C_2F_5)_3^-$	6.89
$B(CF_3)_4^-$	7.52	$B(C_2F_5)_4^-$	8.55
$BF_3(SO_2CF_3)^-$	4.47	$BF_3(SO_2C_2F_5)^-$	3.98
$BF_2(SO_2CF_3)_2^-$	5.83	$BF_2(SO_2C_2F_5)_2^-$	5.23
$BF(SO_2CF_3)_3^-$	7.62	$BF(SO_2C_2F_5)_3^-$	7.18
$B(SO_2CF_3)_4^-$	8.55	$B(SO_2C_2F_5)_4^-$	8.21

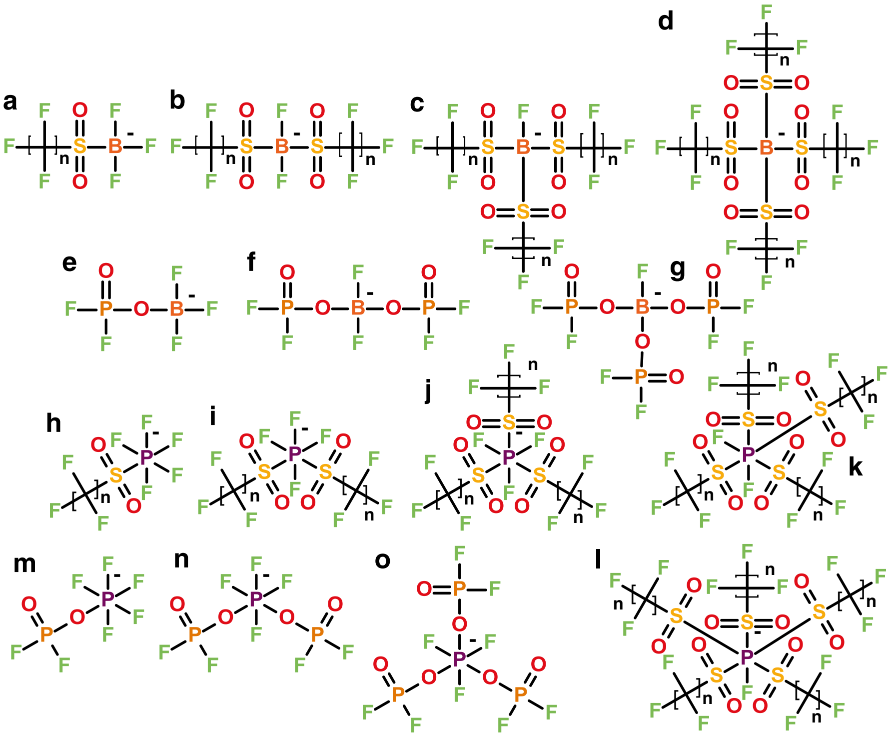


Fig. 1.18 Examples of BF_4^- and PF_6^- analogue anions with perfluoroalkylsulfonyl and phosphorodifluoridato groups: (**a**) $BF_3(SO_2C_nF_{2n+1})^-$ [189], (**b**) $BF_2(SO_2C_nF_{2n+1})_2^-$ [189], (**c**) $BF(SO_2C_nF_{2n+1})_3^-$ [189], (**d**) $B(SO_2C_nF_{2n+1})_4^-$ [189], (**e**) $BF_3(PO_2F_2)^-$ [210], (**f**) $BF_2(PO_2F_2)_2^-$ [210], (**g**) $BF(PO_2F_2)_3^-$ [210], (**h**) $PF_5(SO_2C_nF_{2n+1})^-$ [204], (**i**) $PF_4(SO_2C_nF_{2n+1})_2^-$ [204], (**j**) $PF_3(SO_2C_nF_{2n+1})_3^-$ [204], (**k**) $PF_2(SO_2C_nF_{2n+1})_4^-$ [204], (**l**) $PF(SO_2C_nF_{2n+1})_5^-$ [204], (**m**) $PF_5(PO_2F_2)^-$ [210], (**n**) $PF_4(PO_2F_2)_2^-$ [210], and (**o**) $PF_3(PO_2F_2)_3^-$ [210]

good compatibility with Al at high potential, as well as a high stability with a graphite anode and nickel oxide-based cathode. The cell cycling behavior is comparable to an electrolyte with $LiPF_6$ and far better than one with $LiBF_4$. Cells with LiFAB also had improved capacity retention relative to cells with $LiPF_6$ after storage at 60 °C [190]. For cells with graphite anodes and $LiCoO_2$ cathodes, however, comparable performance to $LiPF_6$ electrolytes was found at room temperature, but inferior performance was found at elevated temperature reportedly due to a degradation reaction of the LiFAB electrolyte with the cathode [192]. Electrolytes with variants of BF_4^- anions with perfluoroalkylsulfonyl groups ($-SO_2C_nF_{2n+1}$) (Fig. 1.18a–d) have conductivity values somewhat higher than those with anions with the corresponding perfluoroalkyl groups (Table 1.2) [189]. Analogous anions with the fluorine atoms of BF_4^- replaced with $-PO_2F_2$ groups have also been reported (Fig. 1.18e–g) [210].

The $LiBF_3Cl$ salt (Figs. 1.16i and 1.17e) has a higher solubility than $LiBF_4$ in aprotic solvents and hinders crystallization of electrolytes to a greater extent at low temperature [196]. In common with $LiBF_4$, the $LiBF_3Cl$ salt passivates Al well at high potential. The salt also makes a more favorable SEI with graphite than comparable electrolytes with $LiBF_4$ [196].

Lithium salts with fluorinated phenylfluoroborate anions have been reported (Fig. 1.17f–i) [198]. The conductivity values for 0.5 M salt in PC:DMC (v:v) electrolytes at 30 °C are reported to be $LiBF_4$ (3.7 mS cm^{-1}), $LiBF_3(C_6F_5)$ (Fig. 1.17f) (4.0 mS cm^{-1}), and $LiBF_3C_3F_7$ (Fig. 1.17a) (6.1 mS cm^{-1}), but the t_{Li+} values are 0.31, 0.71, and 0.43, respectively [198]. The lithium tetrakis(pentafluorophenyl)borate salt (*LiBArF*) (i.e., $LiB(C_6F_5)_4$) (Figs. 1.16l and 1.17i) [199, 200] has a very weakly coordinating anion with 20 fluorine atoms per Li^+ cation. Although this salt does form crystalline solvates with fully solvated Li^+ cations (i.e., $(AN)_4$:$LiB(C_6F_5)_4$ [211] and $(Et_2O)_4$:$LiB(C_6F_5)_4$ [212]), it also crystallizes as solvates in which the Li^+ cations are partially coordinated by the anion fluorine atoms (i.e., $(Et_2O)_1$:$LiB(C_6F_5)_4 \cdot CH_2Cl_2$ [212], $(\text{toluene})_1$:$LiB(C_6F_5)_4\cdot$toluene [212], and $(\text{benzene})_1$:$LiB(C_6F_5)_4$ [213]). Thus, the extensive fluorination of the anion actually facilitates the coordination of the anion to the Li^+ cations (relative to $LiBPh_4$). The properties of a wide variety of different $LiBR_4$ salts with varying alkyl and/or aryl groups and substituents ($–CH_3$, $–OCH_3$, –F, $–CF_3$) have been extensively explored as electrolytes by researchers at Exxon Research and Engineering Company in the early 1980s [161, 214]. The anodic oxidative stability of the salts varies by more than 1.6 V depending upon the R group, with aryl groups resulting in higher stability than alkyl groups and the addition of electron-withdrawing substituents further increasing the stability [157, 160]. Related salts have been reported, such as $LiB(OC_6F_5)_4$ [215] and $LiB(SC_6F_5)_4$ [215, 216], but these salts tend to result in PEO-based electrolytes with a very low conductivity.

Lithium trifluoroalkoxyborate salts (Fig. 1.17j) are liquid at room temperature with a neat salt conductivity on the order of 10^{-4} S cm^{-1} [202]. Carbonate-based electrolytes (EC:PC:DMC 1:1:3 v:v:v) with these salts have a conductivity >3 mS cm^{-1} at 20 °C (higher than comparable electrolytes with $LiBF_4$) [202].

Analogues of PF_6^- anions (Fig. 1.17k–o) have also been reported [63, 70, 203–209]. Electrolytes (1 M salt in EC:DMC 50:50 w:w) with the $LiPF_3(C_2F_5)_3$ (*LiFAP*) salt (Figs. 1.16m and 1.17m) have a conductivity which is only slightly lower than those with $LiPF_6$ [70]. Half cells with Li metal and $LiMn_2O_4$ cycled better with the electrolyte with LiFAP (relative to those with $LiPF_6$) [70, 208]. LiFAP was found to not undergo hydrolysis (in sharp contrast to $LiPF_6$) and to have improved thermal stability relative to $LiPF_6$ [63, 70, 207, 209]. In addition, carbonate solvent-based electrolytes with a mixture of LiFAP and $LiPF_6$ were reported to have superior cycling performance (relative to comparable electrolytes with the individual LiFAP and $LiPF_6$ salts), especially at 80 °C [206]. Anions in which the PF_6^- fluorine atoms have been replaced with $–SO_2C_nF_{2n+1}$ or $–PO_2F_2$ groups have also been reported (Fig. 1.18h–o) [204, 210].

1.6 Advanced Salts—Perfluoroalkylacetates, -Sulfonates, and -Phosphates

The experimental gas-phase acidity values (ΔG_{acid} in kcal mol^{-1}) for alkyl- and perfluoroalkylacetates and sulfonates follow the order HCO_2CH_3 (341.1) < HCO_2CF_3 (316.3) ≤ HSO_3CH_3 (315.0) < HSO_3F (299.8) < HSO_3CF_3 (299.5) (Fig. 1.1) [21, 26]. The fluorocarbonate anion (i.e., CO_2F^-) is highly reactive and may be considered to be a fluorine anion, F^-, solvated by CO_2 [217]. Other perfluoroalkylacetate anions (i.e., $CO_2C_nF_{2n+1}^-$) (Figs. 1.19a and 1.20a) have not been used to any significant extent for lithium battery electrolytes. The lithium salts with these anions tend to be highly aggregated (extensive anion…Li^+ cation coordination interactions) in PC electrolytes [218]. This is in agreement with the results noted above for the $(AN)_n$–$LiCO_2CF_3$ mixtures which have a very low solvation number over the entire concentration range (Fig. 1.5) and very low conductivity relative to other $(AN)_n$–LiX mixtures (Fig. 1.11).

Fluorosulfonic acid (i.e., HSO_3F) (Fig. 1.19b) was first reported in 1918 [236]. $LiSO_3F$-based electrolytes with several aprotic solvents were found to have a similar oxidative stability to electrolytes with other lithium salts such as $LiSO_3CF_3$ [237]. The conductivity of 1 M $LiSO_3F$ electrolytes with PC and GBL is 1.4 and 3.6 mS cm^{-1}, respectively [237]. For a mixed-solvent electrolyte, 1 M $LiSO_3F$ in GBL:DME (1:1 mol:mol), however, the conductivity is 7.4 mS cm^{-1} at 25 °C [237] (to be compared with 1 M GBL:DME (1:1 mol:mol) electrolytes with $LiBF_4$, $LiPF_6$, and $LiAsF_6$ which have conductivity values of 7.7, 11.2, and 11.8 mS cm^{-1} [238]). The crystal structure for $LiSO_3F$ has been reported [239].

Trifluoromethanesulfonic acid (i.e., HSO_3CF_3) (Fig. 1.19b) and the corresponding sodium salt were first reported in 1954 [240, 241]. A 3M patent in 1956 then reported the preparation of a variety of perfluoroalkylsulfonic acids and the corresponding sodium and potassium salts [242]. The use of $LiSO_3CF_3$ in battery electrolytes began in the 1970s and early 1980s [243, 244]. Lithium salts with perfluoroalkylsulfonate anions (Figs. 1.19b and 1.20b, c) tend to be more dissociated

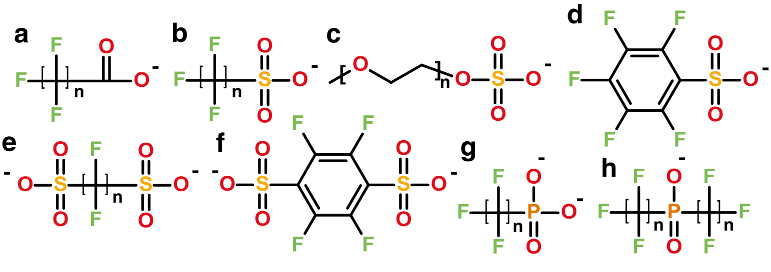


Fig. 1.19 Examples of fluoroalkylacetate, -sulfonate, and -phosphate anions: (**a**) $CO_2C_nF_{2n+1}^-$ [218], (**b**) $SO_3C_nF_{2n+1}^-$ [219–222], (**c**) $SO_4(C_2H_4O)_nCH_3^-$ [223, 224], (**d**) $SO_3(C_6F_5)^-$ [175, 221], (**e**) $SO_3(CF_2)_nSO_3^{2-}$ [175, 222, 225], (**f**) $SO_3(C_6F_4)SO_3^{2-}$ [226], (**g**) $PO_3(C_nF_{2n+1})^{2-}$ [72, 227–232], and (**h**) $PO_2(C_nF_{2n+1})_2^-$ [72, 227, 230, 233–235]

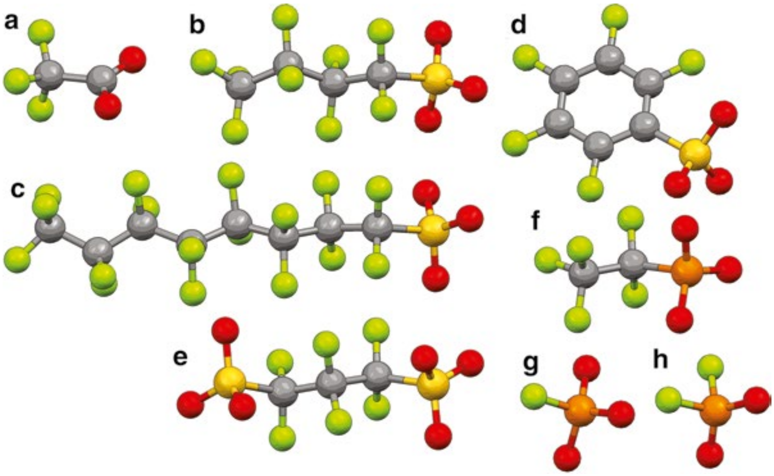


Fig. 1.20 Anion structures: (**a**) $CO_2CF_3^-$, (**b**) $SO_3C_4F_9^-$, (**c**) $SO_3C_8F_{17}^-$, (**d**) $SO_3(C_6F_5)^-$, (**e**) $SO_3C_3F_6SO_3^{2-}$, (**f**) $PO_3C_2F_5^{2-}$, (**g**) PO_3F^{2-}, and (**h**) $PO_2F_2^-$ (C—*gray*, O—*red*, F—*light green*, P—*orange*, S—*yellow*)

than those with perfluoroalkylacetate anions due to the larger size of the sulfur atoms relative to carbon (making the anion "softer") and more extensive resonance (and thus charge delocalization) due to the additional oxygen atom. This results in the sulfonate salts having a higher conductivity than the corresponding acetate salts (Table 1.3) [219, 221]. The conductivity of electrolytes with the perfluoroalkylsulfonate anions is not correlated with the mass of the anions (Table 1.3). All of the electrolytes with these salts, however, have a conductivity which is significantly lower than for the comparable electrolyte with $LiPF_6$ (Tables 1.1 and 1.3, Fig. 1.14). The crystal structure for $LiSO_3CF_3$ is known for both low- and high-temperature phases [245–247]. The conductivity of amorphous PEO-based polymer electrolytes with the perfluoroalkylsulfonate salts follows the order [220]:

$$\mathrm{LiSO_3C_{10}F_{21} < LiSO_3CF_3 < LiSO_3C_8F_{17} < LiSO_3C_4F_9 < LiTFSI}$$

This ordering is likely due to a reduction in the partial negative charge on the anion oxygen atoms upon increasing chain length from $–CF_3$ to $–C_4F_9$ (which reduces the ionic association tendency of the anions). An additional increase in the chain length (i.e., $–C_8F_{17}$ and $–C_{10}F_{21}$), however, does not then significantly decrease the ionic association interactions further and the more bulky anions decrease the anion mobility (and perhaps the Li^+ cation mobility due to the correlated interactions of the cations with the anions), thus lowering the conductivity (relative to $LiSO_3C_4F_9$).

Note that lithium salts with nonfluorinated alkylsulfonate (e.g., $LiSO_3CH_3$) or benzenesulfonate (e.g., $LiSO_3(C_6H_5)$) anions have a very low solubility in aprotic solvents and a correspondingly low conductivity (Table 1.3) [219]. Lithium salts with oligoethersulfate anions (Fig. 1.19c) are soluble in EC:DMC mixtures, but these

Table 1.3 Conductivity of electrolytes with various lithium salts at 25 °C and 0.1 M salt in PC:DME (1:2 v:v) [219, 221]

Lithium salt (anion)	Conductivity (mS cm^{-1})	Molecular weight
PF_6^-	4.4	152
$CO_2CF_3^-$	0.4	120
$SO_3CH_3^-$	[a]	102
$SO_3CF_3^-$	2.3	156
$SO_3C_4F_9^-$	2.3	306
$SO_3(C_6H_5)^-$	0.1–0.2[b]	164
$SO_3(C_6F_5)^-$	1.1	254
$SO_3C_8F_{17}^-$	1.9	506
$N(COCF_3)_2^-$	0.8	215
$N(SO_2CF_3)_2^-$	4.0	287
$N(SO_2C_2F_5)_2^-$	3.8	387
$N(SO_2C_4F_9)(SO_2CF_3)^-$	3.5	437
$N(SO_2CF_3)(C_6F_4SO_2F)^-$	3.0	347
$N(SO_2CF_3)(SO_2C_8F_{17})^-$	3.2	637
$N(SO_2OCH_2CF_3)_2^-$	3.0	347
$N(SO_2OCH_2CF_2CF_3)_2^-$	3.0	447
$N(SO_2OCH_2CF_2CF_2H)_2^-$	2.9	411
$N(SO_2OCH(CF_3)_2)_2^-$	3.1	483
$C(SO_2CF_3)_3^-$	3.6	418
$C(SO_2OCH_2CF_3)_3^-$	2.9	508
$B(C_6H_3\text{-}3,5\text{-}(CF_3)_2)_4^-$	2.7	870
$PO_2(C_2F_5)_2^-$	0.6	308

[a] Practically insoluble
[b] ca. 0.02 M

have a much lower conductivity ($<10^{-3}$ S cm^{-1} at 30 °C) than comparable electrolytes with $LiPF_6$ [223, 224]. The lithium salt with fluorinated benzenesulfonate anions (i.e., $LiSO_3(C_6F_5)$) (Figs. 1.19d and 1.20d) is somewhat more soluble in aprotic solvents, but the conductivity of such electrolytes is relatively low (lower than for electrolytes with $LiSO_3CF_3$) (Table 1.3) [219, 248].

Monofluorophosphoric acid (i.e., H_2PO_3F) (Figs. 1.19g and 1.20g) and difluorophosphoric acid (i.e., HPO_2F_2) (Figs. 1.19h and 1.20h) were first reported in 1929 and 1927, respectively [227–229, 235]. Lithium salts with these anions have been prepared [230, 231, 233, 234]. The related $HPHO_2F$ acid and corresponding $LiPHO_2F$ salt, however, were much more challenging to prepare [249]. Trifluoromethanephosphonic acid (i.e., $H_2PO_3CF_3$) (Fig. 1.19g) and bis(trifluoromethane)phosphonic acid (i.e., $HPO_2(CF_3)_2$) (Fig. 1.19h) were synthesized in 1954–1955 [250, 251]. The relative acidity of these anions was reported to follow the order [251]:

$$HNO_3, HCO_2CF_3, HCO_2C_3F_7 < H_2AsO_3CF_3 < HAsO_2\left(CF_3\right)_2$$

$$H_2PO_3CF_3, HCl < H_2SO_4 < HBr < HPO_2\left(CF_3\right)_2 < HClO_4$$

The lithium trifluoromethanephosphate salt (i.e., $Li_2PO_3CF_3$) (Fig. 1.19g) has also been synthesized [252]. This salt was reportedly soluble, forming a 1 M electrolyte with a PC:DME mixture which was utilized for battery cycling [252]. Perfluorodiphenylphosphinic acid (i.e., $HPO_2(C_6F_5)_2$) and the crystal structure of the corresponding potassium salt have also been reported [253].

1.7 Advanced Salts—Imides, Methides, and Phosphorylimides

The bis(fluorocarbonyl)imide acid (i.e., $H[N(COF)_2]$) (Fig. 1.21a) was first reported in 1973 [291], but anions with the X–CO–N–CO–X (X = C or F) backbone have not received much attention for battery electrolytes. In contrast, a diverse range of lithium salts with imide (sometimes called amide) anions with the X–SO_2–N–SO_2–X (X = C or F) backbone have been prepared. This difference in focus is due to the difference, for example, in the acidifying capability of the –$COCF_3$ and –SO_2CF_3 groups (Fig. 1.2)—experimental gas-phase acidity values (ΔG_{acid} in kcal mol^{-1}) are $HN(COCF_3)_2$ (307.5) and $HN(SO_2CF_3)_2$ (291.8) (ref: H_2SO_4 (302.2)) (Fig. 1.2) [20, 26]. The X–SO_2–N–SO_2–X backbone is able to adopt two low-energy conformations (Fig. 1.3f) [114–117]. This flexibility, combined with the extensive charge delocalization due to resonance and the electron-withdrawing fluorine atoms [20, 26], tends to make these lithium salts highly soluble. In addition, solvent–salt mixtures with such salts often form crystalline solvates with a low T_m (in contrast with $LiPF_6$) or crystallinity gaps (concentration ranges in which it is difficult or impossible to crystallize some or all of the electrolyte) (Fig. 1.4).

Lithium bis(methanesulfonyl)imide (or dimesylamide) (i.e., $LiN(SO_2CH_3)_2$) (Figs. 1.21b and 1.22a) [258, 292–295] and lithium bis(butanesulfonyl)imide (i.e., $LiN(SO_2C_4H_9)_2$) [258] have been reported. In sharp contrast to the lithium salts with tetraalkylborate anions which are highly soluble in aprotic solvents, lithium salts with nonfluorinated bis(alkanesulfonyl)imide anions have poor solubility in cyclic/acyclic carbonate and ether solvents [258]. This is likely due to the poor Li^+ cation-coordinating ability of the tetraalkylborate anions—thus favoring solvent coordination to the Li^+ cations, whereas the nonfluorinated bis(alkanesulfonyl)imide anions instead readily coordinate the Li^+ cations with the anion oxygen atoms and the lack of electron-withdrawing fluorine atoms results in high electron density on the oxygen atoms (i.e., strong ionic association tendency)—thus restricting the solvent coordination to the Li^+ cations. $LiN(SO_2CH_3)_2$ is insoluble in an EC:DMC mixture and has poor solubility in DMSO (<0.1 M), while $LiN(SO_2C_4H_9)_2$ has low solubility in EC:DMC (<0.1 M) and fair solubility in DMSO (~0.3 M) [258]. The conductivity at 25 °C of DMSO electrolytes with these salts (3.2 mS cm^{-1}) was considerably lower than for a 0.5 M $LiPF_6$ electrolyte with DMSO (9.8 mS cm^{-1}). In addition, ionic liquid salts with the $N(SO_2CH_3)_2^-$ anion have a higher viscosity, lower conductivity, lower thermal stability, and lower electrochemical stability than the corresponding salts with the $TFSI^-$ anion [296].

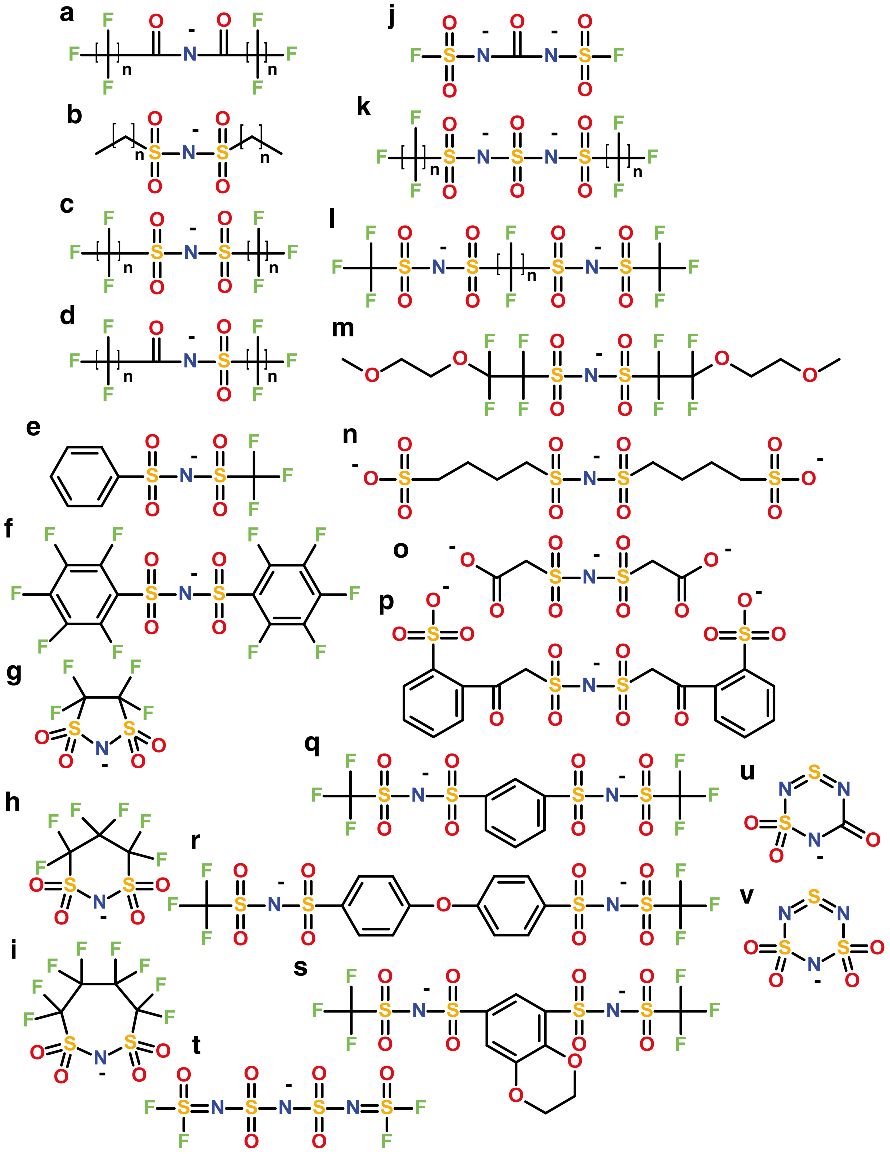


Fig. 1.21 Examples of imide anions: (**a**) $N(COC_nF_{2n+1})_2^-$ [254–257], (**b**) $N(SO_2C_nH_{2n+1})_2^-$ [258], (**c**) $N(SO_2C_nF_{2n+1})_2^-$ [259–270], (**d**) $N(COC_nF_{2n+1})(SO_2C_nF_{2n+1})^-$ [256, 257, 271–273], (**e**) $N(SO_2(C_6H_5))(SO_2CF_3)^-$ [274–276], (**f**) $N(SO_2(C_6F_5))_2^-$ [277], (**g**) $N(SO_2C_2F_4SO_2)^-$ [278, 279], (**h**) $N(SO_2C_3F_6SO_2)^-$ [269, 278, 279], (**i**) $N(SO_2C_4F_8SO_2)^-$ [278, 279], (**j**) $CO(NSO_2F)_2^{2-}$ [280], (**k**) $SO_2(NSO_2C_nF_{2n+1})_2^{2-}$ [281–283], (**l**) $(CF_2)_n(SO_2NSO_2CF_3)_2^{2-}$ [284, 285], (**m**) $N(SO_2C_2F_4O(C_2H_4O)CH_3)_2^-$ [277], (**n**) $N(SO_2C_4H_8SO_3)_2^{3-}$ [286], (**o**) $N(SO_2CH_2CO_2)_2^{3-}$ [286], (**p**) $N(SO_2CH_2CO(C_6H_4)SO_3)_2^{3-}$ [286], (**q**) $(C_6H_4)(SO_2NSO_2CF_3)_2^{2-}$ [287], (**r**) $O((C_6H_5)SO_2NSO_2CF_3)_2^{2-}$ [287], (**s**) $(C_6H_2)(OC_2H_2O)(SO_2NSO_2CF_3)_2^{2-}$ [287], (**t**) $N(SO_2NSOF_2)_2^-$ [288], (**u**) $N(CONSNSO_2)^-$ [289], and (**v**) $N(SO_2NSNSO_2)^-$ [290]

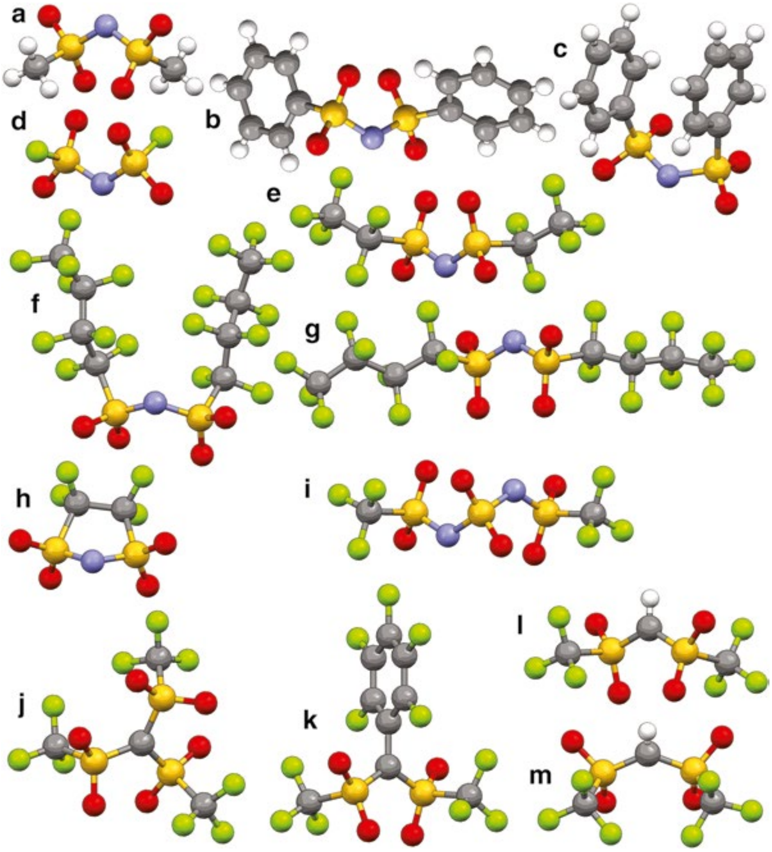
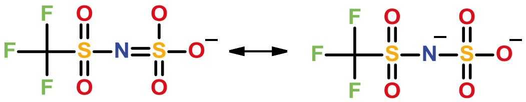
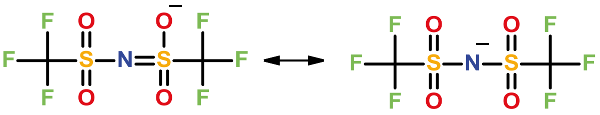


Fig. 1.22 Anion structures: (**a**) $N(SO_2CH_3)_2^-$ (*trans* conformation), (**b**) $N(SO_2(C_6H_5))_2^-$ (*trans* conformation), (**c**) $N(SO_2(C_6H_5))_2^-$ (*cis* conformation), (**d**) $N(SO_2F)_2^-$ (FSI^-), (**e**) $N(SO_2C_2F_5)_2^-$ ($BETI^-$) (*trans* conformation), (**f**) $N(SO_2C_4F_9)_2^-$ (*cis* conformation), (**g**) $N(SO_2C_4F_9)_2^-$ (*trans* conformation), (**h**) $N(SO_2C_2F_4SO_2)^-$, (**i**) $SO_2(NSO_2CF_3)_2^{2-}$, (**j**) $C(SO_2CF_3)_3^-$ ($TriTFSM^-$), (**k**) $C(SO_2CF_3)_2(C_6F_5)^-$, (**l**) $CH(SO_2CF_3)_2^-$ ($TFSM^-$) (*trans* conformation), and (**m**) $CH(SO_2CF_3)_2^-$ ($TFSM^-$) (*cis* conformation) (C—*gray*, N—*blue*, O—*red*, F—*light green*, S—*yellow*)

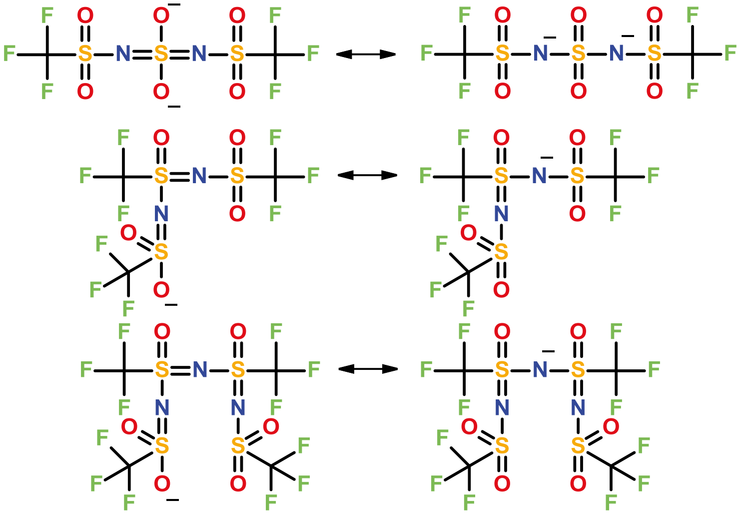
The $=NSO_2CF_3$ group has a very strong acidifying effect when replacing an =O [26]. The (trifluorosulfonyl)(sulfonate)imide anion [297] can be viewed as the SO_4^{2-} anion with a $=NSO_2CF_3$ group replacing an =O:



Similarly, the $TFSI^-$ anion can be viewed as the $SO_3CF_3^-$ anion with a $=NSO_2CF_3$ group replacing an =O:



Additional $=NSO_2CF_3$ groups give [298, 299]



Fluorination of the imide anions results in lithium salts with exceptionally high solubility in common aprotic solvents. Bis(trifluoromethanesulfonyl)imide acid or HTFSI (i.e., $HN(SO_2CF_3)_2$) (Figs. 1.3f and 1.21c) was first reported in 1982 by Foropoulos and DesMarteau [300, 301], while the longer chain anions—$N(SO_2R_F)_2^-$—were reported a decade earlier by Meussdorffer and Niederprum [302]. In 1990, Armand patented the synthesis of LiTFSI and related fluorinated sulfonyl imide salts [303]. *LiTFSI* is now the most widely studied salt for this class of anions, but lithium bis(perfluoroethanesulfonyl)imide (*LiBETI*) (i.e., $LiN(SO_2C_2F_5)_2$) (Figs. 1.21c and 1.22e) has also been widely examined [49, 69, 207, 262, 304–310] with more than 200 publications reported for this latter salt. Electrolytes with LiBETI have a lower conductivity than those with $LiPF_6$ or LiTFSI (Table 1.4), but this salt, like LiTFSI, has an exceptionally high thermal stability and does not undergo hydrolysis due to the high stability of the C–F bonds. In addition, unlike LiTFSI, electrolytes with aprotic solvents and LiBETI are reported to not strongly corrode Al at high potential [66, 68, 311–313]. Numerous other perfluoroalkanesulfonyl imide anions have also been prepared [259, 314], including cyclic anions such as lithium cyclic-1,3-perfluoroethanedisulfonylimide (i.e., $LiN(SO_2C_2F_4SO_2)$) (Figs. 1.21g and 1.22h) and lithium cyclic-1,3-perfluoropropanedisulfonylimide (i.e., $LiN(SO_2C_3F_6SO_2)$)

Table 1.4 Conductivity of electrolytes with various lithium salts at 20 °C and 1 M salt in EC:DMC and EC:DEC (1:1 w:w) [259]

Lithium salt (anion)	Conductivity (mS cm^{-1})
In EC:DMC	
PF_6^-	9.41
$SO_3CF_3^-$	2.51
$N(SO_2CF_3)_2^-$	6.18
$N(SO_2C_2F_5)_2^-$	5.45
$N(SO_2C_4F_9)_2^-$	3.63
$N(SO_2CF_3)(SO_2C_4F_9)^-$	1.55
$N(SO_2C_2F_5)(SO_2C_4F_9)^-$	3.11
$N(SO_2C_3F_6SO_2)^-$	6.86
In EC:DEC	
PF_6^-	6.09
$SO_3CF_3^-$	1.63
$N(SO_2CF_3)_2^-$	4.24
$N(SO_2C_2F_5)_2^-$	3.95
$N(SO_2C_4F_9)_2^-$	2.34
$N(SO_2CF_3)(SO_2C_4F_9)^-$	1.10
$N(SO_2C_2F_5)(SO_2C_4F_9)^-$	2.28
$N(SO_2C_3F_6SO_2)^-$	4.95

(Fig. 1.21h)—PC:DME electrolytes with the latter have the highest conductivity of the imide salts noted in Table 1.4 [314]. The properties of most of these salts have not been extensively explored by the battery research community.

Anions with fluorosulfonyl groups (–SO_2F) have garnered tremendous interest in recent years. Chief among these is lithium bis(fluorosulfonyl)imide (*LiFSI*) (i.e., $LiN(SO_2F)_2$) (Figs. 1.21c and 1.22d) [156, 260, 263, 315–333]. Bis(fluorosulfonyl)-imide acid or HFSI (i.e., $HN(SO_2F)_2$) was first reported in 1962 [334], and, even though the synthesis of LiFSI was reported in 1995 [335], the limited availability and high cost of this salt have restricted its use in research studies until quite recently. Electrolytes with LiFSI typically have a conductivity equivalent to comparable electrolytes with $LiPF_6$ (making this one of the most conductive salts known) [156]. The thermal and hydrolytic stability of the FSI^- anion is lower than for the $TFSI^-$ anion due to the lower stability of the S–F bond (relative to a C–F bond), but the LiFSI salt has improved thermal/hydrolysis stability relative to $LiPF_6$ [156]. It was reported that use of the LiFSI salt in electrolytes results in severe Al corrosion at high potential [320], but it has recently been shown that this is likely due to chloride impurities in the salt rather than the LiFSI salt itself [156]. An additional favorable property (relative to $LiPF_6$) is the wide liquidus range of electrolytes with LiFSI. As for LiTFSI-based electrolytes, LiFSI-based electrolytes tend to form solvates with a low T_m or crystallinity gaps for specific electrolyte compositions.

Lithium salts with asymmetric anions may also be of interest as these tend to be more soluble and form solvates with a lower T_m than for salts with symmetric anions. Examples include lithium (fluorosulfonyl)(trifluoromethanesulfonyl)imide (*LiFTI* or LiFTA) (i.e., $LiN(SO_2F)(SO_2CF_3)$) (Fig. 1.21c) [263–265, 336] and lithium (fluorosulfonyl)(nonafluorobutanesulfonyl)imide (*LiFNFSI*) (i.e., $LiN(SO_2F)$

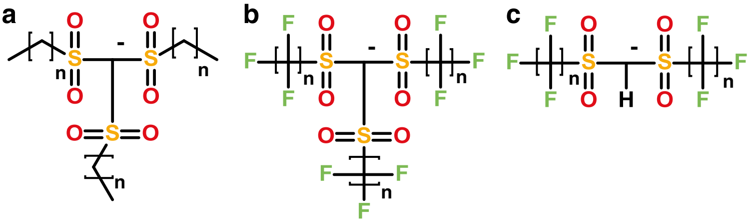


Fig. 1.23 Examples of methide anions: (**a**) $C(SO_2C_nH_{2n+1})_3^-$ [258, 339], (**b**) $C(SO_2C_nF_{2n+1})_3^-$ (TriTFSM$^-$) [111, 340–353], and (**c**) $CH(SO_2C_nF_{2n+1})_2^-$ (TFSM$^-$) [118, 341, 342, 354–360]

($SO_2C_4F_9$)) (Fig. 1.21c) [264, 265, 337]. The LiFNFSI salt has a high thermal stability (>200 °C), forms electrolytes with a high conductivity (comparable to those with $LiClO_4$), and does not significantly corrode the Al current collector at high potential. For battery testing, graphite/$LiCoO_2$ cells with LiFNFSI had a much improved cycling performance over cells with $LiPF_6$ when cycled at elevated temperature (60 °C). Asymmetric imide anions with carbonyl groups such as *TSAC*$^-$ (i.e., $N(COCF_3)(SO_2CF_3)^-$) (Fig. 1.21d) have also been reported [256, 266]. The experimental gas-phase acidity value (ΔG_{acid} in kcal mol^{-1}) for HTSAC (298.2) is somewhat higher than for HTFSI (291.8), but lower than that for HFSI (301.2) [26]. The TSAC$^-$ anion, however, has been found to have a poor electrochemical stability relative to other imide anions. The anodic oxidative stability was slightly lower relative to anions such as FTI$^-$ and TFSI$^-$, but the cathodic reductive stability of the TSAC$^-$ anion was notably poorer (almost 1 V less stable) [273, 338].

Many other variants of imide anions have been reported (Fig. 1.21). In general, these do not have improved properties or other advantages over more widely used anions (i.e., TFSI$^-$ and BETI$^-$). One possible exception to this may be the bis(trifluoromethanesulfonamido) sulfone anion (i.e., $LiSO_2(NSO_2CF_3)_2$) (Figs. 1.21k and 1.22i) [281]. If this dilithium salt has a high solubility in aprotic solvents, then it may offer an advantage in terms of having a greater Li$^+$/anion mass ratio with less fluorine atoms per Li$^+$ cation than for $LiPF_6$ and LiTFSI. The properties of the dilithium salt with this anion, however, are not yet available. The related acid $H_2[CO(NSO_2F)_2]$ (Fig. 1.21j) and the corresponding alkali metal salts (with Na$^+$ or K$^+$) have been reported [280].

Nonfluorinated lithium tris(alkanesulfonyl)methide salts have been examined for their suitability for battery electrolytes (Fig. 1.23a) [258]. The $LiC(SO_2CH_3)_3$ salt has poor solubility in EC:DMC (<0.1 M), but good solubility in DMSO (~0.5 M). Increasing the alkyl chain length from methyl to ethyl (i.e., $LiC(SO_2C_2H_5)_3$) increases the solubility, EC:DMC (~0.3 M) and DMSO (>4 M), while using asymmetric alkyl chain lengths (i.e., $LiC(SO_2CH_3)(SO_2C_2H_5)_2$) further increases the salt solubility, EC:DMC (~0.5 M) and DMSO (>4 M). These salts have a considerably lower conductivity (4.3 mS cm^{-1}), however, in 0.5 M DMSO electrolytes at 25 °C than that for the corresponding $LiPF_6$ electrolyte (9.8 mS cm^{-1}) [258].

Lithium tris(perfluoroalkanesulfonyl)methide salts have also received some attention for battery electrolytes, especially the salt with the $C(SO_2CF_3)_3^-$ anion (*TriTFSM*$^-$) (Figs. 1.22j and 1.23b). The experimental gas-phase acidity values

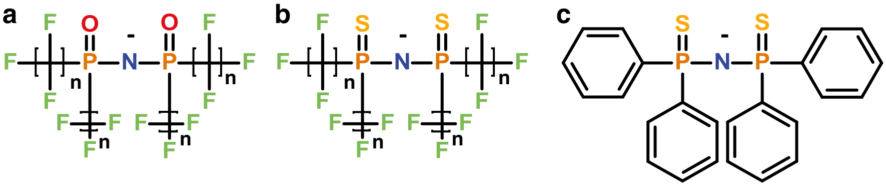
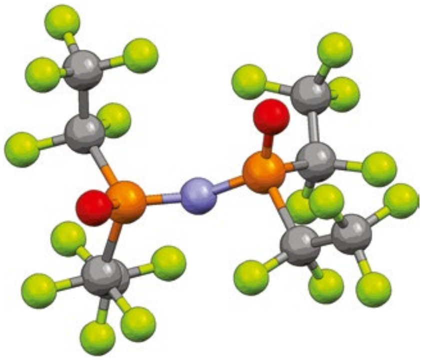


Fig. 1.24 Examples of phosphorylimide anions: (**a**) $N(PO(C_nF_{2n+1})_2)_2^-$ [361–366], (**b**) $N(PS(C_nF_{2n+1})_2)_2^-$ [367], and (**c**) $N(PS(C_6H_5)_2)_2^-$ [368–370]

(ΔG_{acid} in kcal mol^{-1}) are $HC(COCF_3)_3$ (300.6) and $HC(SO_2CF_3)_3$ (289.0) (ref: $HN(SO_2CF_3)_2$ (291.8)) (Fig. 1.2) [20, 26]. Despite the higher acidity of the TriTFSM$^-$ anion relative to TFSI$^-$ and calculations which indicate that the former anion will have a weaker Li$^+$ cation affinity (i.e., lower ionic association tendency), the conductivity of polyether-based electrolytes with LiTriTFSM is lower than for similar electrolytes with LiTFSI [111, 341]. This may perhaps be related to the greater size/mass of the TriTFSM$^-$ anion. For liquid 1 M electrolytes with EC:DMC (50:50 v:v), the conductivity (mS cm^{-1}) at 25 °C is $LiAsF_6$ (11.0), LiTFSI (9.0), and LiTriTFSM (7.1) [343]. Despite the recent attention devoted to LiFSI (i.e., $LiN(SO_2F)_2$), no published work is yet available regarding the properties of electrolytes with lithium tris(fluorosulfonyl)methide salt, $LiC(SO_2F)_3$ [340, 350], and the bis(fluoromethanesulfonyl)methane (i.e., $CH(SO_2F)_2^-$) anion has not yet been reported. Some limited studies have, however, been reported for the related bis(trifluoromethanesulfonyl)methide anion (*TFSM*$^-$) (i.e., $CH(SO_2CF_3)_2^-$) (Figs. 1.22l, m and 1.23c). The acid, bis(trifluoromethanesulfonyl)methane (i.e., $HCH(SO_2CF_3)_2$), was first prepared by Gramstad and Haszeldine in 1956 [355]. Poly(ethylene oxide) (PEO) electrolytes with the lithium salt (LiTFSM) (i.e., $LiCH(SO_2CF_3)_2$) were found in one report to have a lower oxidative and reductive electrochemical stability than for similar electrolytes with LiTFSI [341], whereas another study indicated that such electrolytes with LiTFSM were stable with a voltage stability window of approximately 4.5 V [359]. As for LiTFSI and LiTriTFSM, the LiTFSM salt tends to plasticize poly(ethylene oxide) resulting in amorphous electrolytes with a relatively high conductivity, although somewhat lower than for comparable LiTFSI electrolytes [341, 359].

Lithium perfluoroalkanephosphorylimide salts (Fig. 1.24a) have not yet been reported, but such anions have been explored using QC calculations [361] and the sodium and potassium salts with the $N(PO(C_2F_5)_2)_2^-$ anion (Fig. 1.25) have been prepared [365]. The lithium bis(difluorophosphoryl)imide salt (i.e., $LiN(POF_2)_2$) (Fig. 1.24a), however, has been synthesized [362, 363], as has the acid $HN(PSF_2)_2$ (Fig. 1.24b) [367]. Nonfluorinated alkyl or phenylphosphorylimide salts are also known, such as $LiN(PS(C_6H_5)_2)_2$ (Fig. 1.24c) [368–370] and $LiN(PO(C_6H_5)_2)(PS(CH_3)_2)$ [371]. No data is yet available, however, regarding the electrolyte characteristics of lithium salts with such anions.

Fig. 1.25 Anion structure: $N(PO(C_2F_5)_2)_2^-$ (C—*gray*, N—*blue*, O—*red*, F—*light green*, P—*orange*)



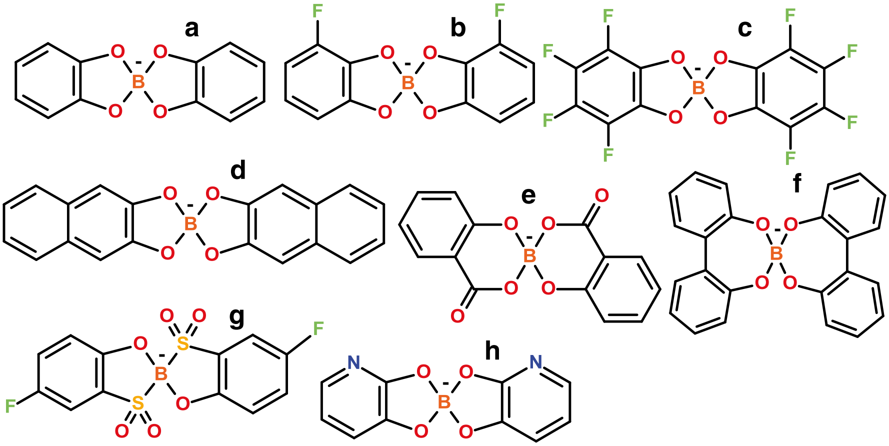


Fig. 1.26 Examples of chelated organoborate anions: (**a**) $B(O(C_6H_4)O)_2^-$ (BBB^-) [169, 372–378], (**b**) $B(O(C_6H_3F)O)_2^-$ ($FLBBB^-$) [373, 379, 380], (**c**) $B(O(C_6F_4)O)_2^-$ ($4FLBBB^-$) [373, 381], (**d**) $B(O(C_{10}H_6)O)_2^-$ (BNB^-) [56, 169, 373–375, 378], (**e**) $B(O(C_6H_4)CO_2)_2^-$ (BSB^-) [56, 373, 375, 382–385], (**f**) $B(O(C_6H_4)(C_6H_4)O)_2^-$ ($BBPB^-$) [56, 169, 373, 375], (**g**) $B(O(C_6H_3F)SO_2)_2^-$ (FSB^-) [386], and (**h**) $B(O(C_5NH_3)O)_2^-$ (BPB^-) [387]

1.8 Advanced Salts—Organoborates, -Phosphates, and -Aluminates

In 1995, Barthel and Gores reported a new class of inexpensive and chemically, electrochemically, and thermally stable salts based upon boron chelate complex anions with aromatic or aliphatic diols or carboxylic acids. The first such salt reported was lithium bis(1,2-benzenediolato(2-)-O,O′)borate (*LiBBB*) (Fig. 1.26a) [372]. The acid with this anion was originally reported in 1949 by Schafer [388]. The LiBBB salt has a high solubility in aprotic solvents (>1 M), but the oxidative stability is relatively low. A number of other nonfluorinated lithium salts with benzenediol nonfluorinated

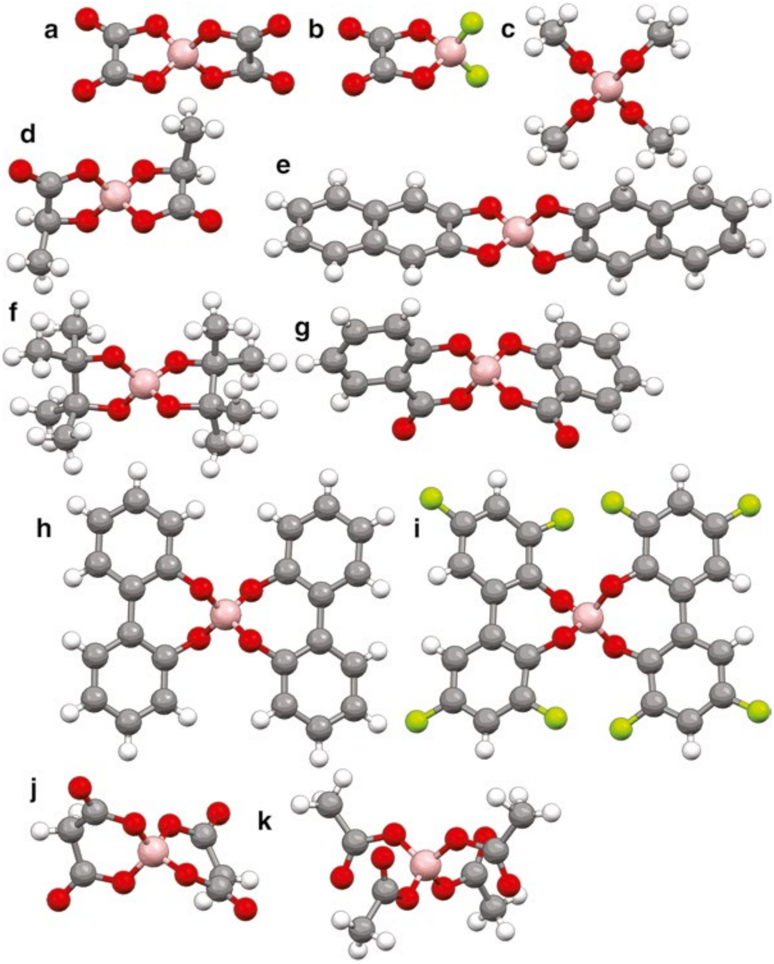


Fig. 1.27 Anion structures: (**a**) $B(CO_2CO_2)_2^-$ (BOB^-), (**b**) $BF_2(CO_2CO_2)_2^-$ ($DFOB^-$), (**c**) $B(OCH_3)_4^-$, (**d**) $B(CO_2CH(CH_3)O)_2^-$, (**e**) $B(O(C_{10}H_6)O)_2^-$ (BNB^-), (**f**) $B(OC(CH_3)_2C(CH_3)_2O)_2^-$, (**g**) $B(O(C_6H_4)CO_2)_2^-$ (BSB^-), (**h**) $B(O(C_6H_4)(C_6H_4)O)_2^-$ ($BBPB^-$), (**i**) $B(O(C_6H_2F_2)(C_6H_2F_2)O)_2^-$, (**j**) $B(CO_2CH_2CO_2)_2^-$(BMB^-), and (**k**) $B(CO_2CH_3)_4^-$ (B—*tan*, C—*gray*, O—*red*, F—*light green*)

chelates were subsequently prepared: bis(2,3-naphthalenediolato(2-)-O,O′)borate (*LiBNB*) (Figs. 1.26d and 1.27e), bis(salicylato(2-))borate (*LiBSB*) (Fig. 1.26e and 1.27g), and bis(2,2′-biphenyldiolato(2-)-O,O′)borate (*LiBBPB*) (Figs. 1.26f and 1.27h) [373]. The conductivity of electrolytes with these salts was found to increase in the order LiBBPB < LiBSB < LiBNB ~ LiBBB (Table 1.5) [169, 374, 375]. All of these electrolytes have a significantly lower conductivity than comparable electrolytes with $LiPF_6$, LiTFSI, and LiBETI (Table 1.5) [375]. The LiBSB-based electrolyte was found to have a relatively high Li cycling efficiency, however, in contrast to the other salts [375]. A number of crystalline solvates have been reported for the

Table 1.5 Conductivity of electrolytes with various lithium salts at 25 °C and 0.3 M salt in PC:DME and PC:2-MeTHF (1:1 equimolar) [375]

Lithium salt (anion)	Conductivity (mS cm^{-1})
In PC:DME	
PF_6^-	9.23
$N(SO_2CF_3)_2^-$	8.05
$N(SO_2C_2F_5)_2^-$	7.55
BBB^-	4.21
BNB^-	4.25
BSB^-	2.45
$BBPB^-$	1.09
in PC:2-MeTHF	
PF_6^-	6.57
$N(SO_2CF_3)_2^-$	5.97
$N(SO_2C_2F_5)_2^-$	5.48
BBB^-	3.07
BNB^-	2.97
BSB^-	1.28
$BBPB^-$	0.92

LiBBB and LiBSB salts: $(H_2O)_2(THF)_1$:LiBBB, $(H_2O)_1(AN)_1$:LiBBB, $(AN)_2$:LiBSB, and $(THF)_2$:LiBSB [389]. For the former solvate, the Li^+ cations are coordinated to the diol oxygen atoms, but for the latter solvate these oxygens do not participate in the cation coordination. Instead, the carbonyl oxygens are coordinated to the Li^+ cations.

The preparation of fluorinated chelated organoborate salts (i.e., $LiB(C_6H_{4-x}F_xO_2)$ demonstrated that with increasing fluorination ($x=0$, 1, or 4) (Fig. 1.26a, b, c) both the conductivity and oxidative stability increased [379, 381]. The oxidative stability limit (vs. Li/Li^+) was found to vary considerably for these salts: 3.6 V for BBB^-, 3.8 V for $FLBBB^-/BNB^-$, 4.1 V for $4FLBBB^-/BBPB^-$, and 4.5 V for BSB^- [373]. It was suggested that these anions anodically degrade on the cathode surface to form thin, electrically insulated, but Li^+ cation-conducting polymeric films which passify the electrode surface from further degradation of the salts or the solvents. A similar high salt solubility and oxidative stability limit was noted for the lithium bis(5-fluoro-2-olato-benzenesulfonato(2-)-O,O′)borate salt (*LiFSB*) (Fig. 1.26g) which has a stability limit of 4.6 V vs. Li/Li^+ [386]. The current density decreased upon repeated cycling on a Pt electrode due to electrode passivation. The salt also passivated an Al electrode at high potential. The introduction of a nitrogen to the benzenediol chelate—lithium bis(2,3-pyridinediolato(2-)-O,O′)borate (*LiBPB*) (Fig. 1.26h), however, resulted in a salt with low solubility in DMC and DEC (in contrast to the other salts prepared), but the solubility was higher in EC or PC [387]. The conductivity of electrolytes with LiBPB was lower than for the other borate salts, but the salt did passivate Pt and Al electrodes, as for the other salts studied [387].

The first organoborate salt to attract significant interest from the broader battery research community was lithium bis(oxalato)borate (*LiBOB*) (i.e., $LiB(CO_2CO_2)_2$) (Figs. 1.27a and 1.28a) [48, 79, 376, 380, 385, 390–484]. The first publication with

LiBOB was from Xu and Angell in 2001 [393], but a German patent application was filed for this salt by Metallgesellschaft AG in 1999 [548]. The acid and tetraalkylammonium salts with BOB$^-$ (called borodicatecholate) and related anions had been reported in 1994 by Ue [549], as well as earlier by others [550]. LiBOB electrolytes have a conductivity which is comparable to or lower than that for electrolytes with $LiBF_4$ [79, 414], moderate stability to hydrolysis, high electrochemical stability (>4.5 V vs. Li/Li^+), and high thermal stability [393, 394, 414, 448]. The salt has a relatively low solubility in acyclic carbonate solvents (i.e., the solubility limit is 0.8 M in EC:DMC 3:7) but is more soluble (>1 M) in nitrile, ester, and cyclic carbonate solvents [447]. The salt has been used as both a primary salt (replacement for $LiPF_6$) and an additive (addition of small amounts to $LiPF_6$ electrolytes). Notably, the electrode surface layers formed by this salt on both the anode and cathode enable cells with LiBOB to have excellent capacity retention when cycled at elevated temperature (≥60 °C) and when cycling cathodes up to 5 V (vs. Li^+/Li).

Lithium difluoro(oxalato)borate (*LiDFOB*) (also called lithium oxalyldifluoroborate (*LiODFB*)) (i.e., $LiBF_2(CO_2CO_2)$) (Figs. 1.27b and 1.28g), like LiBOB, has also received a great deal of attention from the lithium battery research community. This salt was first reported in Central Glass Company patents filed in 2000 [390–392] and then later in a US Army Research Laboratory (ARL) publication in 2006 [491]. Many studies have now demonstrated that LiDFOB is quite useful both as a primary salt (replacement for $LiPF_6$) and as an additive to $LiPF_6$ electrolytes [491–535]. LiDFOB has a higher solubility than LiBOB in linear carbonate solvents, but it is still lower than for other salts such as $LiBF_4$, LiTFSI, and $LiPF_6$. Electrolytes with the LiDFOB salt are better than those with LiBOB at passivating the Al current collector and also tend to have a higher conductivity (i.e., the conductivity of a 1 M LiDFOB electrolyte in EC:DMC (1:1 v:v) at 25 °C is 8.6 mS cm^{-1} [506], which is somewhat lower than the conductivity of comparable electrolytes with $LiPF_6$ or $LiClO_4$ (Table 1.1)). As for LiBOB, enhanced battery performance is noted upon addition of LiDFOB to electrolytes including the cyclability/stabilization of electrode materials (such as $LiFePO_4$ and $Li_4Ti_5O_{12}$) at elevated temperature (60 °C) and of high-voltage cathode materials when cycled to 5 V due to favorable surface layers formed on the anode and cathode [40].

In addition to LiBOB and LiDFOB, a wide variety of additional organoborate anions have been synthesized (Figs. 1.28 and 1.29). Only limited information is available about the properties of the corresponding lithium salts. The $LiB(CO_2C(CF_3)_2O)_2$ salt (Fig. 1.29a), however, reportedly does not undergo hydrolysis and is thermally stable at 100 °C for 1 month, and electrolytes with this salt do not corrode Al at high potential [545]. The conductivity of electrolytes with 1 M salt in EC:DMC 1:1 at 25 °C is 7.0, 8.3, 6.3, and 1.9 mS cm^{-1}, respectively, for the $LiB(CO_2C(CF_3)_2O)_2$, $LiBF_2(CO_2C(CF_3)_2O)$, $LiB(CO_2CH(CF_3)O)_2$, and $LiB(CO_2CH_2C(CF_3)_2O)_2$ salts (Fig. 1.29a–d) [545]. Note that the concentration for the latter salt is only 0.8 M (instead of 1 M) due to its limited solubility in EC:DMC. For the fluorinated salts in Fig. 1.29h–p, the maximum conductivity (mS cm^{-1}) (differing concentrations) for DME-based electrolytes at 25 °C is 5.88 (Fig. 1.29h), 5.39 (Fig. 1.29i), 6.57 (Fig. 1.29j), 6.89 (Fig. 1.29k), 6.39 (Fig. 1.29l), 7.89 (Fig. 1.29m),

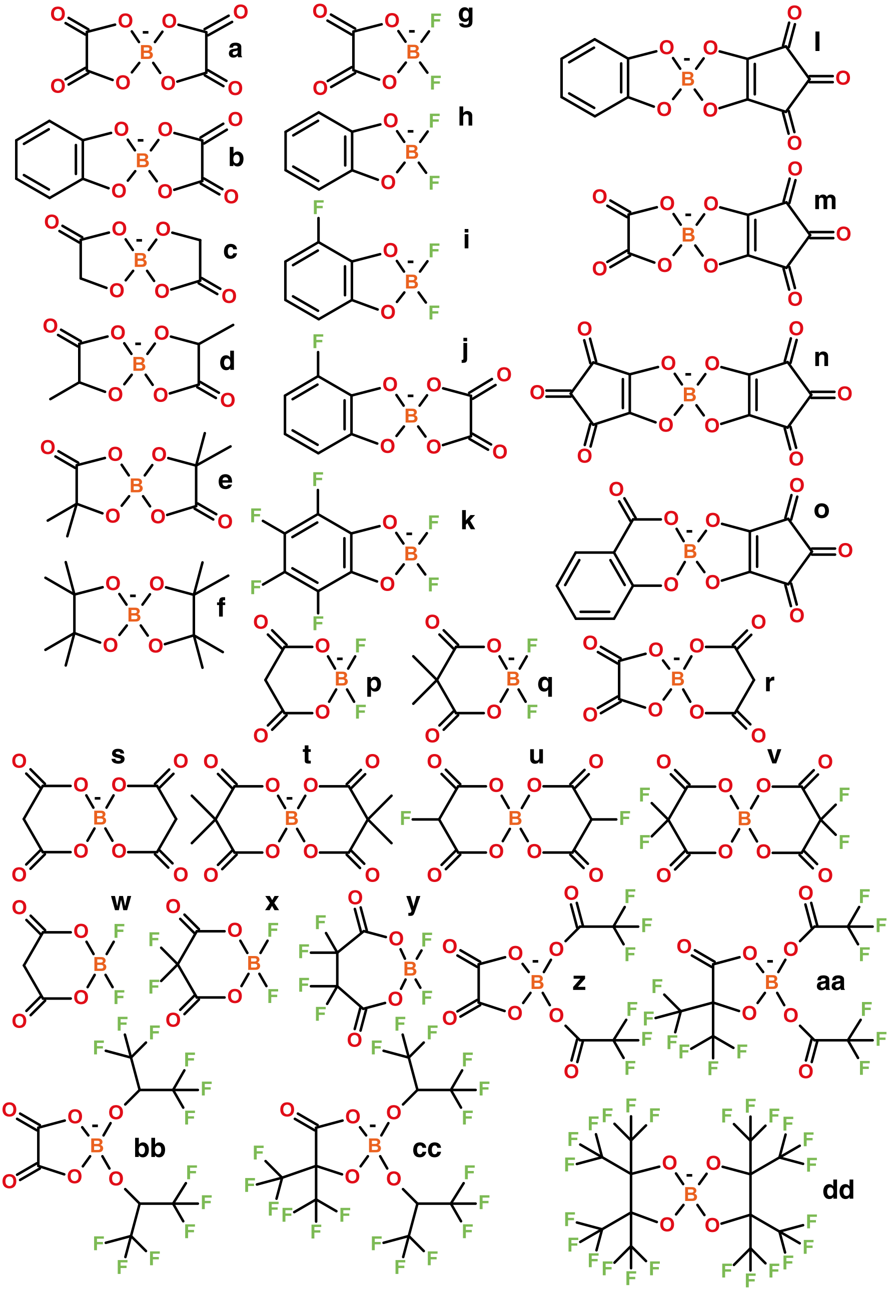


Fig. 1.28 Examples of chelated organoborate anions: (**a**) $B(CO_2CO_2)_2^-$ (BOB^-) [48, 79, 376, 380, 385, 390–484], (**b**) $B(O(C_6H_4)O)(CO_2CO_2)^-$ ($BDOB^-$) [376, 377, 485, 486], (**c**) $B(CO_2CH_2O)_2^-$ [384, 487], (**d**) $B(CO_2CH(CH_3)O)_2^-$ [384, 487, 488], (**e**) $B(CO_2C(CH_3)_2O)_2^-$ ($BMLB^-$) [385], (**f**) $B(OC(CH_3)_2C(CH_3)_2O)_2^-$ [489, 490], (**g**) $BF_2(CO_2CO_2)^-$ ($DFOB^-$) [390–392, 491–535], (**h**) $BF_2(O(C_6H_4)O)^-$ ($DFBDB^-$) [485, 536, 537], (**i**) $BF_2(O(C_6H_3F)O)^-$ ($FLDFBDB^-$) [485, 537],

7.55 (Fig. 1.29n), 7.79 (Fig. 1.29o), and 8.39 (Fig. 1.29p) [551, 552]. The conductivity of the electrolytes thus increases with increasing fluorination of the anions and is dependent upon the positioning of the fluorine atoms (with the *para* position less favorable for increasing the conductivity). Increased fluorination of the anions also increased the oxidative stability of DME- or EC:DMC-based electrolytes with the salts [551, 552]. With regard to Al passivation at high potential for these electrolytes (as well as those with EC:DMC), only the electrolytes with Fig. 1.29h, j, and n anions passivated the Al electrode—the electrolytes with the other six anions did not [551, 552]. Seemingly, the anions with fluorine atoms in the *ortho* or the *para* position on the benzene ring do not decompose to leave a passivating film on the Al surface.

The lithium bis(perfluoropinacolato)borate (LiBPFPB) salt (Fig. 1.28dd) is also reported to have a high oxidative stability [547]. No information is available about the Al corrosion behavior of electrolytes with this salt, but a 0.6 M LiBPFPB electrolyte with DME at 25 °C has a conductivity of 11.1 mS cm^{-1} [547]. The conductivity of a 1 M electrolyte with the salt in PC at 25 °C, however, is 2.1 mS cm^{-1} [547]—for comparison, the conductivity for a 1 M $LiAsF_6$ electrolyte with PC at 20 °C is 5.28 mS cm^{-1} [5].

A number of lithium salts with tetrakis(haloacyloxy)borate anions (i.e., $LiB(CO_2R)_4$) have also been synthesized (Fig. 1.30a–d) [553, 554]. These are fluorinated and/or chlorinated versions of the tetra(acetato)borate anion (i.e., $B(CO_2CH_3)_4^-$) (Figs. 1.27k and 1.30e). The acid and cesium salt with the $B(CO_2CF_3)_4^-$ anion were first reported in 1971 [561]. The lithium salt (i.e., $LiB(CO_2CF_3)_4$) was subsequently reported in 1972 [562]. Electrolytes with these non-chelate salts have a relatively high conductivity (although lower than for $LiPF_6$), high oxidative stability, and high cycle efficiency with a graphite electrode. The most conductive salt is $LiB(CO_2CF_3)_4$ (comparable to the conductivity of electrolytes with LiTFSI). Lengthening the perfluoralkyl chains from $–CF_3$ to $–C_2F_5$ decreases the conductivity, as does replacing the fluorine atoms with chlorine atoms [553]. A lithium salt with the tetrakis(chlorosulfato)borate anion (i.e., $B(SO_3Cl)_4^-$) has been reported (Fig. 1.30f) [556], as has the acid with the tetrakis-(trifluoromethanesulfonato)borate anion (i.e., $B(SO_3CF_3)_4^-$) (Fig. 1.30g) [557]. Note that this anion differs from the $B(SO_2CF_3)_4^-$ anion noted in Fig. 1.18d.

Fig. 1.28 (continued) (**j**) $B(O(C_6H_3F)O)(CO_2CO_2)^-$ ($FLBDOB^-$) [377, 380, 485, 486, 536], (**k**) $BF_2(O(C_6F_4)O)^-$ ($4FLDFBDB^-$) [486, 537], (**l**) $B(O(C_6H_4)O)(C_5O_5)^-$ ($BDCB^-$) [538], (**m**) $B(CO_2CO_2)(C_5O_5)^-$ (OCB^-) [538], (**n**) $B(C_5O_5)_2^-$ (BCB^-) [382, 383, 538], (**o**) $B(O(C_6H_4)CO_2)(C_5O_5)^-$ (CSB^-) [382, 383], (**p**) $BF_2(CO_2CH_2CO_2)^-$ [539], (**q**) $BF_2(CO_2C(CH_3)_2CO_2)^-$ [539], (**r**) $B(CO_2CO_2)(CO_2CH_2CO_2)^-$ (MOB^-) [540], (**s**) $B(CO_2CH_2CO_2)_2^-$ (BMB^-) [385, 484, 540, 541], (**t**) $B(CO_2C(CH_3)_2CO_2)_2^-$ [539], (**u**) $B(CO_2CHFCO_2)_2^-$ [540, 542], (**v**) $B(CO_2CF_2CO_2)_2^-$ [392, 543], (**w**) $BF_2(CO_2CH_2CO_2)^-$ [392, 543], (**x**) $BF_2(CO_2CF_2CO_2)^-$ [392, 543], (**y**) $BF_2(CO_2C_2F_4CO_2)^-$ [392, 543], (**z**) $B(CO_2CO_2)(CO_2CF_3)_2^-$ [544], (**aa**) $B(CO_2C(CF_3)_2O)(CO_2CF_3)_2^-$ [392], (**bb**) $B(CO_2CO_2)(OCH(CF_3)_2)_2^-$ [392], (**cc**) $B(CO_2C(CF_3)_2O)(OCH(CF_3)_2)_2^-$ [392, 543, 545, 546], and (**dd**) $B(OC(CF_3)_2C(CF_3)_2O)_2^-$ ($BPFPB^-$) [484, 541, 547]

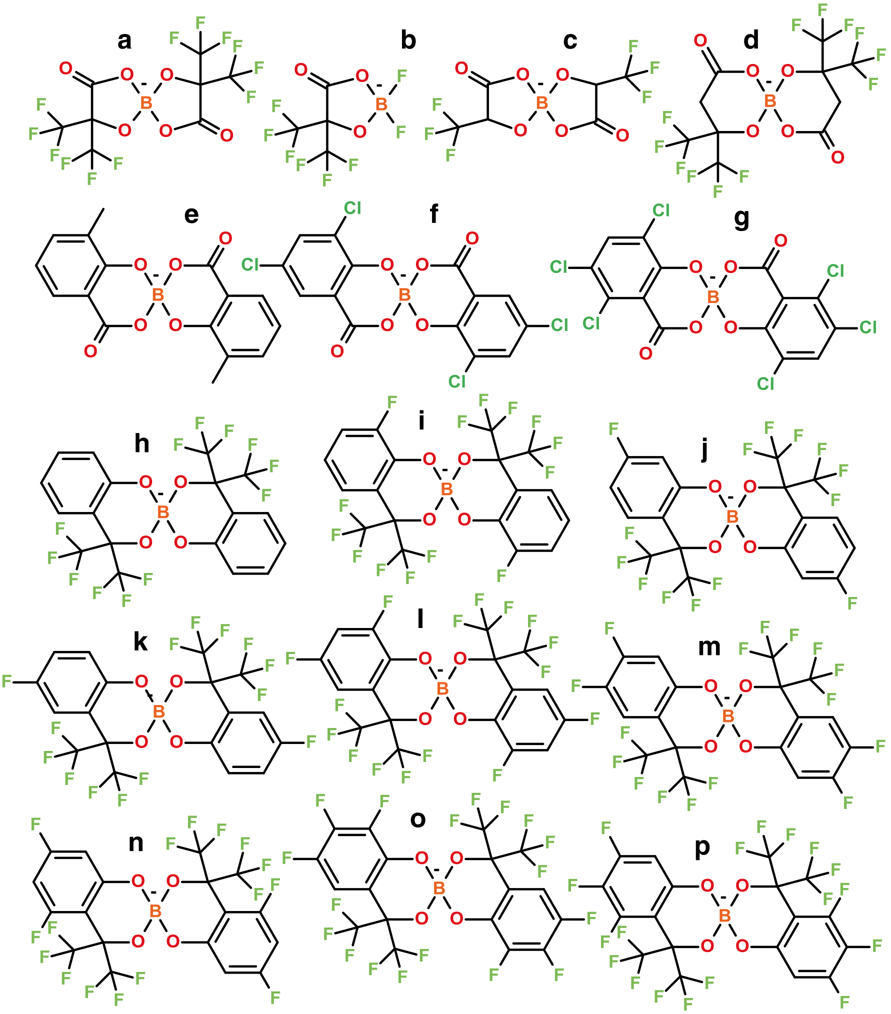


Fig. 1.29 Additional examples of chelated organoborate anions: (**a**) $B(CO_2C(CF_3)_2O)_2^-$ [392, 545, 546], (**b**) $BF_2(CO_2C(CF_3)_2O)^-$ [392, 545, 546], (**c**) $B(CO_2CH(CF_3)O)_2^-$ [392, 545, 546], (**d**) $B(CO_2CH_2C(CF_3)_2O)_2^-$ [392, 545, 546], (**e**) $B(CO_2(C_6H_3(CH_3))O)_2^-$ (3-MLBSB$^-$) [56], (**f**) $B(CO_2(C_6H_2Cl_2)O)_2^-$ (DCLBSB$^-$) [56], (**g**) $B(CO_2(C_6HCl_3)O)_2^-$ (TCLBSB$^-$) [56], (**h**) $B(O(C_6H_4)C(CF_3)_2O)_2^-$ [551, 552], (**i**) $B(O(C_6H_3F)C(CF_3)_2O)_2^-$ [551, 552], (**j**) $B(O(C_6H_3F)C(CF_3)_2O)_2^-$ [551, 552], (**k**) $B(O(C_6H_3F)C(CF_3)_2O)_2^-$ [551, 552], (**l**) $B(O(C_6H_2F_2)C(CF_3)_2O)_2^-$ [551, 552], (**m**) $B(O(C_6H_2F_2)C(CF_3)_2O)_2^-$ [551, 552], (**n**) $B(O(C_6H_2F_2)C(CF_3)_2O)_2^-$ [551, 552], (**o**) $B(O(C_6HF_3)C(CF_3)_2O)_2^-$ [551, 552], and (**p**) $B(O(C_6HF_3)C(CF_3)_2O)_2^-$ [551, 552]

The reaction of trialkoxyborates with butyllithium produces salts which are liquid (for $n \geq 2$) at ambient temperature without solvents (i.e., ionic liquids) (Fig. 1.30h) [558]. The ambient temperature conductivity for the $n = 3$ salt is 2×10^{-5} S cm^{-1}, while 1 M electrolytes of the salt in EC:PC have a conductivity $<10^{-3}$ S cm^{-1}.

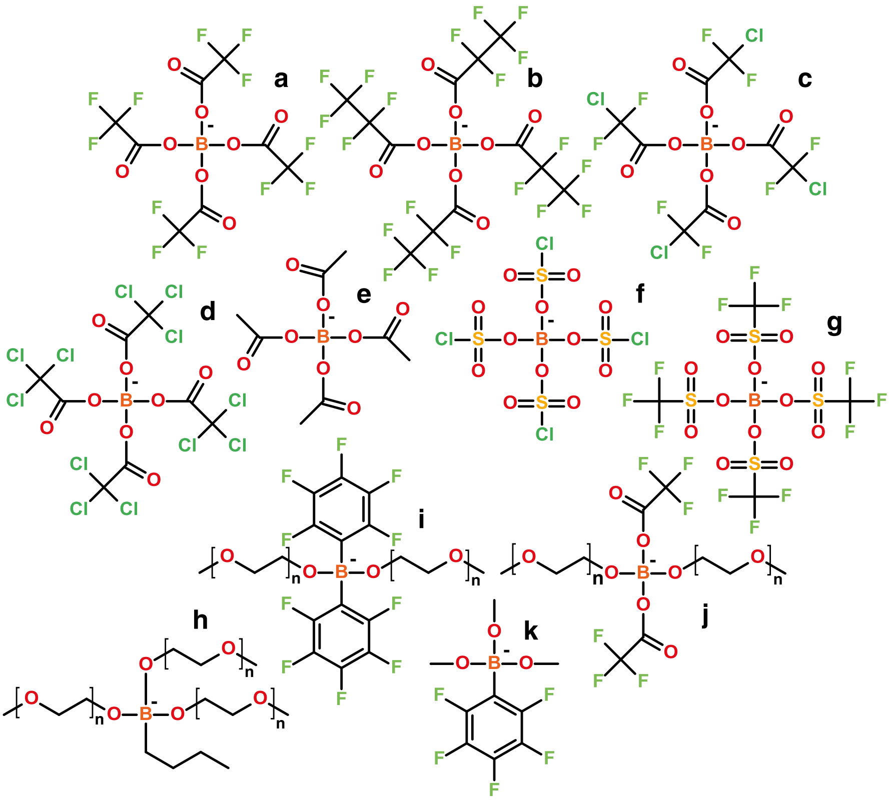


Fig. 1.30 Examples of nonchelated organoborate anions: (**a**) $B(CO_2CF_3)_4^-$ [553, 554], (**b**) $B(CO_2C_2F_5)_4^-$ [553, 554], (**c**) $B(CO_2CF_2Cl)_4^-$ [553, 554], (**d**) $B(CO_2CCl_3)_4^-$ [553, 554], (**e**) $B(CO_2CH_3)_4^-$ [554], (**f**) $B(SO_3Cl)_4^-$ [555, 556], (**g**) $B(SO_3CF_3)_4^-$ [557], (**h**) $B(O(CH_2CH_2O)_nCH_3)_3(C_4H_9)^-$ [558], (**i**) $B(C_6F_5)_2(O(CH_2CH_2O)_nCH_3)_2^-$ [559], (**j**) $B(CO_2CF_3)_2(O(CH_2CH_2O)_nCH_3)_2^-$ [559], and (**k**) $B(C_6F_5)(OCH_3)_3^-$ [560]

The nonfluorinated lithium tris(1,2-benzenediolato(2)-O,O′)phosphate (*LiTBP*) salt (Figs. 1.31a and 1.32a), reported by Sasaki and co-workers, has a relatively low thermal (<200 °C) and electrochemical (about 3.7 V (vs. Li/Li^+)) stability [378, 564, 565]. Adding a methyl group—lithium tris(4-methyl-1,2-benzenediolato(2)-O,O′) phosphate (*Li4-MLTBP*) (Fig. 1.31b)—improves the thermal stability somewhat, but decreases the electrolyte conductivity (relative to LiTBP) [566]. Adding a fluorine atom—lithium tris(3-fluoro-1,2-benzenediolato(2)-O,O′)phosphate (*Li3-FLTBP*) (Fig. 1.31c)—improves the electrolyte conductivity and thermal/electrochemical stability (relative to LiTBP and Li4-MLTBP) [564, 565]. The conductivity of 0.5 M electrolytes with EC:DMC at 25 °C is about 2.62, 2.25, and 3.16 mS cm^{-1}, respectively, for LiTBP, Li4-MLTBP, and Li3-FLTBP (as compared to 9.66 mS cm^{-1} for $LiPF_6$) [378, 564, 566]. Fully fluorinating the anion—lithium tris(3,4,5,6-tetrafluoro-1,2-benzenediolato(2)-O,O′)phosphate (Fig. 1.31d)—further increases

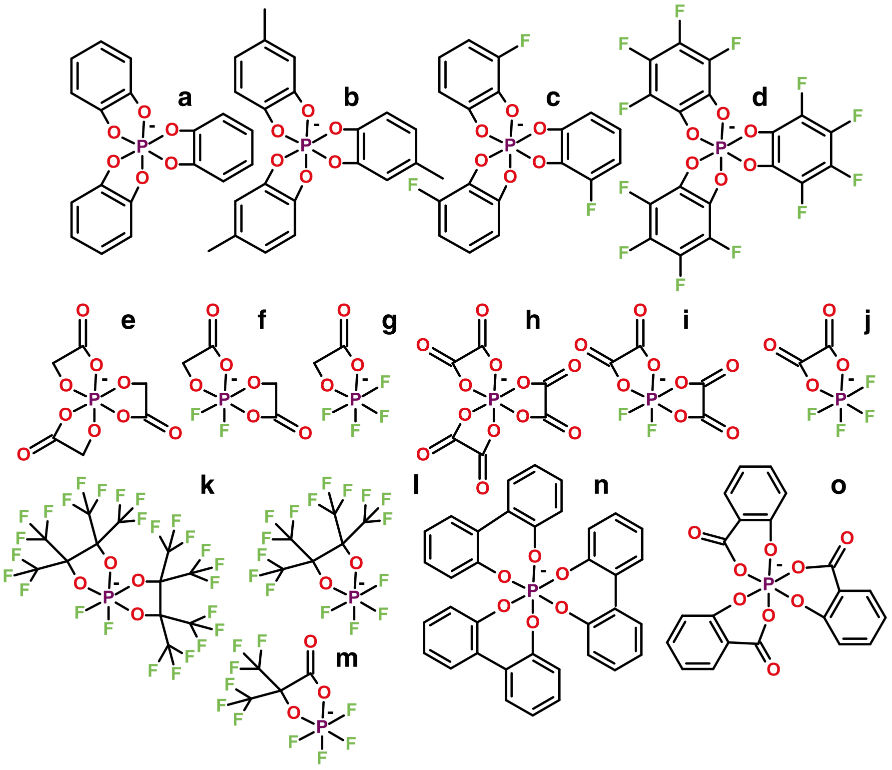


Fig. 1.31 Examples of chelated organophosphate anions: (**a**) $P(O(C_6H_4)O)_3^-$ (TBP$^-$) [378, 563–567], (**b**) $P(O(C_6H_3(CH_3))O)_3^-$ (4-MLTBP$^-$) [564, 566], (**c**) $P(O(C_6H_3F)O)_3^-$ (3-FLTBP$^-$) [564, 567], (**d**) $P(O(C_6F_4)O)_3^-$ [567], (**e**) $P(CO_2CH_2O)_3^-$ [568], (**f**) $PF_2(CO_2CH_2O)_2^-$ [568], (**g**) $PF_4(CO_2CH_2O)^-$ [568], (**h**) $P(CO_2CO_2)_3^-$ (TOP$^-$) [568, 569], (**i**) $PF_2(CO_2CO_2)_2^-$ [568], (**j**) $PF_4(CO_2CO_2)^-$ (FOP$^-$) [392, 543, 568, 570–576], (**k**) $PF_2(OC(CF_3)_2C(CF_3)_2O)_2^-$ [577], (**l**) $PF_4(OC(CF_3)_2C(CF_3)_2O)^-$ [577], (**m**) $PF_4(CO_2C(CF_3)_2O)_2^-$ [545, 546], (**n**) $P(O(C_6H_4)(C_6H_4)O)_3^-$ (TBPP$^-$) [578], and (**o**) $P(CO_2(C_6H_4)O)_3^-$ (TSP$^-$) [579]

the electrochemical stability, but the conductivity of a 0.6 mol kg^{-1} EC:DEC (2:1) electrolyte at 25 °C is relatively low (2.1 mS cm^{-1}) [567]. The high mass, high fluorination (12 F/Li$^+$), and lack of improved properties (relative to $LiPF_6$) have limited interest in this salt.

The nonfluorinated lithium tri(oxalato)phosphate (*LiTOP*) (i.e., $LiP(CO_2CO_2)_3$) salt (Figs. 1.31h and 1.32b) has a high solubility and high (oxidative) electrochemical stability [569]. The related lithium tetrafluoro(oxalato)phosphate salt (*LiFOP*) (i.e., $LiPF_4(CO_2CO_2)$) (Fig. 1.31j) was first formed in electrolytes with mixtures of $LiPF_6$ and LiBOB [576]. The properties of this salt have been extensively characterized in battery electrolytes [570–576]. In many respects, LiFOP has similar properties to $LiPF_6$. Both salts have a similar thermal stability and result in electrolytes with a high conductivity [574, 575], but a carbonate solvent-based electrolyte (i.e., 1 M in EC:DEC:DMC 1:1:1) with LiFOP may be stored at 85 °C for weeks

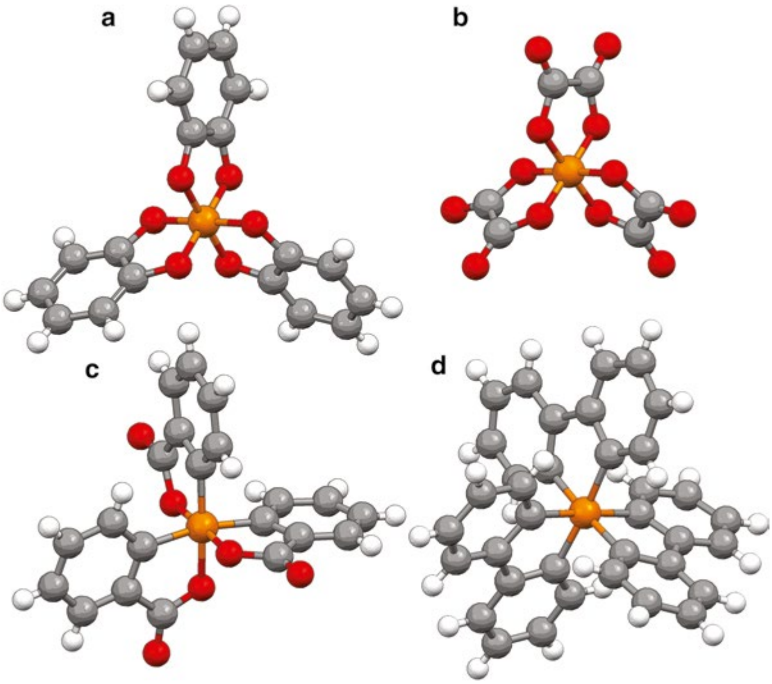


Fig. 1.32 Anion structures: (**a**) $P(O(C_6H_4)O)_3^-$, (**b**) $P(CO_2CO_2)_3^-$ (TOP⁻), (**c**) $P(CO_2(C_6H_4))_3^-$ (not TSP⁻), and (**d**) $P((C_6H_4)(C_6H_4))_3^-$ (not TBPP⁻) (C—*gray*, O—*red*, P—*orange*)

with no evident degradation, whereas extensive salt degradation occurs for a similar electrolyte with $LiPF_6$ [574]. As for the LiBOB and LiDFOB salts, the addition of small amounts of LiFOP is found to improve the capacity retention of MCMB/NMC cells, as well as the stability of the lithiated negative electrode [573]. This is attributed to the oxalate group reacting to form surface layers on both the cathode and carbon anode [574]. The first cycle irreversible capacity losses due to SEI formation on the anode, however, are strongly dependent upon the type of carbon used for the anode [571, 572].

Other organophosphate anions have also been reported, such as those with perfluoropinacol (Fig. 1.31k, l), biphenylene (i.e., tris(2,2′-biphenylylene)phosphate (*TBPP*⁻)) (Fig. 1.31n), and salicylate (i.e., tris(salicylato(2-))phosphate (*TSP*⁻)) (Fig. 1.31o). No information is available about the use of these anions as lithium salts for battery electrolytes.

Fluorinated organoaluminate anions have also been examined for battery electrolytes (Figs. 1.33 and 1.34) [552, 580]. For smaller bidentate ligands (e.g., oxalate), the Al^{III} is typically coordinated by three ligands in solid-state salts, instead of two, due to its larger size relative to boron (Fig. 1.34a) [581]. This results in a six-coordinate $Al(CO_2CO_2)_3^{3-}$ trianion instead of a four-coordinate $Al(CO_2CO_2)_2^-$ anion, although there is spectroscopic evidence for the latter in aqueous solutions as the additional

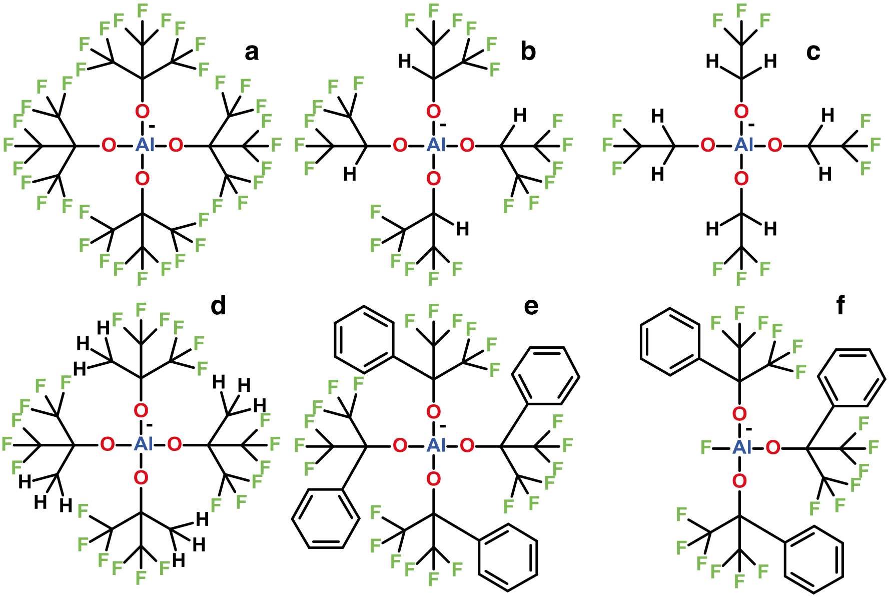


Fig. 1.33 Examples of organoaluminate anions: (**a**) $Al(OC(CF_3)_3)_4^-$ ($Al(PFTB)_4^-$) [552, 580], (**b**) $Al(OCH(CF_3)_2)_4^-$ ($Al(HFIP)_4^-$) [552, 580], (**c**) $Al(OCH_2(CF_3))_4^-$ ($Al(TFE)_4^-$) [552, 580], (**d**) $Al(OC(CH_3)(CF_3)_2)_4^-$ ($Al(HFTB)_4^-$) [552, 580], (**e**) $Al(OC(C_6H_5)(CF_3)_2)_4^-$ ($Al(HFPP)_4^-$) [552, 580], and (**f**) $AlF(OC(C_6H_5)(CF_3)_2)_3^-$ ($AlF(HFPP)_3^-$) [552, 580]

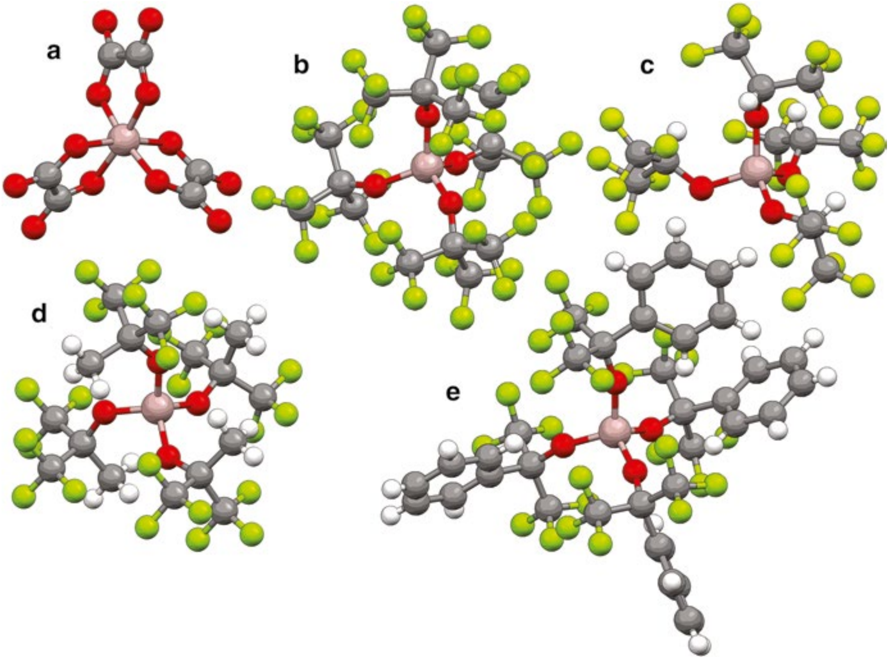


Fig. 1.34 Anion structures: (**a**) $Al(CO_2CO_2)_3^{3-}$, (**b**) $Al(OC(CF_3)_3)_4^-$ ($Al(PFTB)_4^-$), (**c**) $Al(OCH(CF_3)_2)_4^-$ ($Al(HFIP)_4^-$), (**d**) $Al(OC(CH_3)(CF_3)_2)_4^-$ ($Al(HFTB)_4^-$), and (**e**) $Al(OC(C_6H_5)(CF_3)_2)_4^-$ ($Al(HFPP)_4^-$) (C—*gray*, O—*red*, F—*light green*, Al—*tan*)

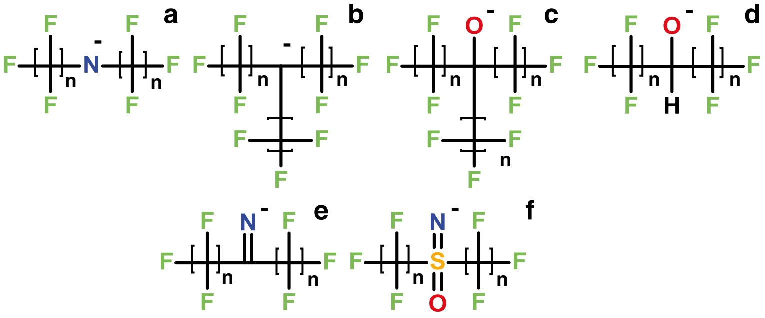


Fig. 1.35 Examples of fluorinated amide, methide, imine, alkoxide, and sulfur oxyimine anions: (**a**) $N(C_nF_{2n+1})_2^-$ [585–594], (**b**) $C(C_nF_{2n+1})_3^-$ [595, 596], (**c**) $OC(C_nF_{2n+1})_3^-$ [597–601], (**d**) $OCH(C_nF_{2n+1})_2^-$ [601–603], (**e**) $N{=}C(C_nF_{2n+1})_2^-$ [604–611], and (**f**) $N{=}S(O)(C_nF_{2n+1})_2^-$ [612–617]

coordination sites are occupied by water molecules (i.e., $Al(CO_2CO_2)_2^-{\cdot}2H_2O$) [582–584]. For more bulky ligands, however, four-coordinate anions are formed. The conductivity values (mS cm^{-1}) (0.2 M salt in DME at 25 °C) for the lithium salts with the anions shown in Fig. 1.33a–f are reported to be 6.4 (Fig. 1.33a), 6.2 (Fig. 1.33b), 1.6 (Fig. 1.33c), 6.2 (Fig. 1.33d), 3.5 (Fig. 1.33e), and 1.3 (Fig. 1.33f) [552, 580]. PC-based electrolytes with the $LiAl(HFIP)_4$ (Figs. 1.33b and 1.34c) and $LiAl(HFPP)_4$ (Figs. 1.33e and 1.34e) salts (0.3 M in PC) both passivated an Al electrode at high potential in a similar manner to a $LiPF_6$ electrolyte [552, 580].

1.9 Advanced Salts—Other Anions

HNF_2 (Fig. 1.35a) is a gas with a T_b of −24 °C [590, 591]. This gas loses hydrogen when contacted with various materials to form tetrafluorohydrazine (N_2F_4). At low temperature (crystalline solid), this acid tends to detonate spontaneously [591]. The $LiNF_2$ salt has not been reported, but it is predicted to be a dimeric complex [592, 593]. $HN(CF_3)_2$ (Fig. 1.35a) is a gas with a T_b of −6 °C [586–588]. The experimental gas-phase acidity value for $HN(CF_3)_2$ is 324.3 kcal mol^{-1}, which is significantly higher (less acidic) than the corresponding values for $HN(COCF_3)_2$ (307.5) and $HN(SO_2CF_3)_2$ (291.8) (Fig. 1.2) [26]. A German patent for Merck has been issued for the preparation of $N(CF_3)_2^-$ salts (Fig. 1.35a), but this does not include the lithium salt [589]. Another report indicates that the alkali metal bis(trifluoromethyl) amides, -phosphides, and -arsenides (i.e., $MN(CF_3)_2$, $MP(CF_3)_2$, and $MAs(CF_3)_2$) all have a high nucleophilic reactivity [594]. For the related methides, the experimental gas-phase acidity values for $HCH(CF_3)_2$ and $HC(CF_3)_3$ (Fig. 1.35b) are 343.9 and 326.6 kcal mol^{-1}, respectively (Fig. 1.2) [26]. The value for the latter is comparable to the gas-phase acidity of HCl (328.1 kcal mol^{-1}) [595, 596].

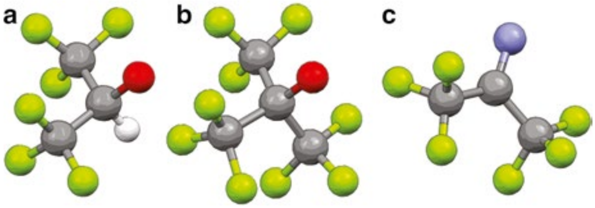


Fig. 1.36 Anion structures: (**a**) $OCH(CF_3)_2^-$, (**b**) $OC(CF_3)_3^-$, and (**c**) $N{=}C(CF_3)_2^-$ (C—*gray*, N—*blue*, O—*red*, F—*light green*)

Trifluoromethanol (i.e., $HOCF_3$) (Fig. 1.35c) is unstable at room temperature due to the elimination of HF [618, 619]:

$$HOCF_3 \rightarrow O{=}CF_2 + HF$$

$MOCF_3$ salts (M = K, Rb, and Cs) (Fig. 1.35c) have been prepared, however, by passing $O{=}CF_2$ through an acetonitrile solution of the fluoride MF [620, 621]:

$$O{=}CF_2 + MF \rightleftarrows MOCF_3$$

and the crystal structures determined [597]. No reaction occurred when LiF or NaF was used [597], and some decomposition occurred for KF, suggesting that the lithium trifluoromethoxide (or trifluoroorthocarbonate) salt is unstable (i.e., harder cations are more reactive). Additional salts with perfluoralkyl groups have also been prepared [598–600]:

$$O{=}CFR_F + MF \rightleftarrows MOCF_2R_F$$

$$O{=}C\left(CF_3\right)_2 + MF \rightleftarrows MOCF\left(CF_3\right)_2$$

with M = Rb or Cs and $R_F = -CF_3$, $-C_2F_5$, or $-C_3F_7$. It was found that the cesium salts were more thermally stable than the corresponding rubidium salts. The $LiOCH(CF_3)_2$ (Figs. 1.35d and 1.36a) and $LiOC(CF_3)_3$ (Figs. 1.35d and 1.36b) salts are both volatile with the former subliming at 50 °C under vacuum (0.05 mmHg) and the latter having a T_b of 218 °C [601].

The difluoromethanimine acid (i.e., $HN{=}CF_2$) (Fig. 1.35e) has been reported [604, 605]. This is a colorless gas which is stable at ambient temperature for hours in the gas phase at pressures lower than 5 mm Hg, but which disproportionates at higher pressure:

$$2\,HN{=}CF_2 \rightarrow H_2NCF_3 + FCN$$

The lithium hexafluoroisopropylidenimine salt (i.e., $LiN{=}C(CF_3)_2$) (Figs. 1.35e and 1.36c) has been reported [606–611]. The strong electron-withdrawing $-CF_3$

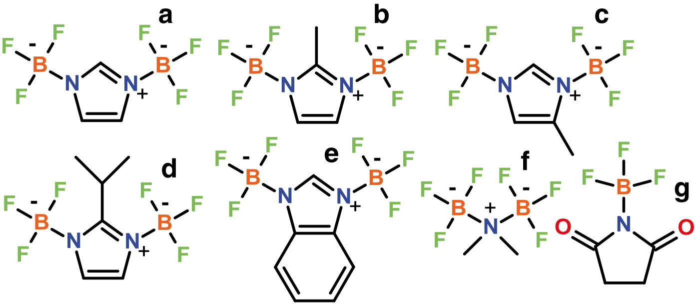


Fig. 1.37 Examples of Lewis acid–Lewis base complex anions: (**a**) $N_2C_3H_3(BF_3)_2^-$ [622–625], (**b**) $N_2C_3H_2(CH_3)(BF_3)_2^-$ [622–625], (**c**) $N_2C_3H_2(CH_3)(BF_3)_2^-$ [622–625], (**d**) $N_2C_3H_2(CH(CH_3)_2)(BF_3)_2^-$ [622–625], (**e**) $N_2C_7H_5(BF_3)_2^-$ [622–625], (**f**) $N(CH_3)_2(BF_3)_2^-$ [622–625], and (**g**) $N_2C_4H_4O_2(BF_3)^-$ [626]

groups make the double bond susceptible to nucleophilic attack. The difluorosulfur oxyimine (i.e., $HN{=}S(O)F_2$) and bis(trifluoromethyl)sulfur oxyimine (i.e., $HN{=}S(O)(CF_3)_2$) acids (Fig. 1.32f) are also known [612–617], and the lithium bis(trifluoromethyl)sulfur oxyimine salt (i.e., $LiN{=}S(O)F_2$) (Fig. 1.32f) has been prepared [615]. No information is available, however, about the properties of this latter salt. Note that none of the anions shown in Fig. 1.35 have resonance to stabilize the negative charge, except for the $N{=}S(O)(C_nF_{2n+1})_2^-$ anions.

A family of lithium salts based upon monoanionic species with one or more Lewis acid groups (i.e., BF_3) complexed to a Lewis base have also been reported (Fig. 1.37) [622–626]. The most promising of these for electrolyte applications is lithium bis(trifluoroborane)imidazolide (i.e., $LiC_3N_2H_3(BF_3)_2$) (Fig. 1.37a). This salt has a high solubility—up to 2 M solutions in EC:EMC (1:3 v:v)—and a conductivity of 5.1 mS cm^{-1} at 20 °C for a 1 M electrolyte (as compared to equivalent $LiBF_4$ and $LiPF_6$ electrolytes with conductivity values of 1.78 and 7.71 mS cm^{-1}, respectively) [622]. The salt also has a reasonably high (>4.5 V vs. Li/Li^+) oxidative stability in DMC. $Li/LiNi_{0.2}Co_{0.8}O_2$ cells with this electrolyte have a comparable performance to cells with $LiPF_6$-based electrolytes [622].

The addition of Lewis acids such as $B(OCH(CF_3)_2)_3$ and $B(OC_6F_5)_3$—the so-called anion receptors—to electrolyte solutions containing highly associated salts, such as LiF and $LiCO_2CF_3$, has been demonstrated to result in much more conductive electrolytes (than the same electrolytes without the anion receptors) [627–636]. An earlier series of publications by Brownstein explored in detail the formation, or lack of formation, in solution of complex fluoroanions when Lewis acids were added to solutions with a wide variety of fluoroanion Lewis bases [637–642]. Two of the strongest superacids known—HSO_3F-SbF_5 (“magic acid”) and HF-SbF_5 (fluoroantimonic acid) [21, 557]—are also examples of complex formation between a Lewis base and Lewis acid.

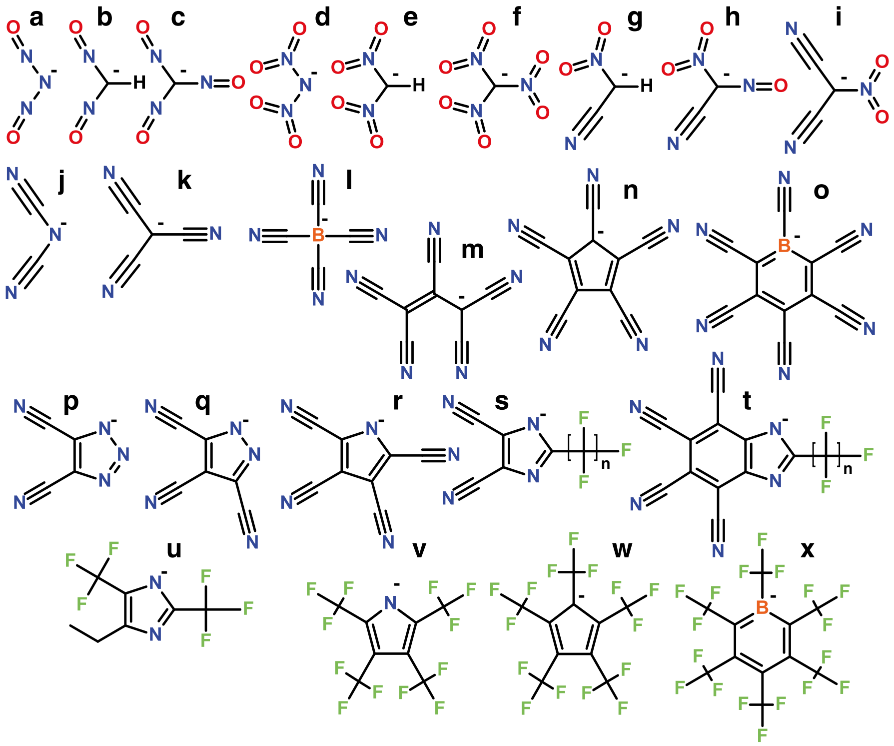


Fig. 1.38 Examples of nitroso-, nitro-, and cyano-substituted anions: (**a**) $N(NO)_2^-$ [643], (**b**) $CH(NO)_2^-$ [644], (**c**) $C(NO)_3^-$, (**d**) $N(NO_2)_2^-$ [645], (**e**) $CH(NO_2)_2^-$ [646], (**f**) $C(NO_2)_3^-$ [646], (**g**) $CH(NO_2)(CN)^-$ [646], (**h**) $C(NO)(NO_2)(CN)^-$ [646], (**i**) $C(NO_2)(CN)_2^-$ [646], (**j**) $N(CN)_2^-$ (DCA^-) [647, 648], (**k**) $C(CN)_3^-$ (TCM^-) [648, 649], (**l**) $B(CN)_4^-$ (TCB^-) [648, 650–654], (**m**) $C(CN)_2{=}C(CN)C(CN)_2^-$ [655–659], (**n**) $C_5(CN)_5^-$ [660–662], (**o**) $C_5B(CN)_6^-$ [663], (**p**) $C_2N_3(CN)_2^-$ ($DCTA^-$) [661, 664–668], (**q**) $C_3N_2(CN)_3^-$ [661, 669–671], (**r**) $C_4N(CN)_4^-$ [661, 672–674], (**s**) $C_3N_2(CN)_2(C_nF_{2n+1})^-$ (TDI^-) ($n=1$), (PDI^-) ($n=2$) and HDI^- ($n=3$) [675–686], (**t**) $C_7N_2(CN)_4(C_nF_{2n+1})^-$ [680], (**u**) $C_3N_2(C_2H_5)(CF_3)_2^-$ [687], (**v**) $C_4N(CF_3)_4^-$ [688, 689], (**w**) $C_5(CF_3)_5^-$ [688, 690–692], and (**x**) $C_5B(CF_3)_6^-$ [663]

The calculated (rather than experimental) gas-phase acidity values for methanides with –NO, $-NO_2$, and –CN substituents are as follows (QC-calculated ΔG_{acid} values in kcal mol^{-1}) relative to CH_4 (407.1): HCH_2NO (353.5), $HCH(NO)_2$ (318.5) (Fig. 1.38b), $HC(NO)_3$ (303.9) (Fig. 1.38c), HCH_2NO_2 (348.5), $HCH(NO_2)_2$ (311.5) (Fig. 1.38e), $HC(NO_2)_3$ (298.0) (Fig. 1.38f), HCH_2CN (363.9), $HCH(CN)_2$ (322.9), and $HC(CN)_3$ (288.8) (Fig. 1.38k) [693]. The addition of a single $-NO_2$ group is therefore expected to be more effective at increasing the acidity than a single –CN group. But, while double and triple substitution significantly increases the acid strength further, the gains are much smaller than for the initial substituent and vary for the different functional groups. Thus, trisubstitution with the –CN groups is more effective (as determined from the calculations) at increasing the acidity than for the –NO and $-NO_2$ groups [693]. Therefore, while resonance effects strongly influence the acidity of these methanides, the magnitude of the effect differs markedly depending upon both the degree of substitution and the identity of the functional groups.

Lithium salts with anions having nitroso or nitro functional groups have not been used for lithium battery electrolytes, despite the strong electron-withdrawing properties of these groups. This is due to the energetic characteristics of such anions [694–697]. For example, lithium dinitrosomethanide (i.e., $LiCH(NO)_2$) (Fig. 1.38b), while stable at room temperature, is reported to be heat/shock sensitive and highly explosive, as well as highly toxic [644]. Anions with mixtures of the –NO, $–NO_2$, and –CN substituents have also been prepared (Fig. 1.38g–i) [646], but salts with the nitrosodicyanomethanide (i.e., $C(NO)(CN)_2^-$), nitrodicyanomethanide (i.e., $C(NO_2)(CN)_2^-$) (Fig. 1.38i), and other related anions are also predicted to be highly energetic and thus favorable for propellant applications [646]. Further, the trinitrogen dioxide or dinitrosamide anion (i.e., $N(NO)_2^-$) is unstable [643]. This may also be the case for the $C(NO)_3^-$ anion as there are no reports for salts with this anion in the scientific literature, although salts with the $CH(NO)_2^-$ anion are known [447]. Lithium salts with the dicyanamide anion (*LiDCA*) (i.e., $LiN(CN)_2$) (Fig. 1.38j), tricyanomethanide (*LiTCM*) (i.e., $LiC(CN)_3$) (Fig. 1.38k), and tetracyanoborate (*LiTCB*) (i.e., $LiB(CN)_4$) (Fig. 1.38l) have been little utilized for lithium battery applications [647–653]. The most likely explanation for this is the limited electrochemical stability of these anions to oxidation, relative to other lithium salt anions.

Other cyanocarbon acids are thought to be some of the strongest acids known. For example, the QC-calculated gas-phase acidity (ΔG_{acid} in kcal mol^{-1}) of 1,1,2,3,3-pentacyanopropene (i.e., $C(CN)_2{=}C(CN)CH(CN)_2$) (267.2) (Fig. 1.38m) and pentacyano-cyclo-pentadienide (i.e., $HC_5(CN)_5$) (250.1) (Figs. 1.38n and 1.39j) (as compared to a similarly calculated value for $HC(CN)_3$ (287.6)) are some of the lowest values known [21]. Sodium salts with the $C(CN)_2{=}C(CN)C(CN)_2^-$ and $C_5(CN)_5^-$ anions have been prepared [655, 656, 698–700].

The lithium salt with the 4,5-dicyano-1,2,3-triazolate anion (*DCTA*⁻) (also known as 1,2,3-triazole-4,5-dicarbonitrile (*TADC*⁻)) (Fig. 1.38p) was first studied by Michot in 1995 [701] and reported in two publications in 2003 [664, 665]. Little has been reported regarding the properties of the lithium salt. Alkali and alkali earth salts with the $DCTA^-$ anion have also been prepared [666, 667]. The thermal stability of these salts was found to be excellent (>350 °C) [668]. In general, triazole salts are known to be energetic. The alkali metal salts, however, were found to have low sensitivity towards impact, friction, electrostatic discharge, and fast heating [668]. The acid with the related pyrazole-3,4,5-tricarbonitrile anion (*PATC*⁻) (Fig. 1.38q) was first reported in 1962 [669], but the lithium salt has not been investigated extensively as an electrolyte salt [661, 670, 671]. Similarly, the acid with the tetracyanopyrrolide anion (*TCP*⁻) (Fig. 1.38r) was also reported in 1962 [672] and the sodium salt with this anion has been prepared [674]. Trifluoromethane-substituted versions of the $C_5(CN)_5^-$ and TCP^- anions have also been reported (Fig. 1.38v, w) [688–692], but the lithium salt has only been reported for $LiC_5(CF_3)_5$ (Fig. 1.39k) [692].

Lithium salts with the 4,5-dicyano-2-(trifluoromethyl)imidazole (*TDI*⁻) (Fig. 1.39h) and 4,5-dicyano-2-(pentafluoroethyl)imidazole (*PDI*⁻) (Fig. 1.39i)

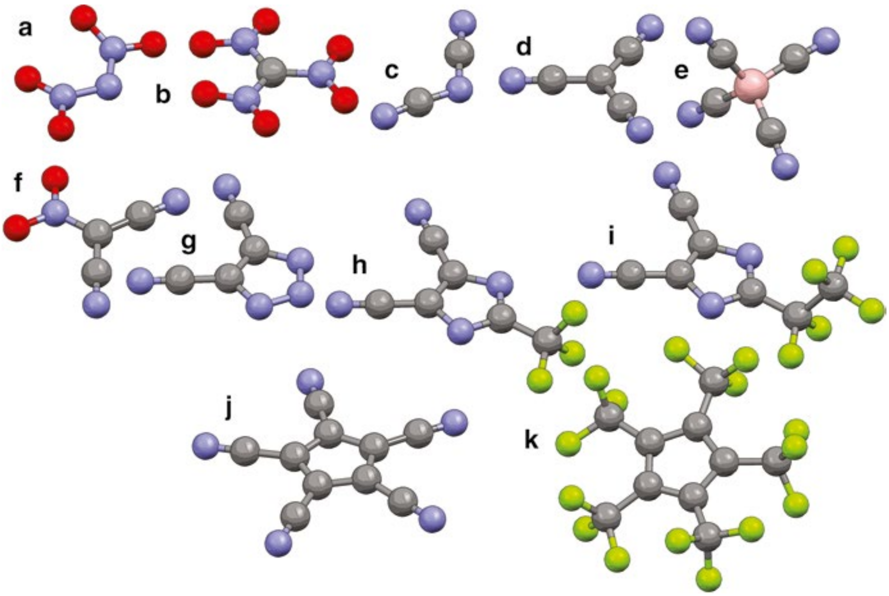


Fig. 1.39 Anion structures: (**a**) $N(NO_2)_2^-$, (**b**) $C(NO_2)_3^-$, (**c**) $N(CN)_2^-$, (**d**) $C(CN)_3^-$, (**e**) $B(CN)_4^-$, (**f**) $C(NO_2)(CN)_2^-$, (**g**) $C_2N_3(CN)_2^-$ (DCTA⁻), (**h**) $C_3N_2(CN)_2(CF_3)^-$ (TDI⁻), (**i**) $C_3N_2(CN)_2(C_2F_5)^-$ (PDI⁻), (**j**) $C_5(CN)_5^-$, and (**k**) $C_5(CF_3)_5^-$ (B—*tan*, C—*gray*, N—*blue*, O—*red*, F—*light green*)

Table 1.6 Conductivity of electrolytes with various lithium salts at 20 °C and 1 M salt in EC:DMC (50:50 w:w) [681]

Lithium salt (anion)	Conductivity (mS cm^{-1})
PF_6^-	10.8
$N(SO_2CF_3)_2^-$	9.0
PDI⁻	6.3
TDI⁻	6.7
DCTA⁻	2.7

anions (Fig. 1.38s) were reported by Niedzicki in 2009 [677]. The acid with TDI⁻ (i.e., HTDI) was first reported by Begland in 1974 [675]. A number of publications related to the use of these lithium salts for battery applications have been published [675–686], including a salt with the 4,5-dicyano-2-(n-heptafluoropropyl)imidazolide (HDI⁻) anion (Fig. 1.38s). The LiTDI, LiPDI, and LiHDI salts have been found to have a high thermal stability (>250 °C), negligible hydrolysis, high oxidative stability on Pt electrodes (4.8 V vs. Li/Li⁺), and passivate Al at high potential [679, 681]. Half cells with $LiMn_2O_4$ electrodes cycled (to 4.3 V vs. Li/Li⁺) with electrolytes (1 M in EC:DMC 50:50 w:w) containing LiTDI or LiPDI have a similar capacity and capacity retention to the same electrode cycled with an electrolyte with $LiPF_6$ [681]. The rate performance of such cells is only slightly diminished relative to the cell with the $LiPF_6$ electrolyte due to the somewhat lower conductivity of the LiTDI and LiPDI electrolytes (Table 1.6) [681]. Although PC-based electrolytes with these salts have been tested to determine the change in impedance with time

Table 1.7 Conductivities and Al repassivation potential of electrolytes with various lithium salts at 25 °C and 1 M salt in PC:DME (1:1 v:v) [704]

Lithium salt (anion)	Conductivity (mS cm^{-1})	Al potential (V vs. Li/Li^{+})
PF_6^-	15	>5
$N(SO_2CF_3)_2^-$	12	3.7
$N(SO_2C_2F_5)_2^-$	9.5	4.4
$N(SO_2CF_3)(CN)^-$	7.2	<4.2
$N(SO_2C_4F_9)(CN)^-$	5.6	>4.2
$C(SO_2CF_3)(CN)_2^-$	12.5	4.7
$C(SO_2C_4F_9)(CN)_2^-$	8.3	>5

upon storage in contact with Li metal electrodes [682], it is not yet clear if these anions are stable when charged to low potential [679] as nitriles are known to have poor reductively stability [702]. A computational study has suggested that the related 4,5,6,7-tetracyano-2-fluoroalkyl benzimidazole anions (Fig. 1.38t) may also have favorable electrolyte properties [680].

A number of other anions have also been reported which do not fit readily into the classifications noted above (Figs. 1.40 and 1.41). Little information is available about the properties of these anions (Table 1.7). The relative ionic association tendency (which influences electrolyte conductivity) for some of these may be estimated by considering the impact of different substituents on an anion's acidity (Figs. 1.1 and 1.2) and the additional information reported above. For example, the experimental gas-phase acidity values (ΔG_{acid} in kcal mol^{-1}) for $HC(C_6F_5)(SO_2CF_3)_2$ (301.3) (Fig. 1.22k) and $HC(C_6F_5)(CN)_2$ (303.6) are higher than those for $HN(SO_2CF_3)_2$ (291.8), $HC(SO_2CF_3)_3$ (289.0), and $HC(CN)_3$ (~294) due to the weaker electron-withdrawing effect of the $-C_6F_5$ group relative to $-SO_2CF_3$ and –CN for trisubstitution (in contrast to monosubstitution) (Figs. 1.1 and 1.2) [26].

Finally, a relatively unique but diverse class of anions for battery electrolytes are the carboranes and boranes [711–727]. These anions are often chemically inert and superweakly coordinating [728]. Carboranes are composed of clusters of carbon and boron atoms, whereas boranes only have boron. These carbon and boron atoms are typically bonded to H, F, Cl, Br, and/or I atoms or other groups [718]. A variety of classifications are used for carboranes and boranes including *closo*- for a complete polyhedron (e.g., $B_nX_n^{2-}$ with X = H, F, Br, Cl, I, and $6 \leq n \leq 12$) and for polyhedron missing one, two, or more vertices: *nido*- (e.g., B_5X_9, B_6X_{10}), *arachno*-, etc. The basicity of the dodecaborate $[B_{12}X_{12}]^{2-}$ anions increases in the order X = F < Cl < Br < I [721]. It is noteworthy that carborane synthesis is time consuming and costly, whereas *closo*-dodecaborates have similar stability, but are easier and cheaper to prepare [713]. Lithium *closo*-borane salts (i.e., $Li_2B_{10}Cl_{10}$ and $Li_2B_{12}Cl_{12}$) dissolved in $SOCl_2$ were used in electrolytes for $Li/SOCl_2$ liquid cathode cells in 1979 [729]. Johnson and Whittingham then used these salts for electrolytes for the early Exxon work with Li/TiS_2 cells in 1980 [712–714]. These salts were found to be poorly soluble in individual ether solvents, but did have a higher solubility in dioxolane: DME mixtures [712]. More recently, $Li_2B_{12}F_nH_{12-n}$ (n = 9 and 12) salts have been

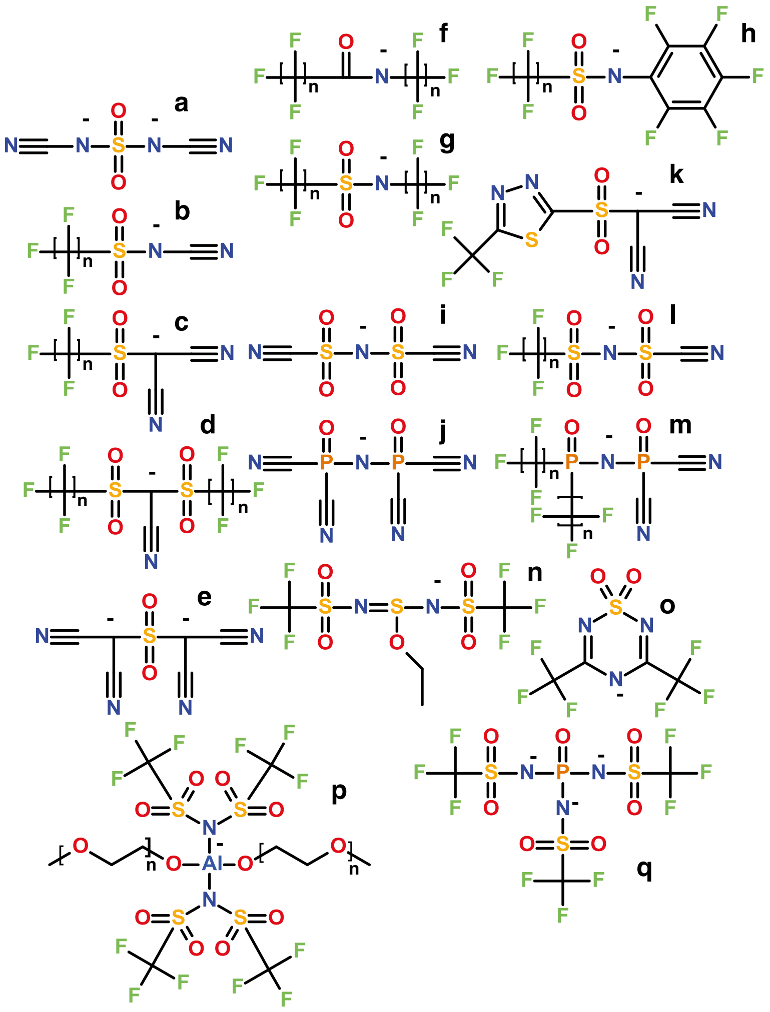


Fig. 1.40 Examples of additional anions: (**a**) $SO_2(NCN)_2^-$ [703], (**b**) $N(SO_2C_nF_{2n+1})(CN)^-$ [704], (**c**) $C(SO_2C_nF_{2n+1})(CN)_2^-$ [704–706], (**d**) $C(SO_2C_nF_{2n+1})_2(CN)^-$ [705, 706], (**e**) $SO_2(C(CN)_2)_2^-$ [706], (**f**) $N(COC_nF_{2n+1})(C_nF_{2n+1})^-$ [254], (**g**) $N(SO_2C_nF_{2n+1})(C_nF_{2n+1})^-$ [707], (**h**) $N(SO_2C_nF_{2n+1})(C_6F_5)^-$ [708], (**i**) $N(SO_2(CN))_2^-$ [361, 709], (**j**) $N(PO(CN)_2)_2^-$ [361], (**k**) $C(SO_2C_2N_2S(CF_3))(CN)_2^-$ [706], (**l**) $N(SO_2C_nF_{2n+1})(SO_2CN)^-$ [361], (**m**) $N(PO(C_nF_{2n+1})_2)(PO(CN)_2)^-$ [361], (**n**) $S(OC_2H_5)(NSO_2CF_3)_2^-$ [289], (**o**) $N(C(CF_3)NSO_2NC(CF_3))^-$ [255], (**p**) $Al(N(SO_2CF_3)_2)_2(O(CH_2CH_2O)_nCH_3)_2^-$ [559], and (**q**) $PO(NSO_2CF_3)_3^{3-}$ [710]

developed by Air Products (as the Stabilife fluorinated electrolyte salts) with a number of intriguing electrolyte properties. A TGA analysis of the $Li_2B_{12}F_{12}$ salt (*Li₂DFB*) (Figs. 1.42 and 1.43) indicates that no mass loss occurs up to 450 °C. The salt is also inert in 98 % sulfuric and 70 % nitric acid, as well as 3 M KOH (in contrast with the $B_{12}H_{12}^{2-}$ anion which reacts with sulfuric acid) [718]. The conductivity of Li_2DFB electrolytes is lower than for comparable electrolytes with $LiPF_6$ [717, 718], but the

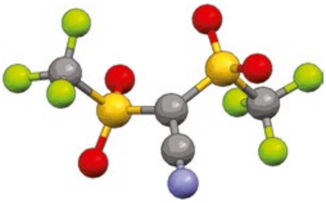


Fig. 1.41 Anion structure: $C(SO_2CF_3)_2(CN)^-$ (C—*gray*, N—*blue*, O—*red*, F—*light green*, S—*yellow*)

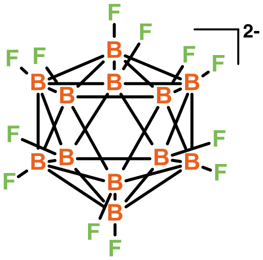


Fig. 1.42 Example of a fluorinated *closo*-dodecaborate anion: $B_{12}F_{12}^{2-}$ [715–726]

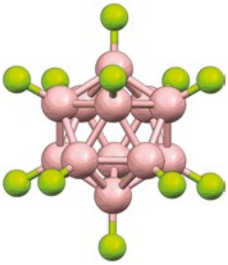


Fig. 1.43 Anion structure: $B_{12}F_{12}^{2-}$ (B—*tan*, F—*light green*)

electrolyte solvent/salt formulation can be tuned to obtain promising cell cycling performance, especially at elevated temperature (60 °C) [718]. In addition, the $B_{12}F_{12}^{2-}$ anion is not reduced at 0 V (vs. Li/Li^+) and undergoes a quasi-reversible oxidation reaction at 4.7 V (vs. Li/Li^+) to form the stable radical cation $B_{12}F_{12}^-$ [717]. The potential at which this oxidation reaction occurs can be tuned by changing the anion structure. The $Li_2B_{12}H_3F_9$ salt, for example, has a reversible oxidation reaction at 4.5 V (vs. Li/Li^+) [716, 717]. These anions are thus able to provide overcharge protection for 4+ V Li-ion batteries, as has been demonstrated for MCMB/spinel cells [716, 724]. Note that many organic molecules designed for overcharge protection have poor solubility in carbonate solvents [724]. This is not the case for the $Li_2B_{12}F_9H_3$ salt which has a reasonably high solubility (≥0.5 M) while also serving as a divalent Li^+ cation salt [717, 723].

1.10 Adoption Criterion for New Salts

Lithium salts have not been prepared for many of the anions noted above. For other anions, the corresponding lithium salts have been reported, but only in a single report with or without some limited electrolyte data provided. Such salts are often not widely available to the greater battery research community…and many of the practitioners in this community do not have the time, resources, or expertise necessary to prepare and purify such salts. Thus, the useful properties and/or limitations of the salts often remain unknown and unutilized.

Ultimately, when developing new lithium salts, it is important to ask what advantages are sought over the widely utilized salt $LiPF_6$. This salt has been a cornerstone of Li-ion battery R&D for well over two decades. But it is clear that $LiPF_6$ has limitations. Properties or features of salts which would make them attractive candidates for commercial battery electrolytes include the following:

(a) Simplified synthesis—If low-cost and/or nontoxic reagents which are easily handled are available for salt synthesis, this may reduce the overall expense of the salt production. Waste from the synthesis/purification methods is another consideration, as is the ease of salt purification to an electrochemical-grade material. The ownership of the intellectual property (IP) rights may be another important factor.

(b) Reduced hazards—In some cases, the reagents, reaction intermediates, and/or final salts are highly toxic or have other undesirable properties (e.g., energetic materials, corrosive) which requires specialized handling of the materials and makes them undesirable for commercial batteries. The degradation products (if battery failure occurs) may also be highly toxic, as has been noted for the fluoro-organic products obtained from the reaction of $LiPF_6$ and carbonate solvents at high temperature when in contact with cathode materials [730]. Thus, the elimination of fluorine from the anions is a worthy goal, but one which is difficult to achieve based upon the information noted in this review.

(c) Hydrolysis—$LiPF_6$ readily hydrolyzes when contacted with water, especially at elevated temperature. This makes the synthesis, storage, and handling of this salt more onerous, thus adding cost to its use. A salt which is not susceptible to hydrolysis would greatly simplify the handing/transportation of the salt for large-scale battery production.

(d) Divalent anions—Divalent anions may offer a means of increasing the Li^+ cation content (for a fixed number of anions) or decreasing the amount of salt needed to retain the same number of Li^+ cations in the electrolyte. This is dependent upon both of the Li^+ cations having weak interactions with the anions, which is difficult to achieve, and the salt having a moderate–high solubility in aprotic solvents, which is also difficult to achieve.

(e) Redox shuttle mechanism—The use of a salt which has other functionality, such as a redox shuttle mechanism (e.g., $B_{12}F_{12}^{2-}$) (Figs. 1.42 and 1.43) which protects the cell against overcharging, would be desirable.

(f) Thermal stability—The use of salts, either as primary salts or as additives, which result in electrolytes with high thermal stability (alone and in contact with electrodes)—enabling cell cycling at >60 °C—is a long sought after trait.

(g) New solvents—$LiPF_6$ was optimized for use with the cyclic/acyclic carbonate solvents used for Li-ion batteries. Often the use of $LiPF_6$ for electrolytes with other solvents does result in the most conductive electrolytes (relative to other salts), but there are exceptions. For example, $LiPF_6$ reacts with GBL (forming brown or black solutions), but this solvent has many promising features for electrolytes [91–96]. Thus, $LiPF_6$ has generally been replaced with $LiBF_4$, LiTFSI, LiBOB, LiDFOB, etc. when GBL is used as an electrolyte solvent.

(h) Low-temperature operation—The state-of-the-art carbonate-based electrolytes with $LiPF_6$ do not perform well at low temperature. Typically, approaches to optimize electrolytes for low temperature involve the addition of other solvents (e.g., methyl butyrate) to the carbonate solvents [478]. The difference in conductivity for electrolytes with different salts, however, becomes less significant at low temperature (Fig. 1.14). $LiPF_6$ tends to make high-melting crystalline solvates (Fig. 1.4) with a wide variety of solvents. This may be a factor for long running-time operation of batteries at low temperature. Thus, other salts which form solvates with a low T_m or do not crystallize (for specific compositions) (Fig. 1.4)—such as LiFSI, LiTFSI, or LiBETI—may be useful for such applications.

(i) SEI formation—Salts which preferentially decompose to form protective layers on the anode, cathode, Al current collector, etc. (i.e., LiBOB, LiDFOB) are likely to be required for electrodes such as Si alloys, sulfur, and high-voltage cathodes [54, 731, 732]. In particular, the formation of HF in electrolytes with $LiPF_6$ (perhaps due to the reaction of the anions with solvent molecules, instead of water), especially at high potentials and high temperature, is deemed to be particularly problematic [49–54]. Replacing the $LiPF_6$, the use of HF scavengers and the formation of protective layers are all ways to mitigate this problem.

Ultimately, the electrolyte formulations must meet the demanding criterion necessary for long-term battery operation (high stability with selective reactions, high safety, etc.). Conductivity remains an important consideration for high-power applications. Increasingly, salt mixtures (rather than just solvent mixtures) are being used to tailor electrolyte formulations. This trend is likely to increase in the future due to the necessity of simultaneously optimizing so many electrolyte properties which influence the battery's usable energy and power, lifetime, safety, cost, etc. New salts therefore remain one of the key variables available for the development of advanced electrolyte formulations.

1.11 Summary

A diverse range of anions have been prepared over the past three decades for lithium salts intended for lithium battery electrolyte applications. Many of these anions were originally prepared in efforts to generate stronger superacids. In general, it is found that selectively fluorinating the anions decreases the anion… Li^+ cation (ionic association) interactions, thereby increasing the conductivity of electrolyte solutions with the corresponding lithium salts. Anion fluorination also tends to increase the anodic stability of the anions to oxidation at high potential—an important consideration for electrolyte formulations intended for use with high-voltage cathodes. In recent years, nitrile groups have also been used in lieu of fluorination. In some cases, however, salt stability is undesireable. For example, a common approach to improving electrolytes is the use of salt additives—sacrificial anions which degrade to produce interfacial layers with the anode, cathode and/or Al current collector—which stablize the interfaces with the electrolyte resulting in dramatic improvements in battery performance. New salts therefore remain one of the key variables available for the development of advanced electrolyte formulations with tailored properties for demanding energy storage applications.

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