A Tale of Two Additives: Effects of Glutaric and Citraconic Anhydrides on Lithium-Ion Cell Performance

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The use of electrolyte additives is an important method to improve lithium-ion cell lifetime and performance without significantly affecting costs. This work evaluates two organic anhydrides, glutaric anhydride (GA) and citraconic anhydride (CA), as additives in Li(Ni0.6Mn0.2Co0.2)O2 (NMC622)/graphite and Li(Ni0.5Mn0.3Co0.2)O2 (NMC532)/graphite pouch cells, using ultrahigh precision coulometry and high-temperature storage. The additives were tested singly and in binary blends. GA-based additive blends give high coulombic efficiencies (CEs) and good storage performance. However, GA leads to substantial impedance during formation. Most notably, GA is extremely effective at suppressing gas during cell formation and storage. Whereas CA-containing blends yield good CEs, they show rapid voltage drop during storage. Both additives may provide specific benefits for target applications. Long-term cycling data indicates that GA is a negative electrode SEI-forming additive that is useful for capacity retention and limiting cell impedance growth when used as a binary blend with vinylene carbonate or lithium difluorophosphate. These results are also intended to facilitate comparison between chemically related additives in order to better understand the underlying chemistry behind their function in lithium-ion cells.

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negative electrodes were made of an artificial graphite (AG), except as noted; select prolonged cycling tests used cells with natural graphite (NG) negative electrode material. The cells were cut below the heat seal in an argon-atmosphere glove box, dried under vacuum at 80°C for 14 h, and then returned to the glove box for filling. All solutions in this work used 1.2 mol L−1 LiPF6 (BASF, ≥99.9%) in a 3:7 solvent blend, by mass, of ethylene carbonate (EC) and ethylmethyl carbonate (EMC), as received from BASF (<20 ppm H2O). The additives, GA (Sigma, 95%), CA (Aldrich, 98%), VC (BASF, ≥99.8%), DTD (Guangzhou Tinci Materials Tech. Co. Ltd., ≥98%), and LiP2O5F2 (called LFO in this work, Shenzhen CapChem Tech. Co. Ltd.) were added singly or as binary blends to this electrolyte solution in the indicated mass percentages. Cells were filled with 1.0 ± 0.1 g of solution, sealed at −90 kPa gauge pressure using a compact vacuum sealer (MSK-115A, MTI Corp.), and immediately held at 1.5 V at room temperature (21−25°C) to prevent corrosion of the copper current collector during the ∼24 h wetting period that followed. Cells were then loaded into temperature-controlled boxes (40.0 ± 0.1°C) and connected to a Maccor 4000 Series automated test system (Maccor Inc.). Because gas formation is frequently observed during formation, storage, and cycling, the pouch cells were clamped using soft rubber (at about 25 kPa gauge pressure) during all electrochemical testing, which has previously been observed to significantly improve the experimental precision.

Electrochemical testing.—SEI formation was performed by charging cells at C/20 to 4.3 V (at 40°C), holding at 4.3 V for 1 h, discharging at C/20 to 3.8 V, and then holding cells at 3.8 V for 1 h. Cells were weighed under water before and after formation, allowing the change in displacement volume to be determined using the Archimedes principle. Cells were then degassed by cutting the pouch open in an argon-atmosphere glove box and resealed using the compact vacuum sealer. Cells were weighed again and electrochemical impedance spectroscopy (EIS) was then measured at 10.0 ± 0.1°C using a BioLogic VMP3 instrument (100 kHz − 10 mHz, ±10 mV sinusoidal amplitude). Following formation, cells were either taken for ultrahigh precision coulometry (UHPC) cycling or high temperature storage. In this work, Rct represents the width of the depressed semicircle in the Nyquist plot.

For UHPC cycling, cells were maintained at 40.0 ± 0.1°C and cycled at C/20 between 3.0−4.3 V. Measurements were performed with the custom-built Dalhouse UHPC system, described previously,8,37 or with a Novonix UHPC charging system. For storage, cells were maintained at 40.0 ± 0.1°C while they were cycled at C/10 between 2.8−4.3 V twice and then held at 4.3 V for 24 h. Cells were then moved to a storage box with fixed temperature of 60.0 ± 0.1°C and the cell voltage was recorded at open circuit for 500 h. Following storage, cells were charge-discharge cycled at C/10 between 2.8−4.3 V twice again and then charged to 3.8 V. Cells were then taken for EIS testing at 10°C, as described above, and then weighed under water again to measure gas evolution during storage.

For long-term cycling, cells were maintained at 40.0 ± 0.1°C and cycled at C/3 between 3.0−4.3 V using a Neware testing system. A slow cycle was performed every 50 cycles at C/20 to evaluate impedance growth.

Results and Discussion

Cells were prepared with GA- and CA-containing electrolyte solutions. Since these are previously untested additives, multiple concentrations were tested. When used individually, GA and CA were added in 1% or 2%, by weight, which was based on the amounts used in SA and MA studies.25−28 When used in combination with DTD, MMDS, or LFO, the anhydrides were tested at 2% concentration. This is based on the prediction that GA and CA may act as the ‘primary’ film-forming additive at the negative electrode surface. This hypothesis was based on previous studies that combined DTD, MMDS, or LFO with negative electrode SEI-forming additives such as PES, VC, or FEC.10,11,32,33,38,39 Finally, GA and CA were tested at lower concentrations, i.e., 0.5% and 1%, when used in combination with VC. This is because co-additives tend to behave as the ‘secondary’ additive when used in combination with VC.

Figure 2 shows the differential capacity (dQ/dV vs Ecell) of SEI formation for cells containing GA- and CA-based additive blends in NMC622b/gr pouch cells. At these relatively low cell voltages, the electrode potential at the positive is approximately constant at Epos ∼3.6 V vs Li/Li+ whereas the potential at the negative electrode decreases relatively quickly.38,40 Therefore, the features in Figure 2 correspond to reduction processes at the negative electrode surface. The peak positions may be used to estimate reduction potentials relative to the lithium electrode according to 1:

\[
E_{cell} = E_{pos} - E_{neg} \tag{1}
\]

Control cells prepared without any additives exhibit a peak that onsets at 2.90−2.95 V, corresponding to the expected reduction of EC on the graphite surface at 0.65−0.70 V vs Li/Li+. Cells that contained 2% VC show a reduction feature that onsets at 2.75−2.80 V, corresponding to 0.80−0.85 V vs Li/Li+. The addition of VC suppresses the EC reduction peak, indicating the graphite surface has been effectively passivated. This is consistent with the expectation that VC reduction forms a passive SEI on graphite negative electrodes.2

Cells prepared with GA-based additives exhibit a new reduction feature at 2.40−2.45 V (Figure 2a), corresponding to Ered ∼1.15−1.20 V vs Li/Li+. The addition of 1% GA greatly suppresses EC reduction and all cells prepared with 2% GA (including binary blends) suppress EC reduction completely. These results indicate that GA reduction forms a passivating SEI on the graphite electrode surface. Moreover, the GA reduction feature occurs at lower cell voltage than the feature corresponding to VC reduction at ∼2.8 V. As a result, in cells prepared with a blend of 2% VC and 0.5% GA, the magnitude of the VC reduction feature is greatly decreased. This suggests that 0.5% GA partially passivates the negative electrode. The VC reduction feature is completely absent in cells that contained 2% VC and 1% GA. Therefore, at 1% GA content, the negative electrode is completely passivated by the time the cell charges to the potentials at which VC reduction normally occurs.

In contrast, cells prepared with CA do not show any new reduction features (Figure 2b). The position of EC reduction is shifted by ∼0.05 V, which suggests some chemical interaction occurs, but that the graphite surface is not passivated by CA. The cells prepared with CA/VC blends similarly exhibit a slightly shifted VC reduction peak. Finally, the 2% CA/1% DTD blend shows a peak at ∼2.80−2.85 V, corresponding to Eneg ∼0.75−0.80 V vs Li/Li+. This is a poor match for the reduction potential of DTD, Ered = 1.3−1.4 V vs Li/Li+.

The volume change due to gas evolution during the first cell charge-discharge cycle (i.e., the formation cycle) is shown in Figure 3. In all cell types, the introduction of GA significantly decreased the volume of gas evolved, relative to additive-free control cells. The suppression of formation gas is observed whether GA is used as a single or binary additive. The magnitude of gas suppression is, on average, comparable to that observed for VC-containing cells. In contrast, the addition of CA decreases the volume of gas produced during formation by only a minor amount. That is, cells that contained CA produced a similar volume of gas as the control cells. The exception to this was that cells prepared with both CA and VC displayed significant gas suppression. This decrease is attributed to the VC, rather than the presence of CA. Therefore, GA and VC are effective at suppressing gas during formation, whereas CA is not. This is consistent with the hypothesis that GA and VC form SELs that passivate the negative electrode, whereas CA does not.

The suppression of gas in GA-containing cells may suggest that this additive forms an effective SEI. Although the lack of gas suppression in CA-containing cells could be interpreted as a counter-indication of electrode passivation, some electrolyte additives that lead to passive SELs, such as DTD, nonetheless generate gaseous species as by-products during the course of SEI formation.32,38,41 The Rct of the cells following formation was measured to further explore the effects of
Figure 2. Differential capacity (dQ/dV) plots for the first charging step (i.e., SEI formation) at C/20 and at 40°C for NMC622b/gr cells. Cells contained 1.2 M LiPF₆ in 3EC:7EMC and a) GA- or b) CA-based additive blends, as indicated on the figure.

The introduction of CA also increases the post-formation Rₑ, however the extent of the increase is considerably smaller than is observed for GA. As with GA, the general trends are the same for all four positive electrode types studied in this work. The coulombic efficiency (CE) from UHPC measurements is strongly correlated with the long-term cycling performance and stability of lithium-ion cells. UHPC was therefore chosen as an efficient method to rapidly screen the suitability of GA- and CA-containing electrolytes in NMC/graphite cells. The results for GA-containing cells, shown in Figure 6, illustrate considerable variation in the results depending on the combination of positive electrode material and coating. For example, the CE of the NMC532(sc)u/gr cells was > 99.75% after 16 cycles for all electrolyte compositions tested in this work. In contrast, the NMC532(sc)u/gr cells had a visibly greater distribution in the measured CE values.
This result is especially marked in the last of these cell types, which performed especially poorly with GA-containing solutions. Yet the CE of cells prepared with the uncoated NMC532(sc)u material is nevertheless the worst of the four cell types tested here. Moreover, the greatest CE observed in this work was for binary GA-based blends in NMC532(sc)/gr (Figure 6).

High temperature storage is another practical method to rapidly evaluate the stability of new lithium-ion cell chemistries. The cell

![Figure 3](image1.png)

**Figure 3.** Volume change following cell formation to 4.3 V at 40°C. Cells contained 1.2M LiPF₆ in 3EC:7EMC and GA- or CA-based additive blends, as indicated. The positive electrode material was a) NMC622a, b) NMC622b, c) coated NMC532(sc), or d) uncoated NMC 532(sc).

![Figure 4](image2.png)

**Figure 4.** $R_\text{ct}$ at 3.8 V and at 10°C following cell formation to 4.3 V at 40°C. Cells contained 1.2M LiPF₆ in 3EC:7EMC and GA- or CA-based additive blends, as indicated. The positive electrode material was a) NMC622a, b) NMC622b, c) coated NMC532(sc), or d) uncoated NMC 532(sc).

![Figure 5](image3.png)

**Figure 5.** Nyquist plots for NMC622b/gr pouch cells measured at 3.8 V and at 10°C following cell formation to 4.3 V at 40°C. Cells contained 1.2 M LiPF₆ in 3EC:7EMC and GA-based additive blends, as indicated.
Figure 6. (a-d) Discharge capacity, (e-h) normalized (at cycle 2) charge endpoint capacity, (i-l) coulombic efficiency, and (m-p) ΔV measured from UHPC cycling between 3.0–4.3 V at 40°C and at C/20 on NMC622a/gr, NMC622b/gr, NMC532(sc)c/gr, and NMC532(sc)u/gr cells (as indicated at the top of the figure). Cells were filled with 1.2 M LiPF6 in 3:7 EC/EMC and GA-based additive blends (as indicated on the figure). It is noted that a power disruption event affected the data precision of some cells, especially cycle 14 of several NMC532(sc)u/gr cells.

Voltage drop over time for cells initially at 4.3 V and stored at 60°C is shown in Figure 8 for NMC622a/gr and NMC622b/gr cells. For both cell chemistries, GA clearly outperforms CA at high temperature storage. In both cases, the best storage behavior is observed for the VC/GA blends, although in NMC622a/gr, this electrolyte actually underperforms 2% VC alone. The volume of gas produced during storage (Figure 9) supports the conclusion that GA-containing electrolytes perform better at high-temperature storage than those prepared with CA. However, in the 622a/graphite cells, the use of VC as a co-additive eliminated the gas suppression quality of GA. Given that the VC is not fully reduced in cells that also contain GA (as concluded from Figure 2), there is some of the additive left over in the electrolyte following formation. While the exact mechanism is not known, it is likely that VC oxidation leads to ‘cross-talk’. This occurs when oxidation products generated at the positive electrode cross over to affect the composition and stability of the SEI at the negative electrode.

Due to limited availability of materials and resources and the poor storage behavior of CA in both NMC622 cells, only the GA-containing additives were used for high-temperature storage in NMC532(sc)c/gr and NMC532(sc)u/gr cells. In the cells with the coated positive electrode material, 2% VC/0.5% GA and 2% VC/1% GA binary blends retain their voltage better than cells prepared with VC alone (Figure 10a). In contrast, 2% VC had the least cell voltage drop in NMC532(sc)u/gr cells (Figure 10b).
Figure 7. (a-d) Discharge capacity, (e-h) normalized (cycle 2) charge endpoint capacity, (i-l) coulombic efficiency, and (m-p) \( \Delta V \) measured from UHPC cycling between 3.0 – 4.3 V at 40°C and C/20 on NMC622a/gr, NMC622b/gr, NMC532(sc)/gr, and NMC532(sc)/u/gr cells (indicated at the top of the figure) that contained 1.2 M LiPF<sub>6</sub> in 3EC:7EMC and CA-based additive blends (as indicated on the figure).

It is again observed that GA and VC generally, but not always, decrease the volume of gas produced during high temperature storage (Figure 11). The exception is the combination of 2%GA/1%LFO in an uncoated NMC532(sc)/u/graphite cell, which produced slightly more gas than the additive-free control cells. However, when used in the coated NMC532(sc)/c/graphite cells, GA-containing solutions are observed to produce very little or even no gas after 500 h storage. The results support that GA may be a useful additive for applications where it is important to produce very little gas. The development of higher energy density batteries for portable electronics is one example where cell volume expansion can be catastrophic for device lifetime.

The R<sub>c</sub> following storage is compared for the GA-containing solutions in all four cell types (Figure 12). It is generally observed that the improvements of gas suppression and decreased voltage drop come with a trade-off of greater impedance growth over time. However, the R<sub>c</sub> following storage is nonetheless significantly less than observed immediately following cell formation.

Based on the high temperature storage results, combinations of GA with VC or LFO were identified as interesting candidates for prolonged cycling testing (Figure 13). Four types of pouch cells were used for these tests to examine the suitability of GA-containing electrolytes with different graphite sources [artificial (Figures 13a, 13e) vs. natural (Figures 13b, 13d)], cathode morphologies [polycrystalline (Figures 13a, 13b, 13e, 13f) vs. single crystal (Figures 13c, 13d, 13g, 13h)], and cathode composition [NMC622 (Figures 13c, 13g) vs. NMC532 (Figures 13d, 13h)]. In all cell types, electrolytes that contained only 2%VC or only 1%LFO are shown for comparison.

The results highlight the complex interplay between the electrolyte composition and the electrode compositions and morphologies. As expected, the single crystal positive electrode materials display greater capacity retention (Figures 13c–13d) and slower cell
Figure 8. Cell voltage in (a,b) NMC622a/graphite and (c,d) NMC622b/graphite cells was measured during open circuit storage at 60°C for 500 h, starting at 4.3 V. Electrolyte solutions contained 1.2 M LiPF6 in 3EC:7EMC and (a,c) GA- or (b,d) CA-based additive blends, as indicated on the figure.

Figure 9. Cell volume change due to gas production following 4.3 V storage for 500 h at 60°C and in (a) NMC622a/graphite and (b) NMC622b/graphite cells. Cells contained 1.2 M LiPF6 in 3EC:7EMC and GA- or CA-based additives, as indicated on the figure.

Figure 10. Cell voltage in (a) NMC532(sc)c/graphite and (b) NMC532(sc)u/graphite cells was measured during open circuit storage at 60°C for 500 h, starting at 4.3 V. Electrolyte solutions contained 1.2 M LiPF6 in 3EC:7EMC and GA-based additive blends, as indicated on the figure.

Figure 11. Cell volume change due to gas production following 60°C storage for 500 h and starting at 4.3 V in (a) NMC532(sc)c/graphite and (b) NMC532(sc)u/graphite cells. Cells contained 1.2 M LiPF6 in 3EC:7EMC and GA-based additives, as indicated on the figure.

Conclusions

Two relatively inexpensive organic anhydrides, glutaric anhydride (GA) and citraconic anhydride (CA) were tested as electrolyte additives for lithium-ion cells. The additives were tested singly and as binary additive blend components. Two positive electrode compositions (NMC622 and NMC532), each with two surface coatings, were tested, for a total of four cell material chemistries. The results exemplify the complex interplay that exists between electrolyte composition, electrode materials, and electrode surface coatings. Cells prepared with GA show very little gas production after formation or after high-temperature storage. GA-based additive blends lead to large CE and low ΔV growth during UHPC cycling at 40°C, and small voltage drop during storage at 60°C. However, GA leads to very large impedance during formation. Whereas CA-containing blends perform well in UHPC testing, they performed relatively poorly in
Figure 12. $R_e$ following 4.3 V storage for 500 h at 60°C and in a) NMC532(sc)c/gr and b) NMC532(sc)u/gr cells. Cells contained 1.2 M LiPF$_6$ in 3EC:7EMC and GA-based additives, as indicated on the figure.

Figure 13. (a-d) Normalized discharge capacity and (e-h) $\Delta V$ of pouch cells cycled at 40°C and C/3 charge-discharge rate between 3.0 – 4.3 V. Electrode compositions are indicated at the top of the figure (AG = artificial graphite, NG = natural graphite). Electrolyte additive concentrations are indicated in the figure legend.

high-temperature storage. Both additives may provide specific benefits for some applications, for example GA is very promising as a gas suppressant. Prolonged cycling testing demonstrates that GA is effective at suppressing long-term cell impedance growth in a variety of cell types, when used in combination with VC or LFO. GA/VC and GA/LFO cells also display good long-term capacity retention. It is further hoped that this work will contribute to larger, ongoing efforts to compare chemically similar additives in order to better understand the underlying chemistry behind their function in lithium-ion cells.

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