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New Jersey 08405

# **Measuring Energy Release of Lithium-ion Battery Failure Using a Bomb Calorimeter**

March 2016

Final Report

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16. Abstract <p>The high energy density of lithium-ion batteries (LIB) makes safe shipment as cargo on commercial aircraft a concern because of the potential for initiating or accelerating a fire. LIB failure caused by overheating, mechanical damage, or manufacturing defects results in rapid thermal energy release (thermal runaway), ejection of the cell contents, and the possibility of conflagration, burning, or explosion of the volatile organic electrolytes. Full-scale cargo fire tests at the Federal Aviation Administration have shown that these risks can be mitigated when LIBs are shipped at reduced electrical capacity (state-of-charge [SOC]). To quantify the safety benefit of shipping at reduced SOC, experiments were conducted using a bomb calorimeter to determine the relationship between the SOC of the LIB; its cell potential (volts) and electrical capacity (Coulombs); and the release of stored chemical energy during failure. Commercial LIBs in the form of single cylinders 18 mm in diameter and 65 mm long (18650 cells) were forced into failure in the bomb calorimeter using electrical resistance heating in a nitrogen atmosphere to preclude oxidation of the cell components. Data were collected for the release of stored energy as a function of electrical capacity and cell potential, and the composition of the combustible gases released during failure was determined by infrared spectroscopy. These studies showed that the stored electrochemical energy, which is the product of the actual charge and cell potential, is a better predictor of the energy release at failure than the fraction of the maximum rated charge capacity (SOC).</p>					
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## LIST OF ACRONYMS

LIB	Lithium-ion battery
SOC	State-of-charge

## EXECUTIVE SUMMARY

The high energy density of lithium-ion batteries (LIB) makes safe shipment as cargo on passenger aircraft a concern because of the risk of an internal short circuit causing a fire and the additional hazard associated with the burning of the organic electrolytes in a fire. Full-scale fire tests of bulk LIBs show that the risk and hazard of a fire in bulk shipments of rechargeable LIBs is lower when the fraction of the rated electrical capacity (state-of-charge [SOC]) is reduced. To quantify the safety benefit of shipping at reduced SOC, measurements were made to determine the relationship between the cell potential of the LIB, its charged capacity, and the release of stored energy as heat at failure. Four different LIB secondary cells were charged to various capacities and electrically heated to failure in a bomb calorimeter under inert conditions (nitrogen) to prevent oxidation of the cell components ejected at failure. The energy release of the LIB at failure was determined by a standard method. The volatiles produced by the mixing, reaction, and thermal decomposition of the battery components at failure were weighed to determine mass; the composition of the volatiles was determined by infrared spectroscopy.

Batteries with a high charge capacity contain more stored electrochemical energy at the same cell potential. This electrochemical energy is released inside the battery when a short circuit occurs because of a manufacturing defect, mechanical insult, heating in a fire, or, in this case, electrical resistance heating in a bomb calorimeter. The study found that the energy released from the LIBs at failure measured in the bomb calorimeter was roughly proportional to the charge capacity for these rechargeable batteries, which had similar cell potentials. Failure occurred in seconds because of rapid self-heating (thermal runaway) of the cell by internal discharge of stored electrochemical energy and the mixing, reaction, and thermal decomposition of the cell contents that were expelled by the high-pressure volatiles. The total energy released by all of these processes was measured in the bomb calorimeter and found to be proportional to the product of the charge capacity and voltage (i.e., the stored electrochemical energy).

## 1. INTRODUCTION

Rechargeable lithium-ion batteries (LIBs) are being used at an increasing rate since their introduction [1, 2] because of their high energy density and their ability to be used repeatedly with little to no degradation in performance. LIBs are continuously being researched and developed to produce higher capacities. These higher capacities result in more stored electrochemical energy that may be released rapidly in the event of an internal short circuit caused by a manufacturing defect, contaminant, mechanical insult, or the heat of a fire. The internal short circuit causes rapid electrical heating and an adiabatic temperature rise. It also causes the mixing, reaction, and thermal decomposition of the cell components in an autocatalytic process that culminates in expulsion of the cell contents, a process that is commonly referred to as thermal runaway. Therefore, the fire hazard of lithium-ion rechargeable cells and batteries shipped as bulk cargo on airplanes is the heat generated by thermal runaway that can propagate to adjacent cells, and the conflagration or explosion of the volatile cell contents ejected at failure.

In general, lithium-ion batteries consist of a cathode, anode, and electrolyte. The batteries used in this study consisted of a lithium transition metal cathode (e.g.,  $\text{LiCoO}_2$ ,  $\text{LiNiCoO}_2$ ,  $\text{LiNiCoAlO}_2$ ,  $\text{LiMn}_2\text{O}_4$ , etc.) attached to an aluminum terminal, a graphitic carbon anode attached to a copper terminal, and a liquid electrolyte between the electrodes comprised of a lithium salt and organic solvents. The batteries work by exchanging lithium ions between the positive and negative electrodes. This process is reversible with little to no change in the chemical structures of the components. In the rechargeable LIBs of this study, this layered construction is made in sheet form, which is rolled and inserted into a cylindrical steel jacket 18 mm in diameter by 65 mm in length, which is referred to as an 18650 cell.

In a previous laboratory study by the Federal Aviation Administration, the thermal hazard of an 18650 LIB during thermal runaway was measured using a purpose-built thermal capacitance calorimeter [3, 4]. The thermal hazard measurements of temperature history and mass loss did not include the total energy released as heat during thermal runaway because the cell contents were ejected from the cell at failure and the energy released by the mixing, reaction, thermal decomposition, and conflagration of the cell components occurred largely outside the calorimeter. The energy released by combustion of the contents of lithium metal (non-rechargeable) and lithium-ion (rechargeable) cells at failure was measured separately in a fire calorimeter [3, 4].

In the present study, the total thermal energy at failure of 18650 LIBs was measured in a bomb calorimeter using electrical resistance heating to drive the cell into thermal runaway in an inert atmosphere (nitrogen) to preclude oxidation/combustion of the ejected cell components. In this method, the cell contents are confined to the pressure vessel (bomb) and the total energy released during thermal runaway by the mixing, chemical reactions, and thermal decomposition of the cell components at failure could be measured.

## 2. MATERIALS

Cylindrical lithium-ion rechargeable (secondary) cells measuring 18 mm in diameter and 65 mm in length (18650) were purchased from commercial sources. Assemblies of these electrochemical

cells designed for a specific purpose are called batteries. Table 1 lists the manufacturer; cathode chemistry; rated and measured charge capacity; the nominal and measured cell potential; and the mass of these carbon-anode cells, which are shown in Figure 1.

**Table 1. Room temperature properties of rechargeable lithium-ion 18650 batteries**

Mfgr.	Cathode	Maximum Capacity, $Q_{\max}$ (A-s)		Cell Potential, $\epsilon$ (V)		Mass (g)
		Rated	Measured	Nominal	Measured	
Panasonic	LiMn <sub>2</sub> O <sub>4</sub> - LiNiCoO <sub>2</sub>	11,700	11,200	3.6	4.1	42
Tenergy	LiCoO <sub>2</sub>	9400	8300	3.7	4.1	48
Samsung	LiNiCoAlO <sub>2</sub>	5400	5000	3.7	4.1	42
UltraFire	Unknown	18,000	3600	3.7	4.0	40



**Figure 1. Lithium-ion cells (18650) tested in this study**

### 3. METHODS

#### 3.1 BATTERY CHARGING

The electrochemical cells in table 1 and figure 1 were charged to various capacities ( $Q$ ) using a commercial charging device (HiTec X4AC) that could simultaneously and individually charge four batteries at a time while providing  $Q$  and  $\epsilon$  for the individual cells, as shown in figure 2.



**Figure 2. Battery charger and fixtures used to charge four cells individually and simultaneously**

The charge  $Q$  is the electrical capacity of the cell in Amp-seconds. Zero charge ( $Q = 0$ ) was obtained by draining the cell by first discharging to the minimum 2.9 volts with the charger, then using a small light bulb connected to the terminals to drain the cell to zero volts (as indicated by the absence of luminosity). Using this procedure, the state-of-charge (SOC) recorded and reported for the cells of this study is the absolute fraction of the measured capacity of the cell:

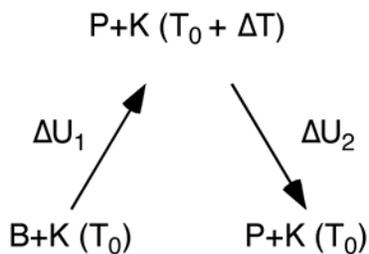
$$SOC(\%) = \frac{Q}{Q_{\max}} * 100 \quad (1)$$

The absolute measure of SOC used in this report differs from the conventional definition of SOC, which is a relative value based on the operating range and rated capacity of the cell. By convention, 10%–15% of the rated capacity is left in the cell at zero SOC to prolong the life of the cell. This could account for some of the differences between the rated and measured values in table 1.

### 3.2 ENERGY MEASUREMENTS

The generation of gaseous products during thermal runaway suggests that the best method to measure the energy of this process is at constant volume under inert conditions. An adiabatic bomb calorimeter is a standard, constant volume method of measuring the heat of a process in which some or all of the products are gases. The calorimeter bomb and water bath are part of a closed, thermally isolated system initially at ambient temperature ( $T_0$ ). The process of thermal runaway of a battery ( $B$ ) in a bomb calorimeter ( $K = \text{bomb} + \text{water bath}$ ) producing products ( $P$ ) under nitrogen (inert) conditions is shown schematically in figure 3 [5]. Because no heat is transferred ( $Q = 0$ ) and no pressure or volume work is done ( $W = 0$ ) in the thermally isolated, constant volume system, the internal energy change is zero for the first step,  $B+K (T_0) \rightarrow P+K (T_0+\Delta T)$ :

$$\Delta U_1 = Q + W = 0 \quad (2)$$



**Figure 3. Energy diagram for bomb calorimeter measurements**

For the second step,  $P+K (T_0+\Delta T) \rightarrow P+K (T_0)$ , the internal energy change is:

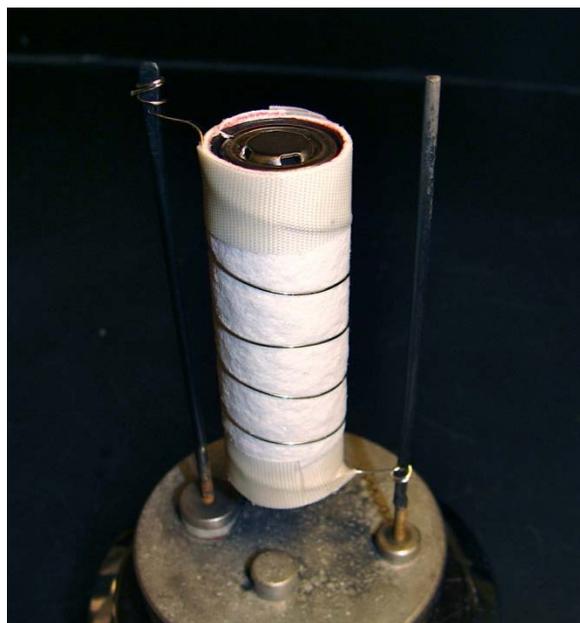
$$\Delta U_2 = C_{K+P} T_0 - C_{K+P} (T_0+\Delta T) = -C_{K+P} \Delta T \quad (3)$$

where  $C_{P+K}$  is the heat capacity of the bomb calorimeter and cell assembly shown in figures 4 and 5, determined by calibration. Because  $U$  is a state function, the internal energy change for the thermal runaway reaction of the battery producing products referenced to room temperature,  $B(T_0) \rightarrow P(T_0)$ , in the constant-volume bomb calorimeter is:

$$\Delta U_{\text{total}} = \Delta U_1 + \Delta U_2 = 0 - C_{P+K} \Delta T = -C_{K+P} \Delta T \quad (4)$$



(a)



(b)

**Figure 4. The (a) battery and components used for bomb calorimeter tests and (b) battery assembled with copper sleeve, ceramic paper, and heater wire attached to terminal posts of the bomb calorimeter prior to wrapping with mineral wool insulation for insertion into the bomb calorimeter**

Figure 5 shows the bomb calorimeter (Parr Instruments Model 1341, Plain Jacket Oxygen Bomb Calorimeter) that was modified to heat a single 18650 cell to failure using electrical resistance

heating and to measure the energy released at failure using a standard procedure with benzoic acid calibration [6].



**Figure 5. Bomb calorimeter setup showing variable AC power supply that powered the heater wire and multimeters that measured the voltage and current for the tests**

The posts in the bomb interior that usually hold the sample cup and connect to the ignition wire were replaced with longer posts that served as leads to electrical resistance wire. Prior to testing, the plastic sheath was removed from the cell, which was placed in a copper sleeve covered with a thin ceramic paper to electrically insulate the copper from a 46-cm length of 24-gauge nickel-chromium (Nichrome) resistance wire with a resistance of 2.2–2.4 Ohms, as shown in figure 4(a). The heating wire was wrapped around the cell/copper/paper assembly and connected to the terminal posts to make an electrical connection and suspend the cell in the bomb, as shown in figure 4(b).

The components in figure 4(a) were assembled as shown in figure 4(b) and then wrapped in ceramic wool (Kaowool) insulation before placing it in the calorimeter pressure vessel (bomb). The bomb was purged several times with ultra-high purity nitrogen to remove all oxygen and sealed at 1 bar of nitrogen pressure. Purging the bomb with nitrogen precluded any heat associated with oxidation of the cell components at failure during the test. A voltage (8 V) was applied to the wire for 15 minutes, and the measured current ( $\approx 3A$ ) and voltage were used to calculate the power delivered to the bomb to heat the cell to the failure temperature of approximately 250°C.

During a test, as the battery is heated, the temperature of the water bath rises slowly until the battery reaches approximately 250°C, at which point thermal runaway commences releasing energy, as indicated by a sharp rise in the water bath temperature. The temperature of the system was monitored and recorded until thermal equilibrium was reached, which usually occurred in 40–60 minutes. During that time, the temperature of the water bath increased between 3°C and 8°C, depending on the charge on the battery. For the initial tests, after the steady temperature rise

was recorded, the system was cooled with ice to the starting temperature, and the same heating program was used to generate a temperature rise of approximately 2°C for the heating program that was subtracted from the temperature rise in the test. For later tests, the time-integrated electrical power (energy) calculated from the recorded current and voltage of the heating wire in the bomb for the 15-minute heating period was subtracted from the energy of cell failure calculated from the temperature rise in the bomb calorimeter.

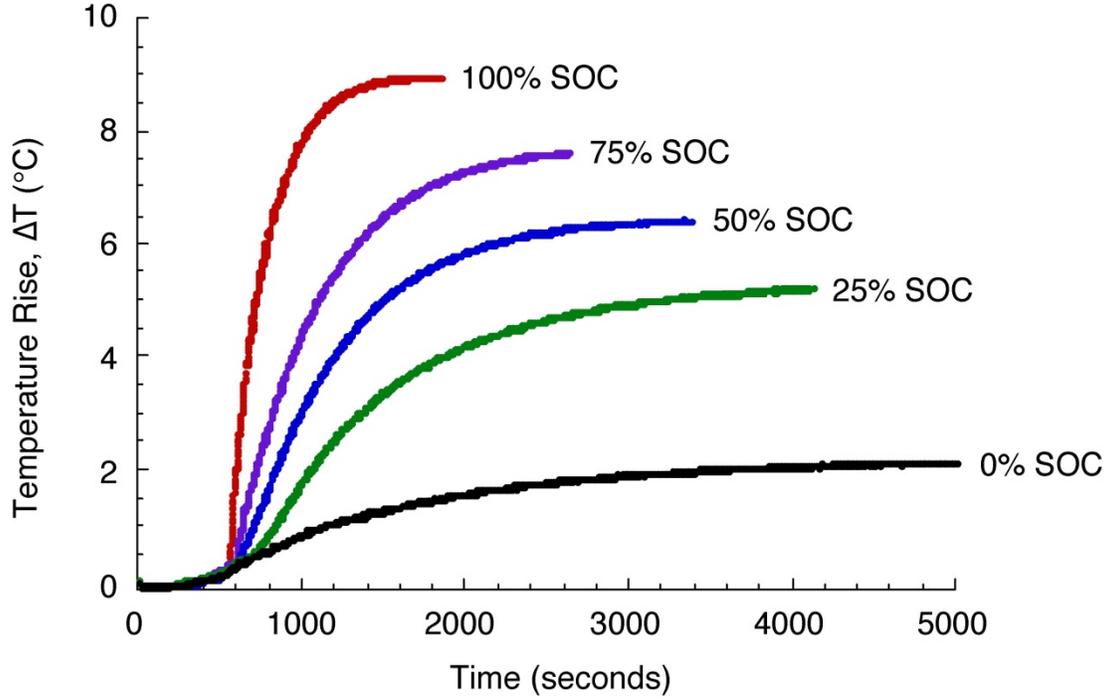
The bomb calorimeter heat capacity ( $C_{K+P}$  of equation 3) was calibrated according to the standard method [6] using combustion of benzoic acid in 30 atmospheres of pure oxygen to generate  $\Delta U_{\text{total}}$ . The contents of the bomb for the calibration were identical to the setup for the battery tests, except that a 44-g aluminum cylinder was used to approximate the thermal mass of a rechargeable 18650 battery (see table 1).

In addition to measuring the temperature rise of the bomb, a gravimetric analysis was also performed. Because the bomb is a closed system, all of the mass should remain constant for the test and subsequent baseline. The bomb was weighed before and after the test to ensure no mass was lost or gained from the bomb leaking/taking on water. Once the bomb was prepared with the sample and purged with nitrogen, it was weighed to get an initial weight. The test (and optional baseline) was then performed. The bomb was then removed from the water bath and blown dry with compressed air. The dry weight was measured and the bomb was vented to release gaseous reaction and decomposition products into a fume hood or into a gas sample bag for further analysis. The bomb was then reweighed to obtain the weight of volatiles generated during thermal runaway.

## 4. RESULTS AND DISCUSSION

### 4.1 ENERGY MEASUREMENTS

Figure 6 shows the temperature rise of the water bath for the LiCoO<sub>2</sub> cell at different SOC. The baseline-corrected steady-state temperature rise,  $\Delta T$ , multiplied by the measured  $C_{K+P} = 10.3$  kJ/°C, gives the total (internal) energy released at cell failure for each LIB at each SOC.

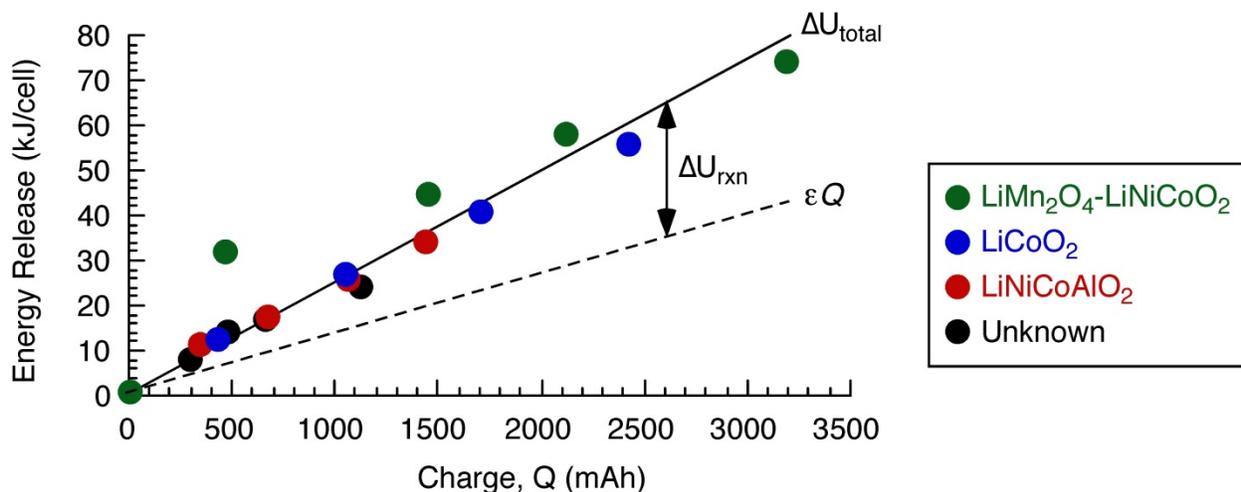


**Figure 6. Baseline corrected temperature rise of the LiCoO<sub>2</sub> cell in the bomb calorimeter at different states of charge**

Figure 7 shows the total energy released from the LIBs at cell failure  $\Delta U_{total}$  versus charge,  $Q$ . The  $\Delta U_{total}$  is roughly proportional to  $Q$  for these LIBs with similar cell potential, which means that high-capacity LIBs will release more thermal energy at failure than low-capacity cells at the same SOC. If the total energy of thermal runaway can be separated into an energy released by mixing, reaction, and thermal decomposition of the cell contents,  $\Delta U_{rxn}$ , and the stored (free) electrochemical energy,  $\Delta G$ ,

$$\Delta U_{total} = \Delta U_{rxn} - \Delta G = \Delta U_{rxn} + \epsilon Q \quad (5)$$

Using the convention that energy release is a positive quantity and applying equation 5 to the slope of the line in figure 7,  $\Delta U_{total} \approx 1.7\epsilon Q$ . This means that  $\epsilon Q$  accounts for approximately 60% of  $\Delta U_{total}$ , whereas  $\Delta U_{rxn}$  accounts for approximately 40% of  $\Delta U_{total}$  measured in the bomb calorimeter.



**Figure 7. Energy release versus charge for the 18650 LIBs**

#### 4.2 VISUAL OBSERVATIONS

It was observed that none of the cells having zero SOC ( $Q = 0$ ) ejected their contents at failure during thermal runaway. By comparison, half of the cells at 20% SOC, most of the cells at 50% SOC (with the exception of the UltraFire cells), and all of the cells at 100% SOC ejected their contents. Figure 8 is a photograph of one cell that did not eject its contents (top) and one that did eject its contents (bottom) during the bomb calorimeter test. All of the batteries lost mass because of the venting of gases at approximately 200°C [3, 4] during the heating program regardless of their state of charge, with failure occurring rapidly at approximately 250°C; liquid electrolyte and solid electrodes and terminals expelled into the bomb at higher SOC. The amount of electrolyte released was not quantified in this study; however, it contributes to the total fire hazard of the batteries [7–11].



**Figure 8. LIBs after thermal runaway in the bomb calorimeter: the top cell did not eject its contents, but the bottom cell did**

#### 4.3 GRAVIMETRIC AND SPECTROSCOPIC ANALYSIS

The mass of volatiles produced by cell failure during the heating program was measured by weighing the pressure vessel (bomb) after the test, venting the pressurized contents to the atmosphere, and reweighing the bomb to determine the mass of volatiles lost by venting. Figure 9 shows that the mass of volatiles produced at cell failure is roughly proportional to the charge on the battery regardless of the maximum capacity (i.e., regardless of the SOC). Infrared spectra for the gases were obtained and several qualitative compounds were identified. Most of the gases that are evolved are hydrogen, flammable hydrocarbons, carbon monoxide, and carbon dioxide, the latter three of which are shown qualitatively in the Fourier transform infrared spectra of the vented gases in figure 10. Though the bomb is purged of all oxygen, decomposition of the cathode releases oxygen that can combine with the carbon anode and electrolytes to form CO and CO<sub>2</sub> [10]. The combustible gases, when released into a confined space, can create an explosive mixture [7].

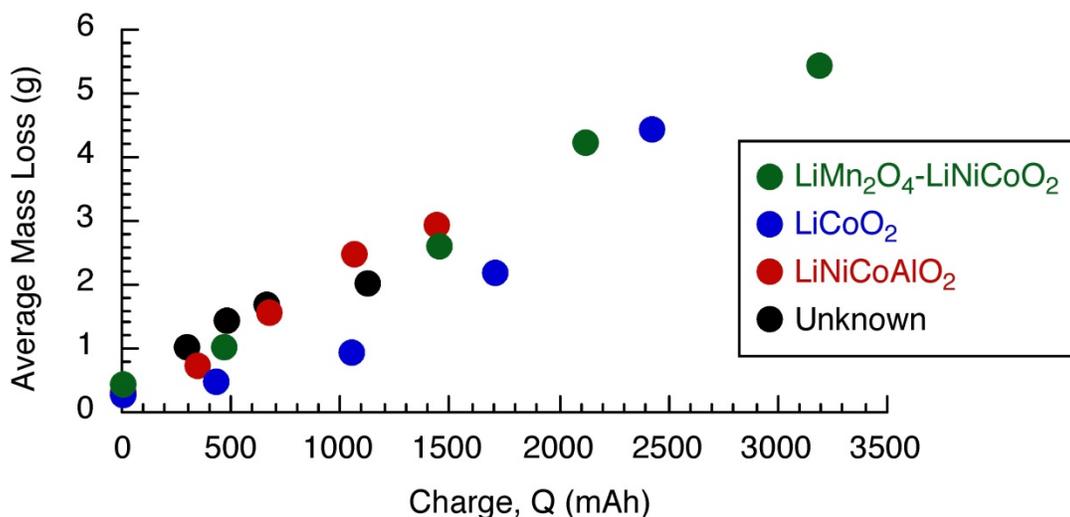


Figure 9. Mass of volatiles produced at cell failure versus charge for the 18650 LIBs

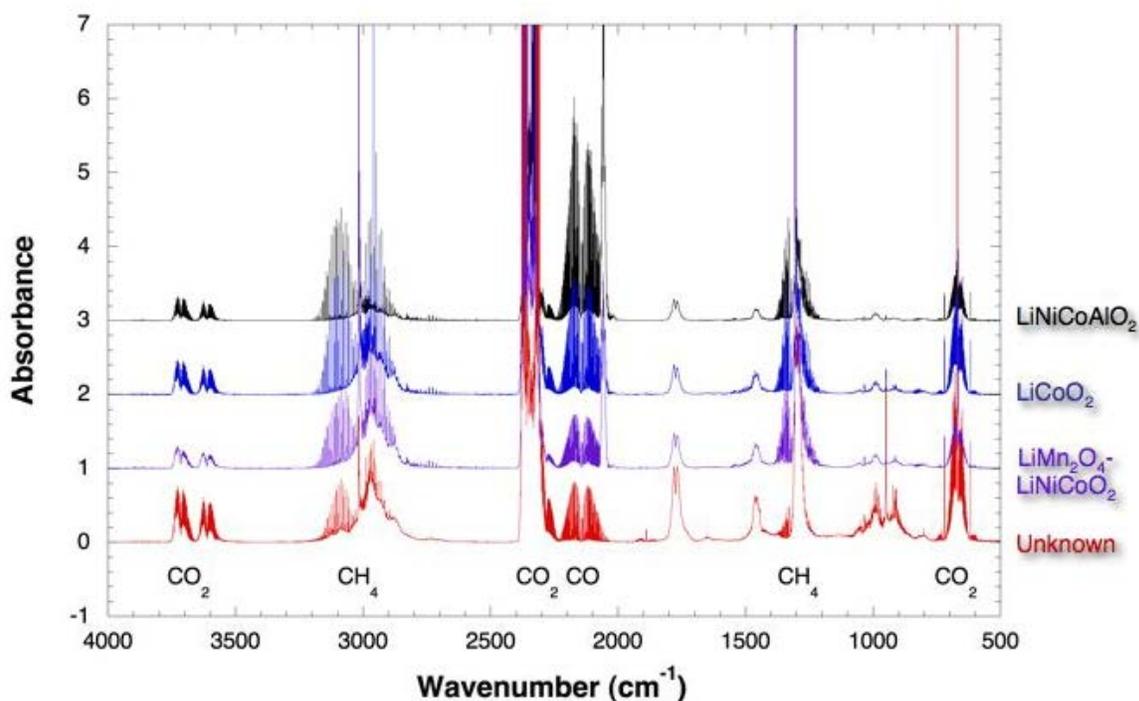


Figure 10. Infrared spectra of the volatiles produced during cell failure of the LIBs

## 5. CONCLUSIONS

A standard test method for measuring heats of combustion of organic compounds using a bomb calorimeter can be adapted to measure the energy and products released when a battery fails and overheats because of an internal short circuit. In the proposed method, electrical resistance heating is used to force the cell/battery to failure in a pressure vessel (bomb) under nitrogen to preclude air oxidation of the expelled battery contents. The energy released at cell failure is

measured by the standard procedure, and the composition and heat of combustion of the volatile products is determined by venting the bomb to suitable analytical instrumentation after the test. The method can be applied using commercial equipment to larger cells and multi-cell battery packs, and the measured energies can be used to estimate the thermal and fire hazards of batteries shipped as bulk cargo.

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