RECHARGE

ADVANCED RECHARGEABLE & LITHIUM BATTERIES ASSOCIATION

Labs test plan, phase 2 results analysis

Dec 9, 2020

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Content

General status of informal working group and test group

Labs test plan

Phase 1 results summary

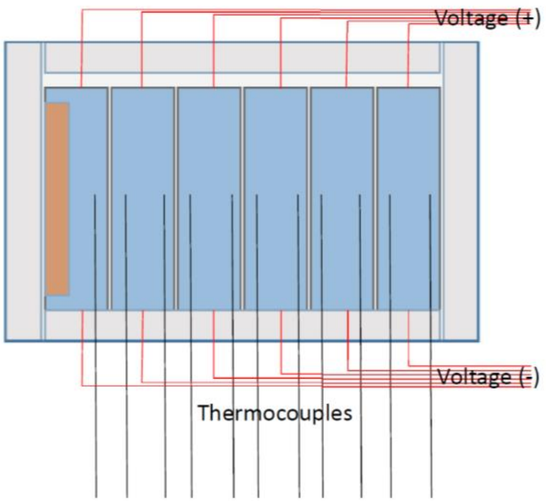
Phase 2 preliminary results

General Status of Informal Working Group (IWG) and Test Group

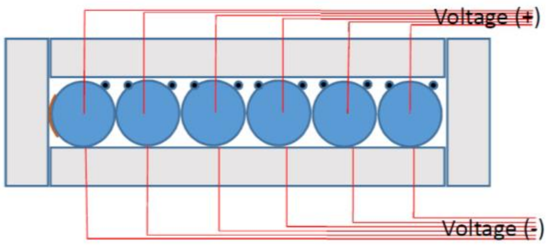
- Last IWG meeting held in Arlington, Texas USA (October 2019)
  - Reviewed data on first round of testing from 7 labs
  - All labs received from PRBA same Li ion cell designs from same manufacturers, tested at 100% state of charge
  - See UN/SCETDG/56/INF.33 for minutes of Arlington meeting
- IWG meeting scheduled for September 2020 in Brussels cancelled due to COVID-19 pandemic
- Test Group meeting scheduled for May 2020 also cancelled, continued web-based meetings, developed second round of tests
  - Additional Li ion cells provided by PRBA to 8 labs, tested at different states of charge
  - Partial results provided today for review by IWG
- Next IWG planned for December 2020 after UN Sub-Committee meeting

Test plan phase 1

- Phase 1: Test of repeatability: 7 labs tested propagation in a row of 6 cells, 3 répétitions of test for pouch and cylindrical, at 100% SOC.



Side View



Top View

Test plan phase 2

- test of effect of heating rate and SOC on propagation for 6 cells in a row (for test labs without gaz analysis, test series 2 and 5): received partial tests of 4 labs has of sept 1.
- test of effect of heating rate and SOC on propagation on gaz hazards, single cell test (for labs with gaz analysis, test series 4 and 6): received partial test of 2 labs has of Sept 1.

Experimental Procedure test series 2-5:

- Charge cells to SoC as in table below
- Heat cells according to Table 1.
- Each test is performed once

Table 1

	20% SoC	30% SoC	50% SoC	70% SoC	100% SoC
5 C/min					Priority #3
20 C/min	Priority #1	Priority #5	Priority #2	Priority #4	

Test phase 2 results: table completed with all results received as of Dec 7th 2020

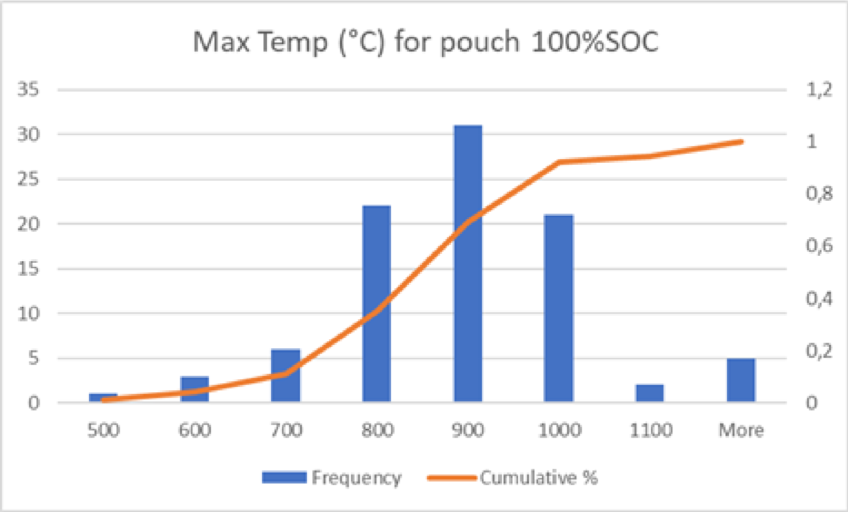
cyclindrical	2450 mAh																	
Pouch	4800 mAh									based on the reading of the thermocouple opposite side of the initiation cell.								
										time of TR	Cell 2	Cell 3	Cell 4	Cell 5	Cell 6	Cell 1	Cell 2	Cell 3
	cell type	heating device type	Heating power	heating ra	SOC	max temp	Temp 3rd	compressi	Closed cha	seconds	delta sec	delta sec	delta sec	delta sec	delta sec	Max temp	Max temp	Max te
L1-1	NMC/cylindrical	Omega heating pad (2,5 x 5	20	10	100	838			N	1050	28	110	194	163	33	711	793	
L1-2	NMC/cylindrical	Omega heating pad (2,5 x 5	20	11	100	815			N	1102	9	67	42	106	187	722	815	
L1-3	NMC/cylindrical	Omega heating pad (2,5 x 5	20	19	100	867			N	650	29	22	92	77	56		834	
L1-4	NMC/Pouch	Omega heating pad (5 x 5 cn	40	5	100	850			N	2208	4	5	5	4	5	534	1400	
L1-5	NMC/Pouch	Omega heating pad (5 x 5 cn	40	9	100	825			N	1212	4	2	1	91	2	794	825	
L2-1	NMC/cylindrical	heating tape		10,7	100	984			N	600	2	13	133	1	50	984	879	
L2-2	NMC/cylindrical	heating tape		10,9	100	909			N	600	0	0	1	33	16	826	879	
L2-3	NMC/Pouch	heating tape		9,6	100	841			N	900						784	780	
L2-4	NMC/Pouch	heating tape		10,2	100	1012			N	900	1	1	6	6	2	928	988	
L3-1	NMC/cylindrical	25 Watt cartirdge heater (4 heaters utilized)		20,9	100	853			Y	575	96	17	60	12	14	853	825	
L3-2	NMC/cylindrical	25 Watt cartirdge heater (4 heaters utilized)		18	100	932			Y	433	94	8	25	17	23	761	932	
L3-3	NMC/cylindrical	25 Watt cartirdge heater (4 heaters utilized)		20,9	100	848			Y	581	82	15	15	16	14	848	842	
L3-4	NMC/Pouch	Capton Heaters, 2in. X 2in., 10W/Sq.In. (2 heat		17,5	100	826			Y	577	43	6	4	9	8	785	805	
L3-5	NMC/Pouch	Capton Heaters, 2in. X 2in., 10W/Sq.In. (2 heat		20,5	100	826			Y	515	7	5	4	1	6	1034	819	
L3-6	NMC/Pouch	Capton Heaters, 2in. X 2in., 10W/Sq.In. (2 heat		15,8	100	826			Y	586	5	3	1	1	0	871	1164	
L4-1	NMC/cylindrical	Kapton thermal pad (Omega) 10W/Inch2 (1Inc		10	100	1000			N	2135	12	21	33	84	33	900	900	
L4-2	NMC/cylindrical	Kapton thermal pad (Omega) 10W/Inch2 (1Inc		10	100	1000			N	2121	13	20	34	82	35	620	900	
L4-3	NMC/cylindrical	Kapton thermal pad (Omega) 10W/Inch2 (1Inc		10	100	1000			N	2438	2	53	50	92	120	1000	1000	
L4-4	NMC/Pouch	Kapton thermal pad (Omega)		10	100	2,5			N	2400	10	7	5	5	4	750	850	
L4-5	NMC/Pouch	Kapton thermal pad (Omega)		10	100	2,5			N	5030	7	5	3	6	3	1000	900	
L4-6	NMC/Pouch	Kapton thermal pad (Omega)		10	100	2,5			N	4955	10	5	5	2	2	800	840	
L5-1	NMC/cylindrical	Minco		18	100	850	35		Y	982	6	2	44	2	2	730,7	855,9	
L5-2	NMC/cylindrical	Minco		18	100	769	35		Y	614						769,1	101,4	
L5-3	NMC/Pouch	Minco (x3)		7	100	832	122		Y	1593	5	6	6	12	13	643,1	772,7	
L6-1	NMC/cylindrical	Heating pad (thin film)		7	100	1800			N	1278	43	49	38	83	32	1294	1320	
L6-2	NMC/cylindrical	Heating pad (thin film)		7	100	1800			N	1130						1130	135	
L6-3	NMC/Pouch	Heating pad (thin film)		10	100	780			N	1639	8	6	6	8	9	680	780	
L6-4	NMC/Pouch	Heating pad (thin film)		8	100	860			N	1675	5	3	10	4	9	860	834	
L7-1	NMC/cylindrical	Heating pad (30x30 mm, 20W)		20	100	802			N	258	74	24	35	24	50	802	736	
L7-2	NMC/cylindrical	Heating pad (30x30 mm, 20W)		20	100	904			N	482	5	27	28	14	30	904	718	
L7-3	NMC/cylindrical	Heating pad (30x30 mm, 20W)		20	100	810			N	378	11	34	53	18	28	764	717	
L7-4	NMC/Pouch	Heating pad (70x60 mm, 55W)		15	100	935			N	770	5	3	3	2		935	924	
L7-5	NMC/Pouch	Heating pad (70x60 mm, 55W)		15	100	1331			N	640	4	4	4	4		863	1331	
L7-6	NMC/Pouch	Heating pad (70x60 mm, 55W)		15	100	1352			N	605	4	4	5	3		1352	912	
L1-21	NMC/cylindrical	Omega heating pad (2,5 x 5	20	5	100	669	50	briks	Y	1980	62	31	81	22	7	669	757	
L1-22	NMC/cylindrical	Omega heating pad (2,5 x 5	20	14	70	738	33	briks	Y	771,428571	120	120	0	170	122	737,91	617,32	7
L1-23	NMC/cylindrical	Omega heating pad (2,5 x 5	20	20	50	578	29	briks	Y	504	353	265	267	264	268	556,71	561,42	5
L1-24	NMC/cylindrical	Omega heating pad (2,5 x 5	20	20	20	200		briks	Y	0	0	0	0	0	0	361	213	

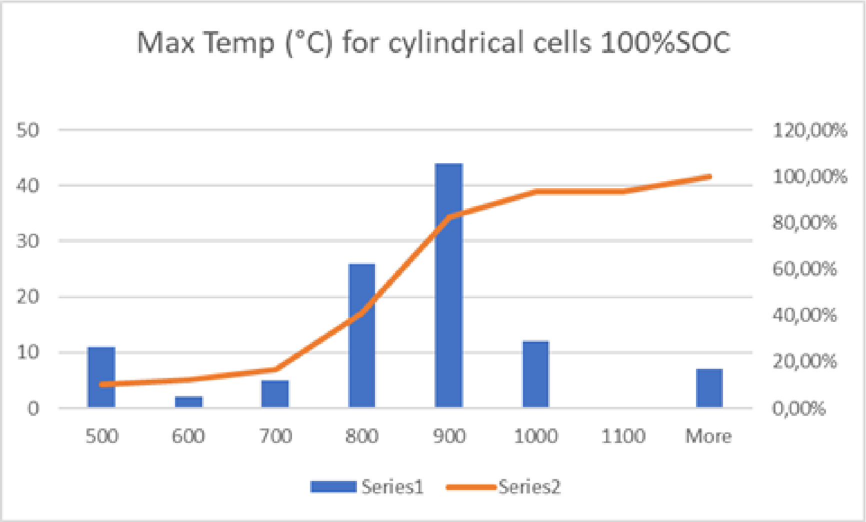
Phase 1 results analysis: Max temperature

- Typical max temperature for both type of cells is about 900°C stdandard deviation around 100°C.

Cylindrical

Pouch



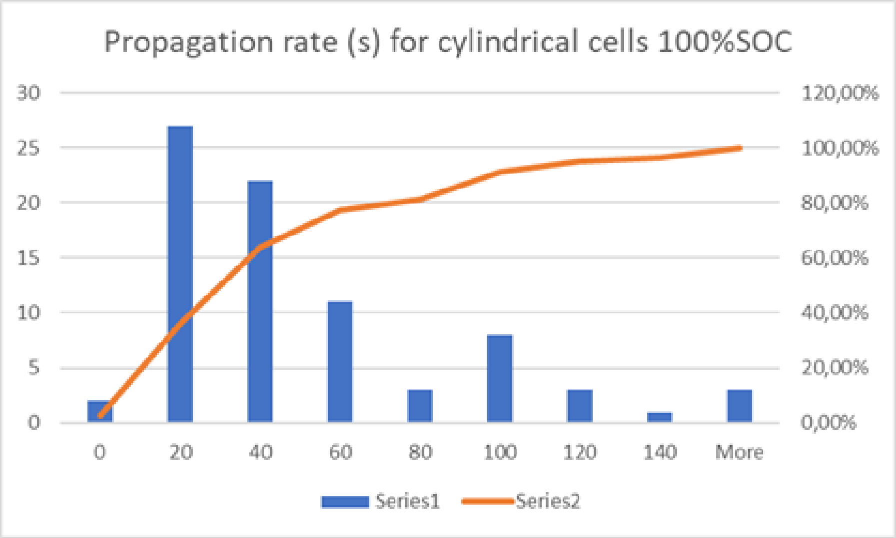


the temperature increase represents an amount of energy of 1.2 MJ/kg (including the contained electrical energy of around 0.6 MJ/kg) . This value is well correlated with literature data.

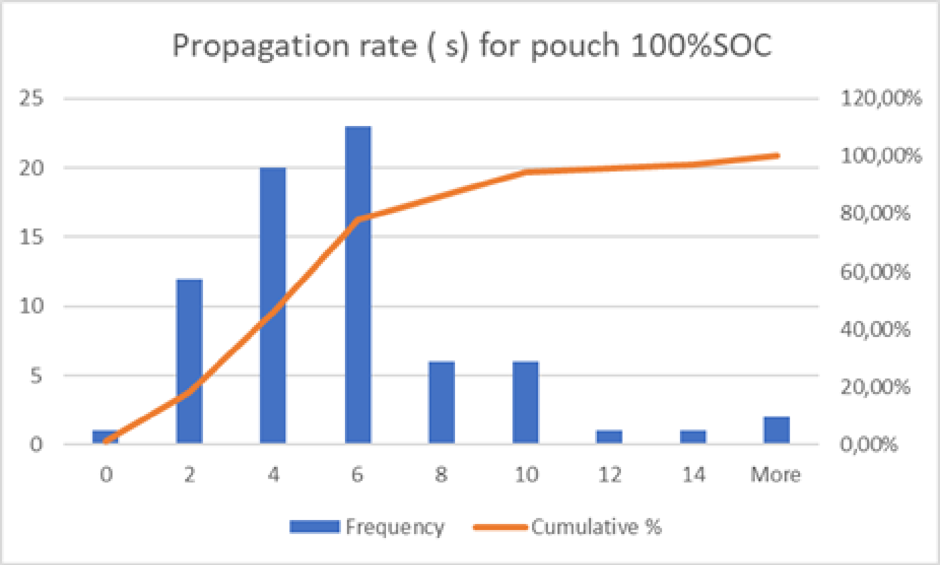
Phase 1 results analysis: Propagation time

- Very different typical time for cyl and pouch cells, due to larger surface of contact

Cylindrical



Pouch

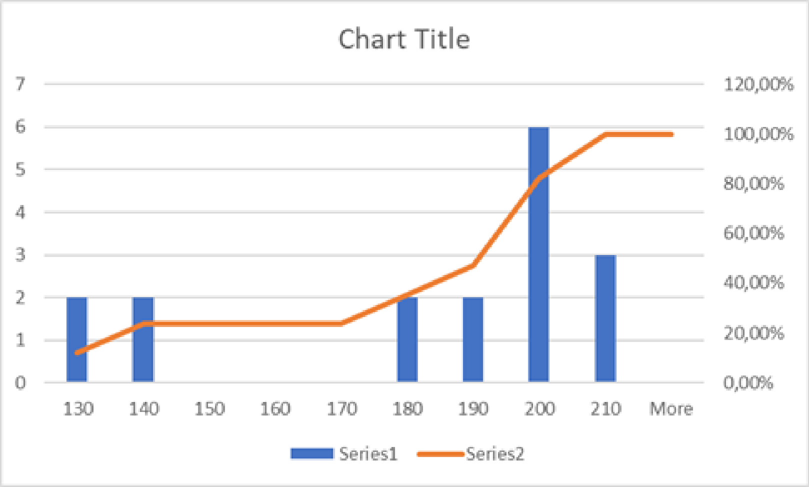


Dispertion observed due to lack of reproducible compression, cells core ejections for cyclindircal cells

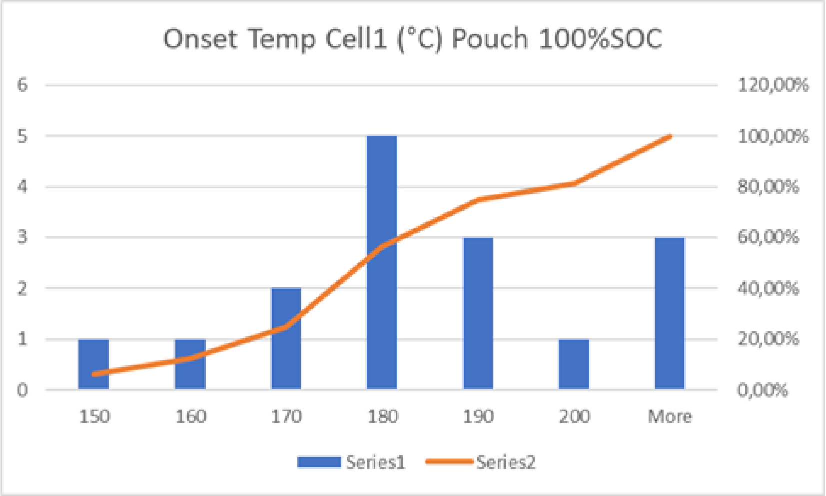
Phase 1 results analysis: TR onset temperature

- Typical temperature of onset around 180°C for pouch, more dispersion with cyclindrical

Cylindrical



Pouch



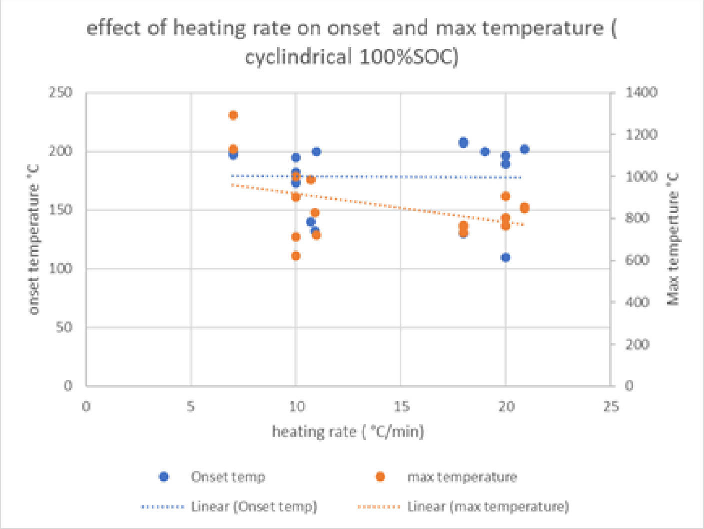
Dispertions observed due to various methods for measure, and dynamics of the TR.

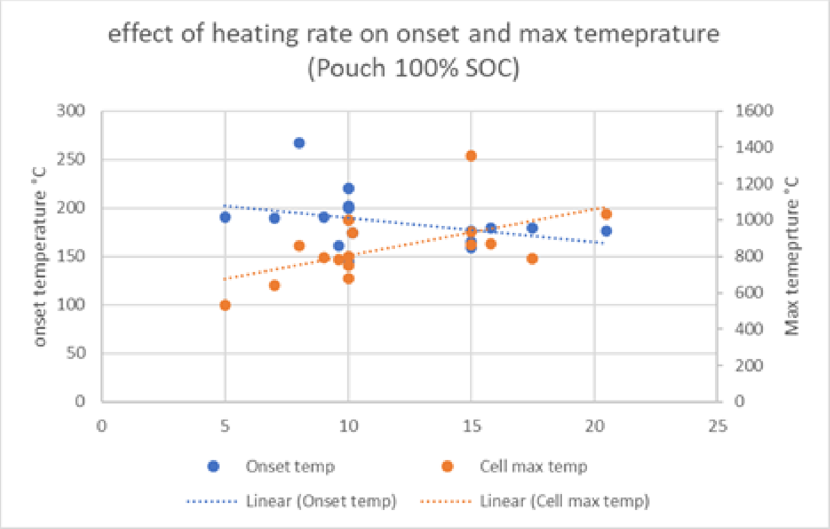
Phase 1 results analysis: TR onset temperature correlation with heating rate

- At 100% SOC, No clear correlation

Cylindrical

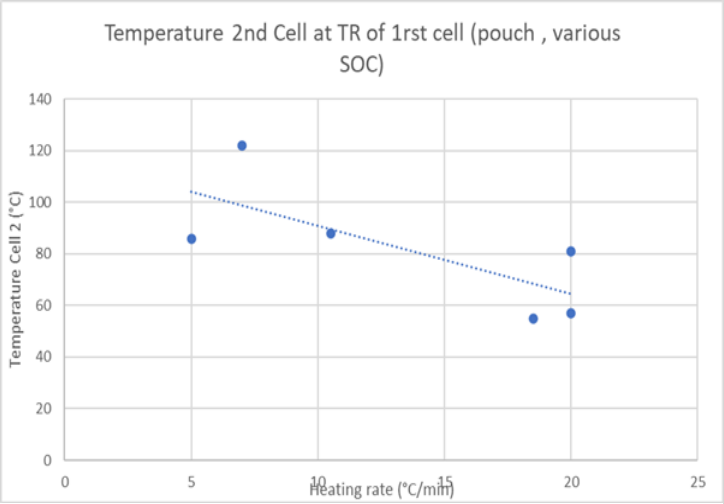
Pouch

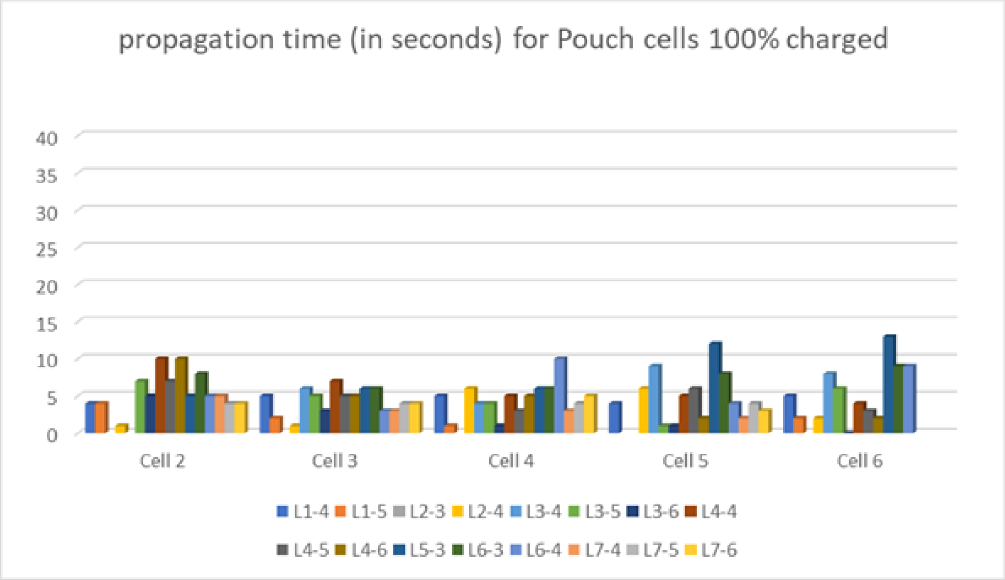




Representativeness of the TR obtained with the heater: propagation rate

- At 100% SOC, Pouch cells, no difference between cell2 and 6 in a row. for the propafation time, despite pre-heating observed.



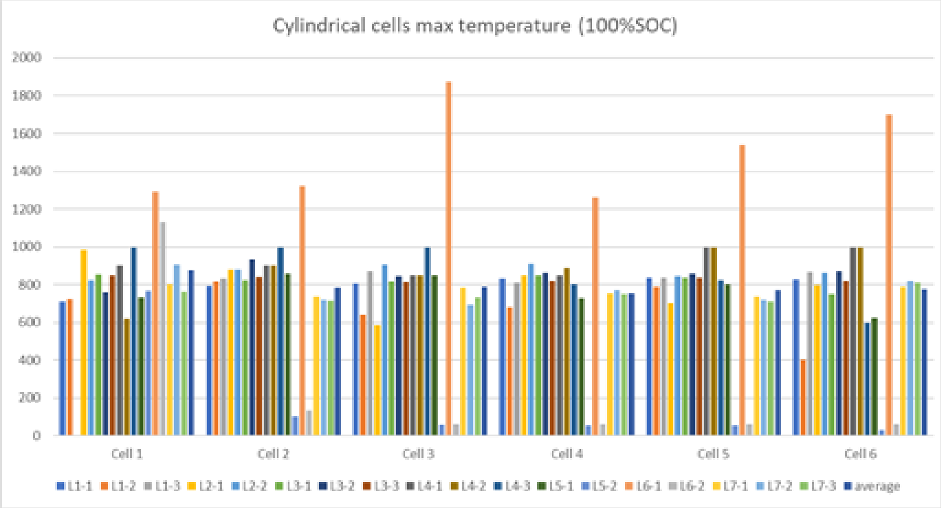


"L1-2": means test of laboratory 1, test number 2. The individual test result for each cell is represented by a single bar. Cell 1 is the initiation cell, cell 2 the next cell in the row, etc…

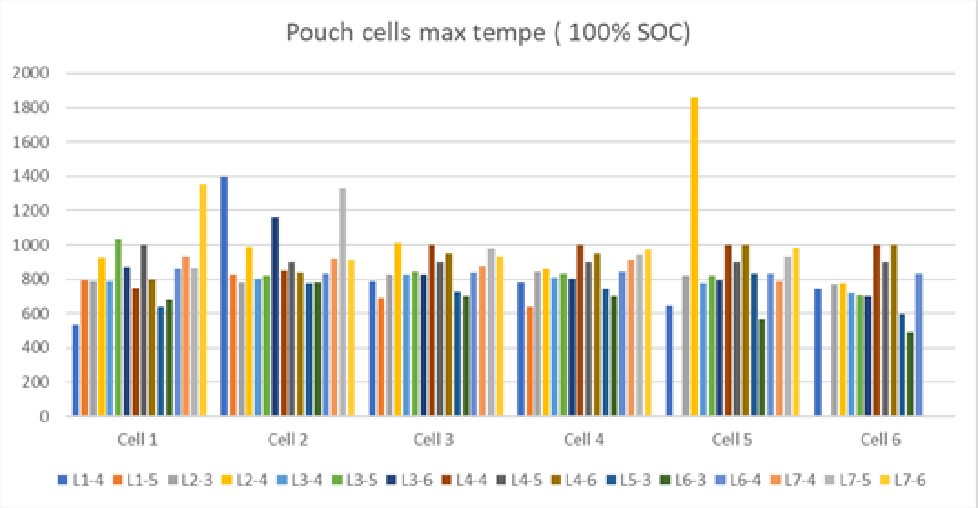
Representativeness of the TR obtained with the heater: max temperature

- At 100% SOC, non difference observed between initiation cell and others cells

Cylindrical

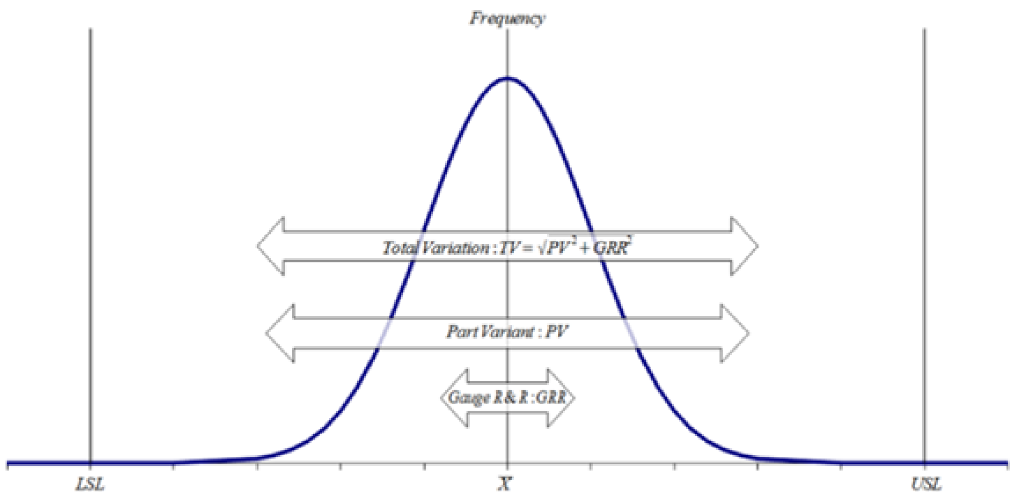


Pouch



Phase 1 results analysis: repeatability and reproducibility

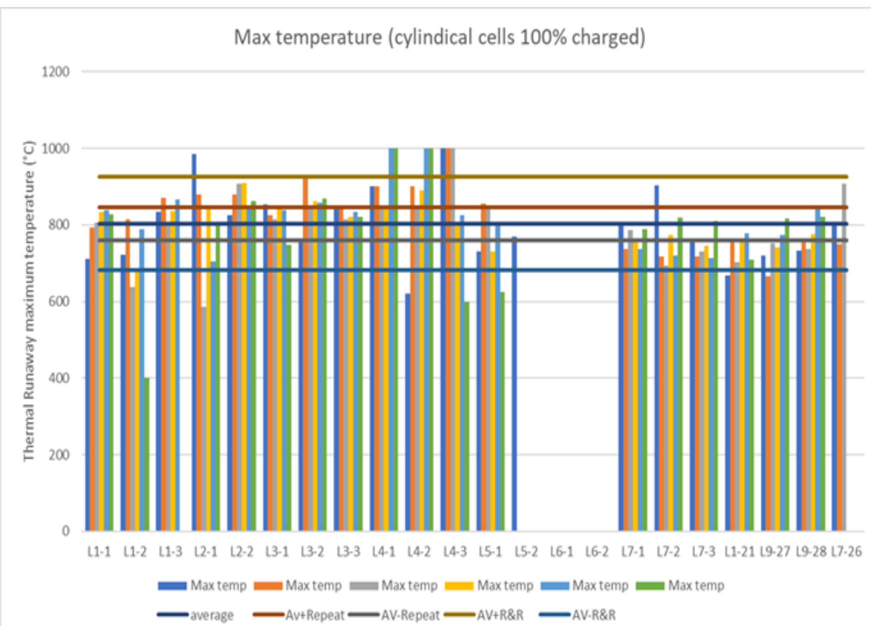
- Repetability (per lab): stability of averages and standard deviation and reproducibility (between labs): stability of averages and standard deviation:.
- The Repeatability and Reproducibility are combined to assess the total potential variations of a measured result, in the R&R approach



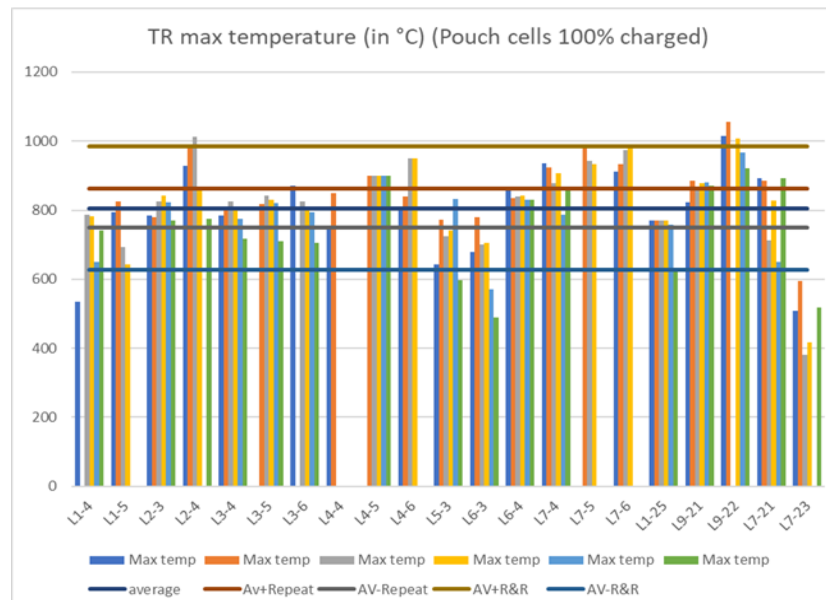
R&R for max temperature

main source of variation for the measure of the maximum temperature is the **reproductibility**, for both type of cells: the repeatability (variation within each laboratory) is in the range of 50°C (about 6% of the average), but the R&R, including the reproducibility (variations between labs average) is more than 100°C. It supports the need to better harmonize the conditions of test between the labs to achieve a better reproducibility (effect of lid!).

Cylindrical



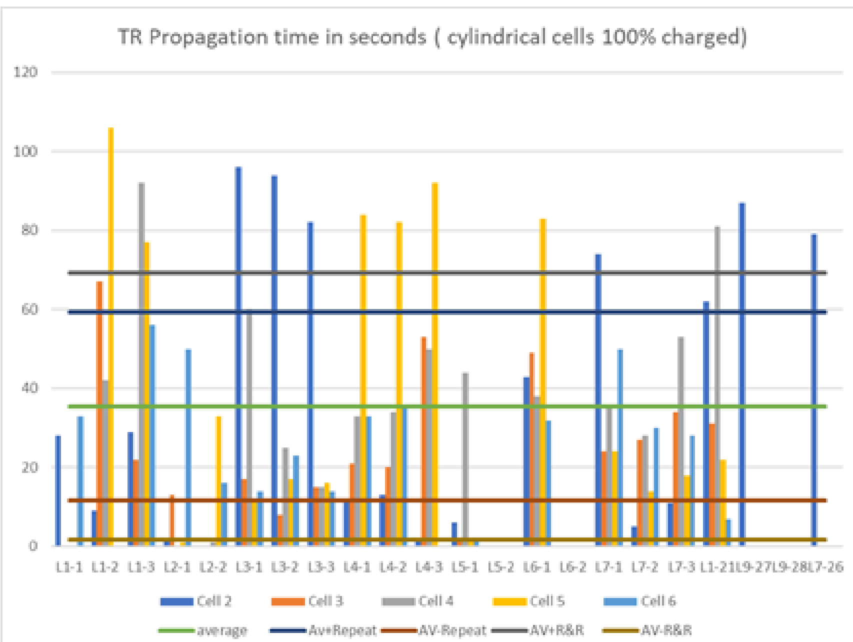
Pouch



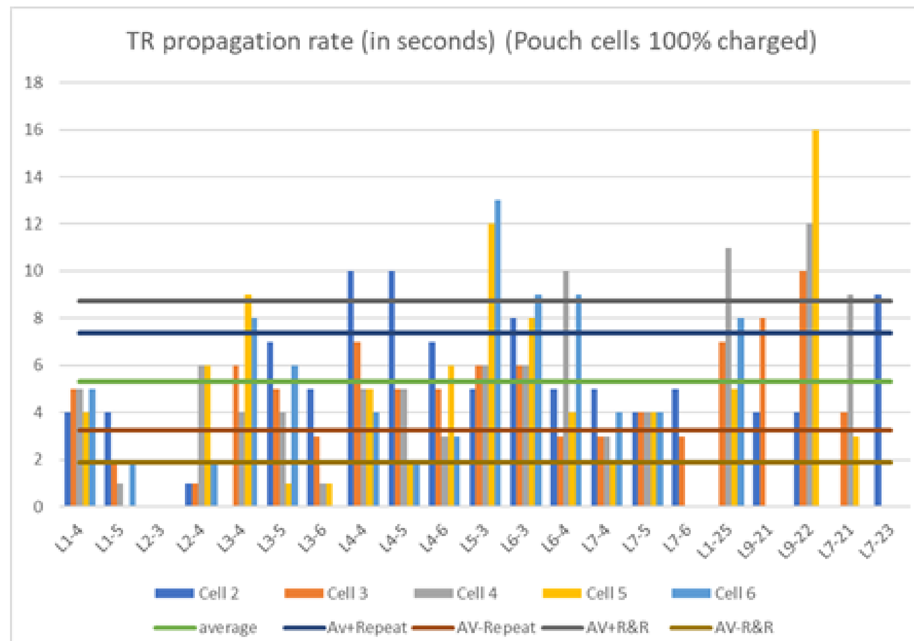
R&R for propagation rate

The more important variation source for the propagation time is the lack of **repeatability,** particularly for the cylindrical cells (67% of the average time). This indicates that the test conditions should be specified more precisely, such has the compression between cells in the case of cylindrical cells.

Cylindrical



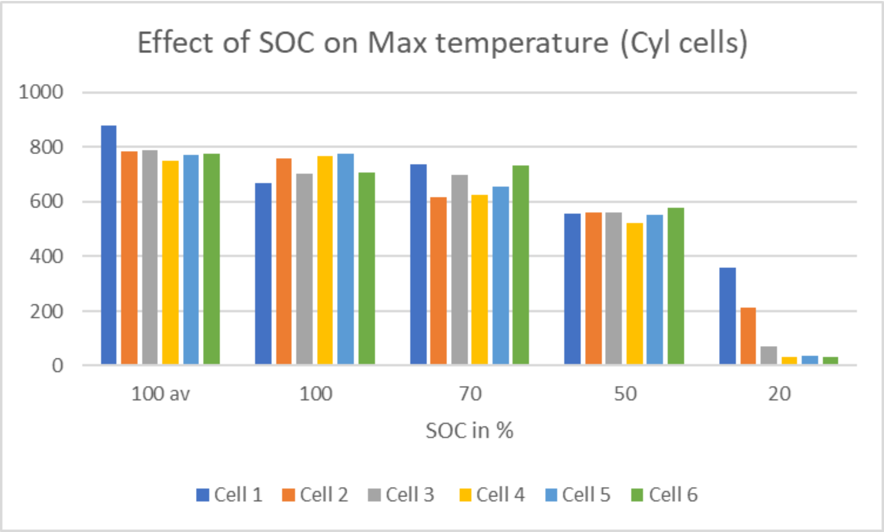
Pouch

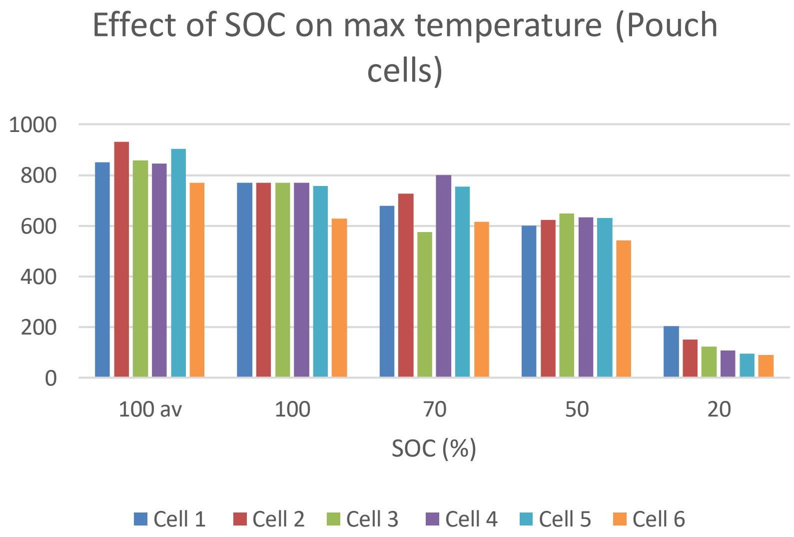


Note: L1-2 means « laboratory 1 test 2 »

Phase 2 results analysis: effect of SOC on max Temp

- Stability of the max temperature during self propagation at various SOC: the initiation method seems of little impact on the reaction, except in absence of propagation (20% SOC)

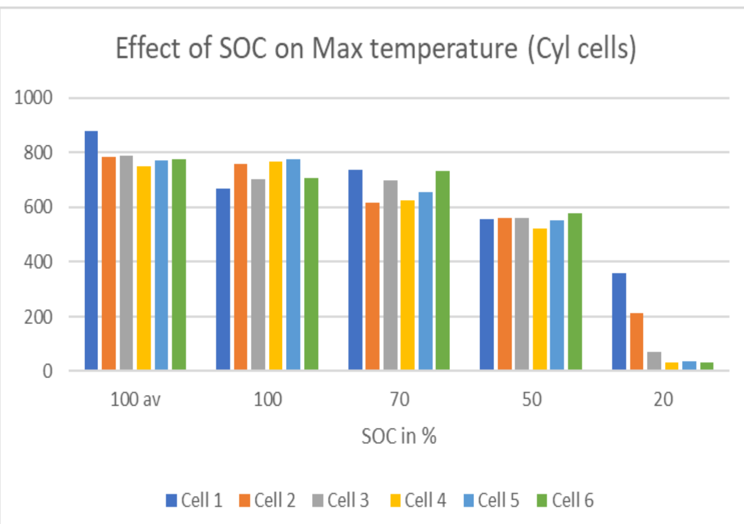




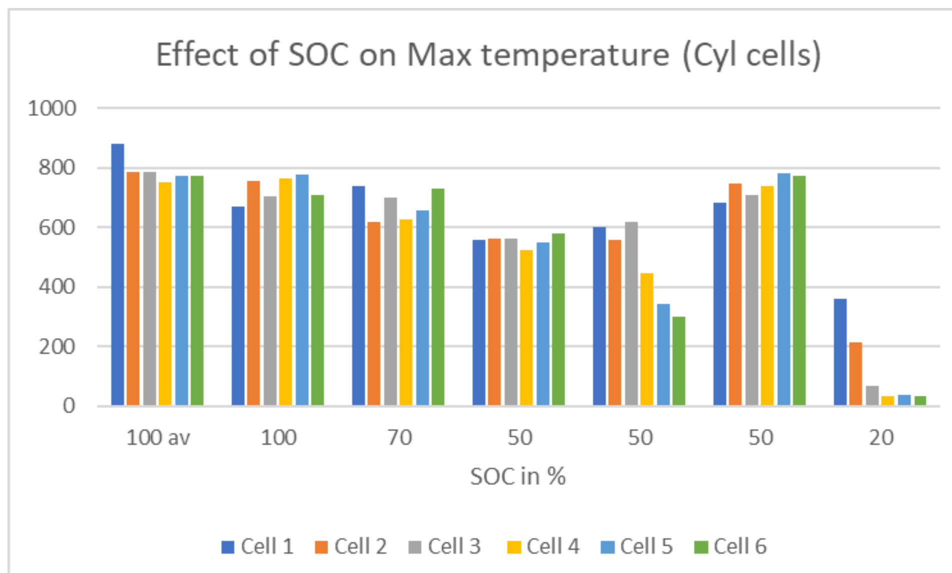
Phase 2 results analysis: effect of SOC on max Temp, reproducibility

- But repetability within a lab is much better than reproducibility interlabs, particumarly at intermediate SOC 30-50%

Results of 1 lab



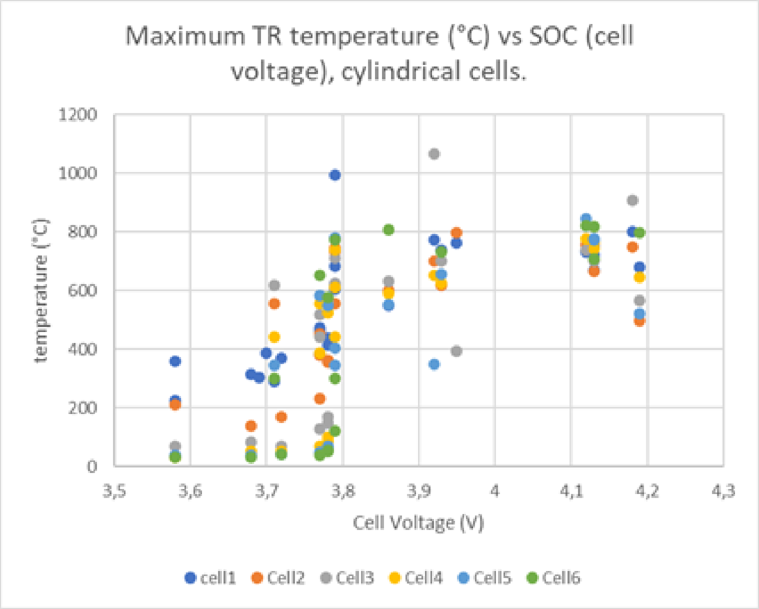
Results of 3 labs at 50% SOC



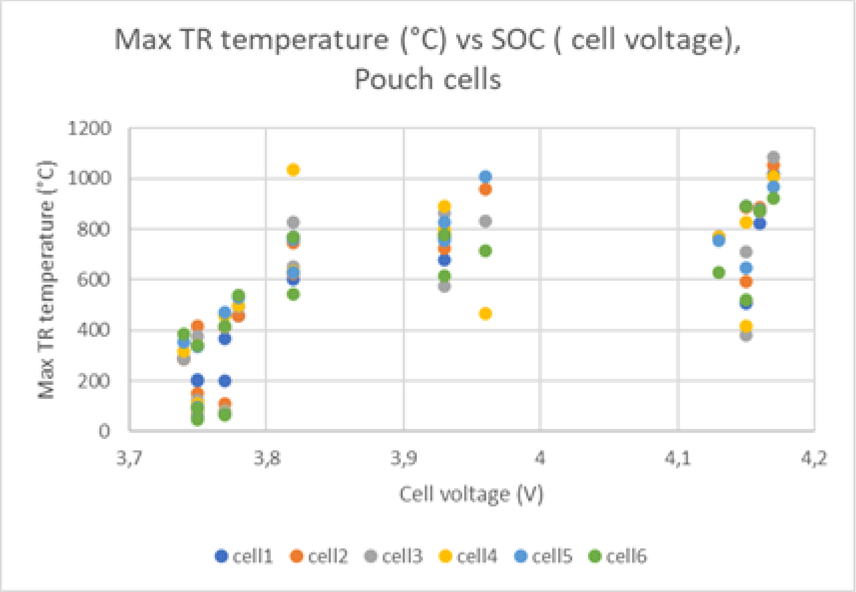
Phase 2 results analysis; effect of SOC on max temp, all results

- Dispersion increases at the point of transition for propagation (30-50% SOC). In case of propagation , the max temp is not much reduced between 100% and 50% SOC.

Cylindrical



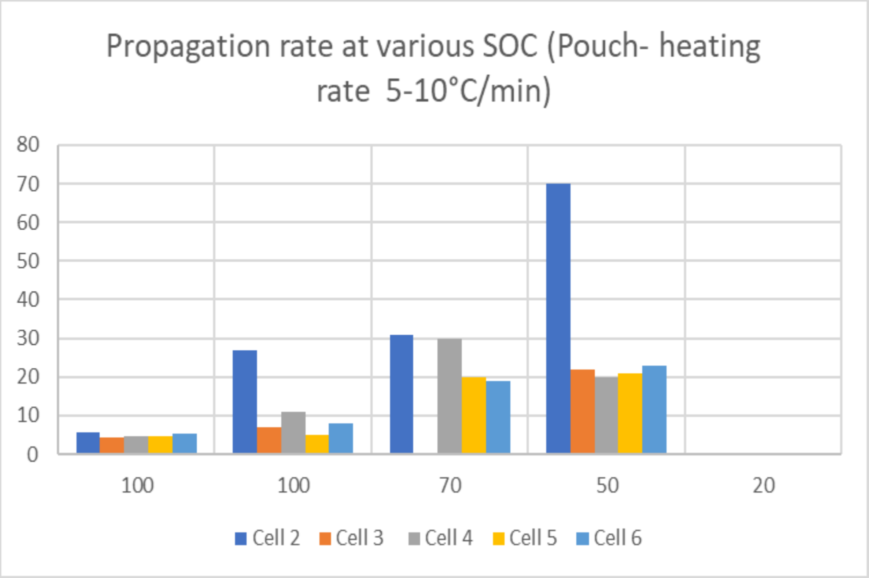
Pouch

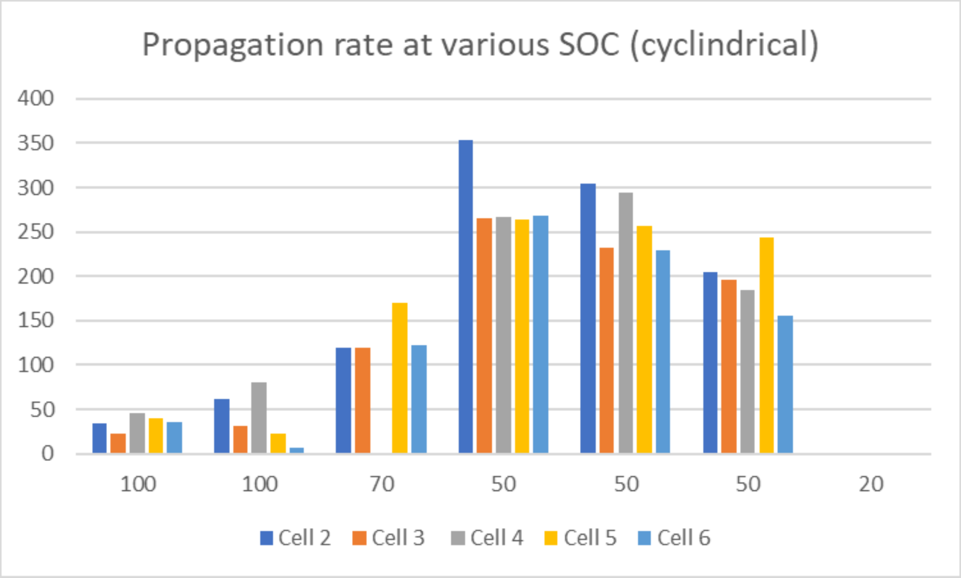


The cell voltage indicates the SOC (20% to 100% corresponds to 3,6V to 4,15V)

Phase 2 results analysis; effect of SOC on propagation time

- Contrary to max temperature, propagation time is changing when measured in a same laboratory.

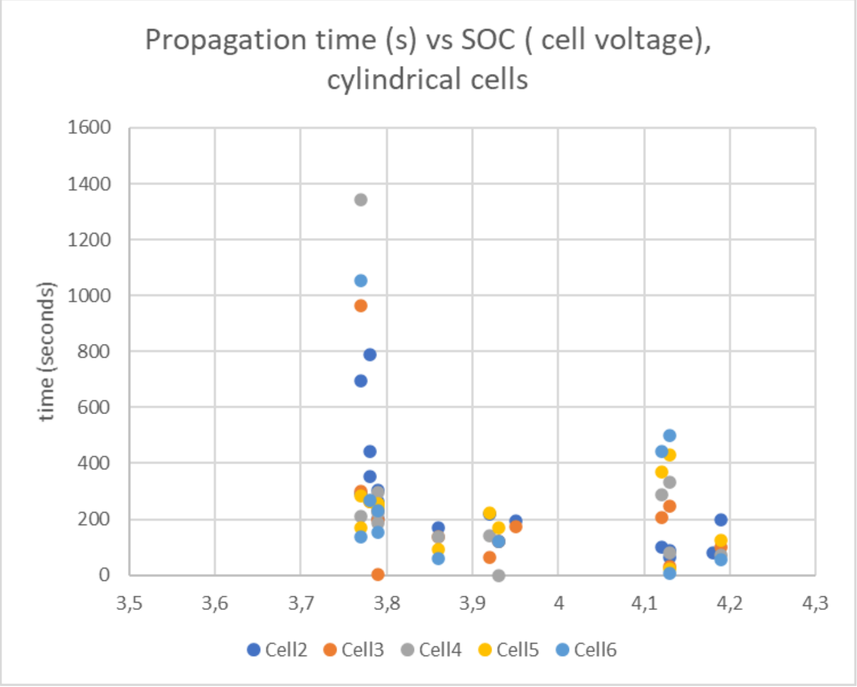


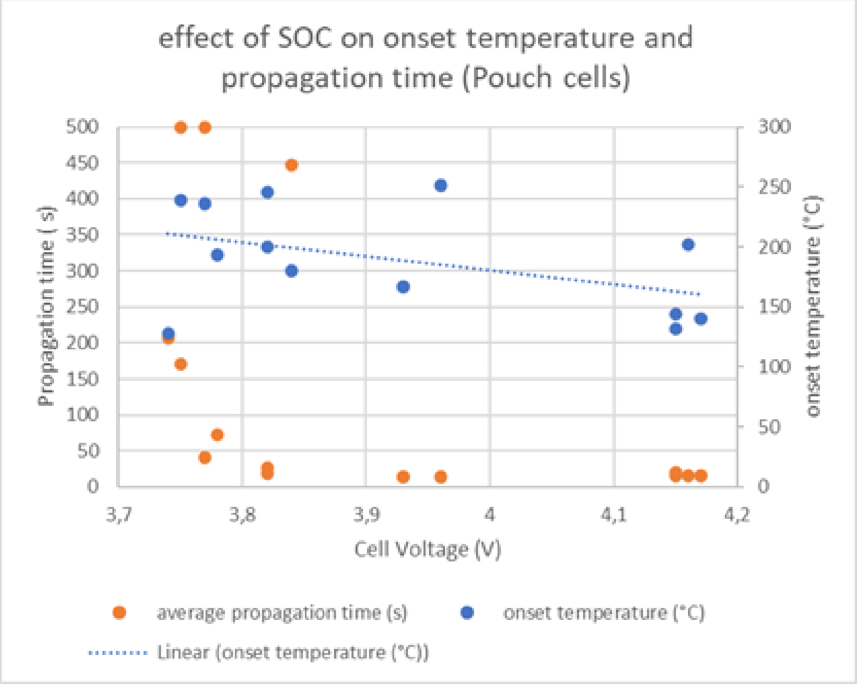


First 100% soc is average of phase 1. The three at 50% SOC are 3 different labs

Phase 2 results analysis; effect of SOC on propagation time and onset temperature, all results

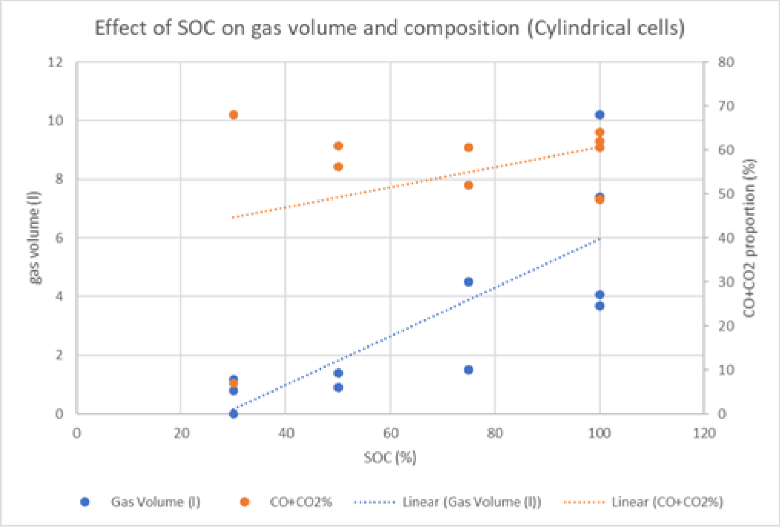
Due to the lack of repeatability, the effect is less clear for all labs

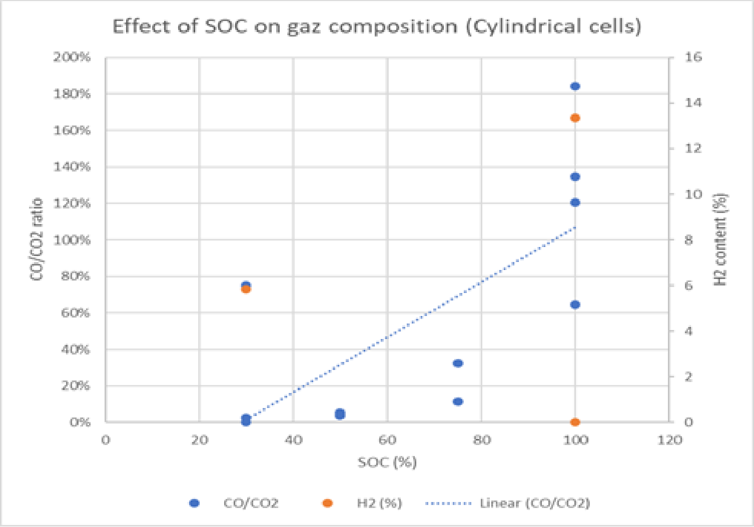




Phase 2 results analysis: gaz analysis under Nitrogen atmosphere.

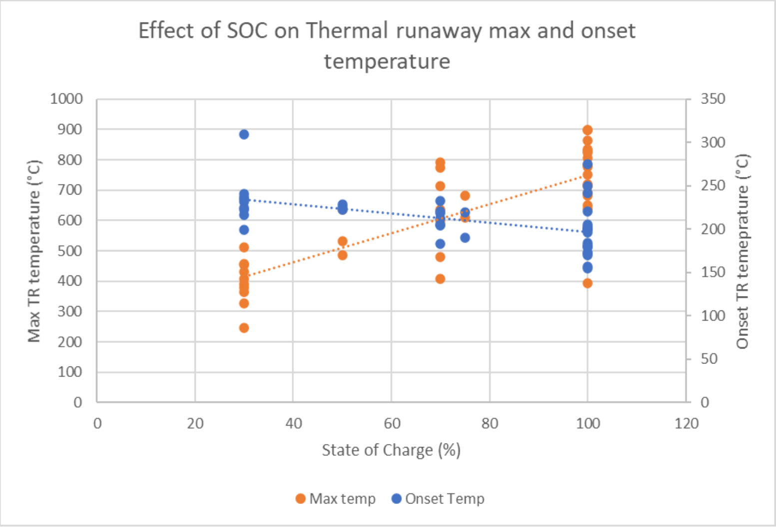
At higher state of charge, the volume of gaz generated is higher, and the gaz composition is less oxidized (hydrogen content is higher and ratio CO/CO2 is higher). As a result, it can be concluded that at higher SOC, the gaz hazard due to flammability or explosion properties will increase

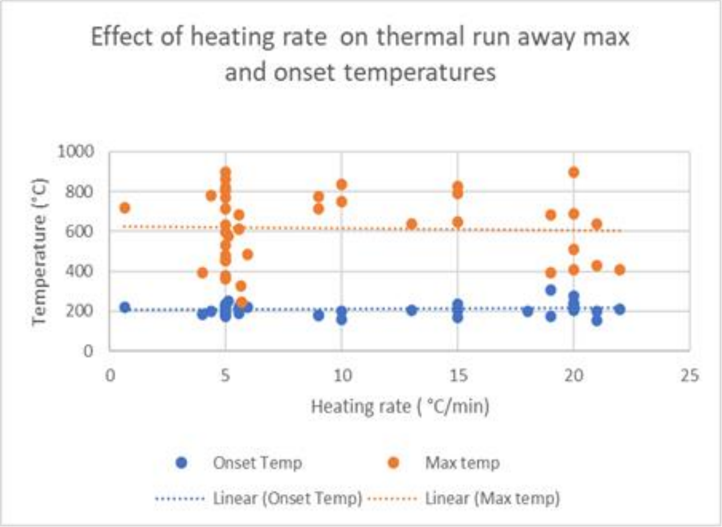




Phase 2 results analysis: gaz analysis under air atmosphere, effect of SOC.

the same results were observed as in propagation test: no significant effect of the heating rate on the TR maximum and onset temperatures, but a significant effect of the high SOC, reducing the onset temperature and increasing the TR maximum temperature





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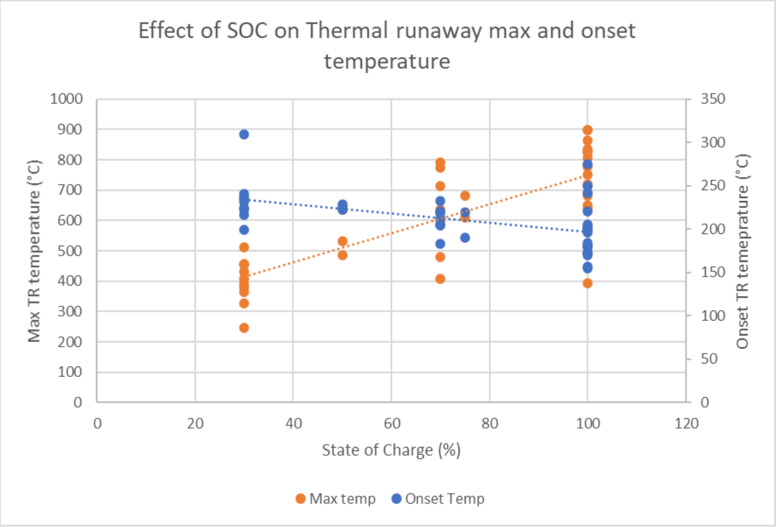
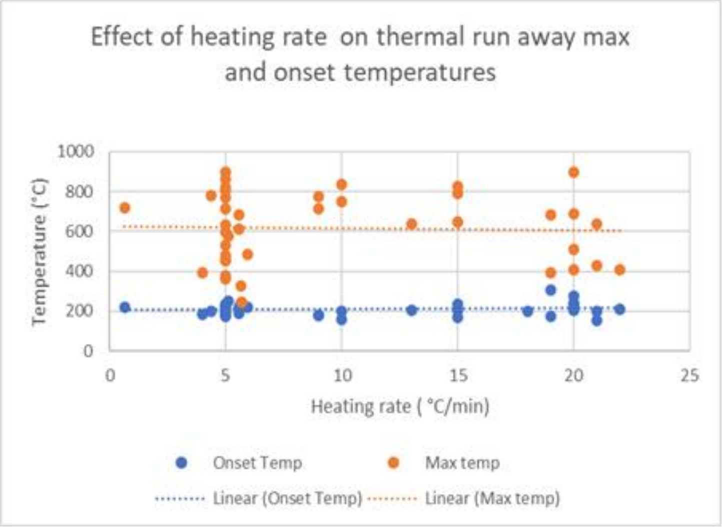
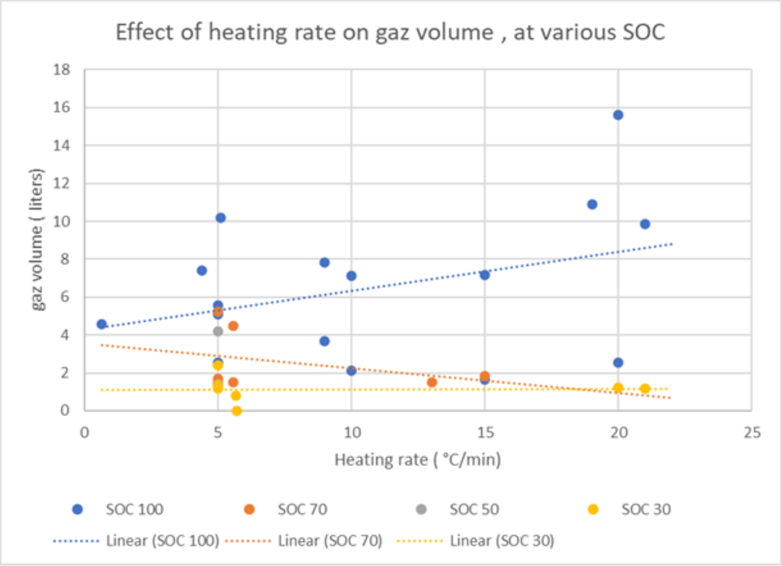




Phase 2 results analysis: gaz analysis under air atmosphere, of heating rate

The quantity of gaz emitted is higher at higher SOC. Nevertheless, the emitted gaz volume is sensitive to the heating rate only at higher SOC, with a factor almost 2 between 5 and 20°C/min



Other relevant data

See

- CATL presentation about large cells testing (Oct 2019)
- LG data about vrious cells testing (Dec 2020)
- FAA presentation about gaz production (ref?)

Preliminary conclusions, for discussion

About the lithium batteries hazards characterization

- The test have been affected by the lack of reproducibility due to variations in the tests methods applied by various labs, but tests demonstrate some level of repeatability of the Thermal runaway hazards: max temperature, onset temperature, gaz volumes, gaz composition.
- Some key parameters influencing the hazards per cell, and their propagation, are tested: cell format, cell SOC, heating rate.
- The analysis of specific toxicity (i.e. HF and HCNcontent), has shown lack of repeatability (within lab) and reproducibility between labs due to various analytical methods. Only a typical range of composition can be assessed.

Preliminary conclusions, for discussion

About the test method:

-the heater method provides a reproducible way to initiate a thermal runaway. Limit of conditions would need to be made more specific to increase repeatability: heating rates, heater types, compression between cells.

-the TR obtained by the heater is close to the one obtained by self-propagation, although the propagation time is very different, demonstrating some robustness of the method.

-A larger dispersion in results is observed when the TR energy is closer to the boundary of non-propagation. The results also indicates that the heating method induces a TR for cells having not enough reaction energy to propagate. In this case, the result obtained is more severe than the expected result of an internal short circuit

Preliminary conclusions, for discussion

About the reproducib:

There are much more difference between labs than within a single lab.

The test decription will have to be more specific and precise:

Identified effect of :

- absence of lid on the test chamber
- various efforts for cells compression during test
- (Partially) heating rate range
- Method for identification (max temperature, onset temperature,..)
- Others to be discussed…

Next steps

-Complete the tests table and analysis (anonymized labs names): done

-Circulate to all labs for review

-pending issues for the December Meeting

- hazards characterization
- method description
- labs comparison

-Others?