

# BATTERY CHARACTERIZATION



## THERMAL ANALYSIS & CALORIMETRY SOLUTIONS

- THERMAL SAFETY •
- THERMAL STABILITY •
- HEAT CAPACITY •
- CHARGE/DISCHARGE HEAT •
- SELF-DISCHARGE HEAT •

## YOUR CHALLENGES

Batteries are essential power sources for every application from your new smartphone, to the development of electric vehicles, Internet of Things (IoT) and Industrial Internet of Things (IIoT).

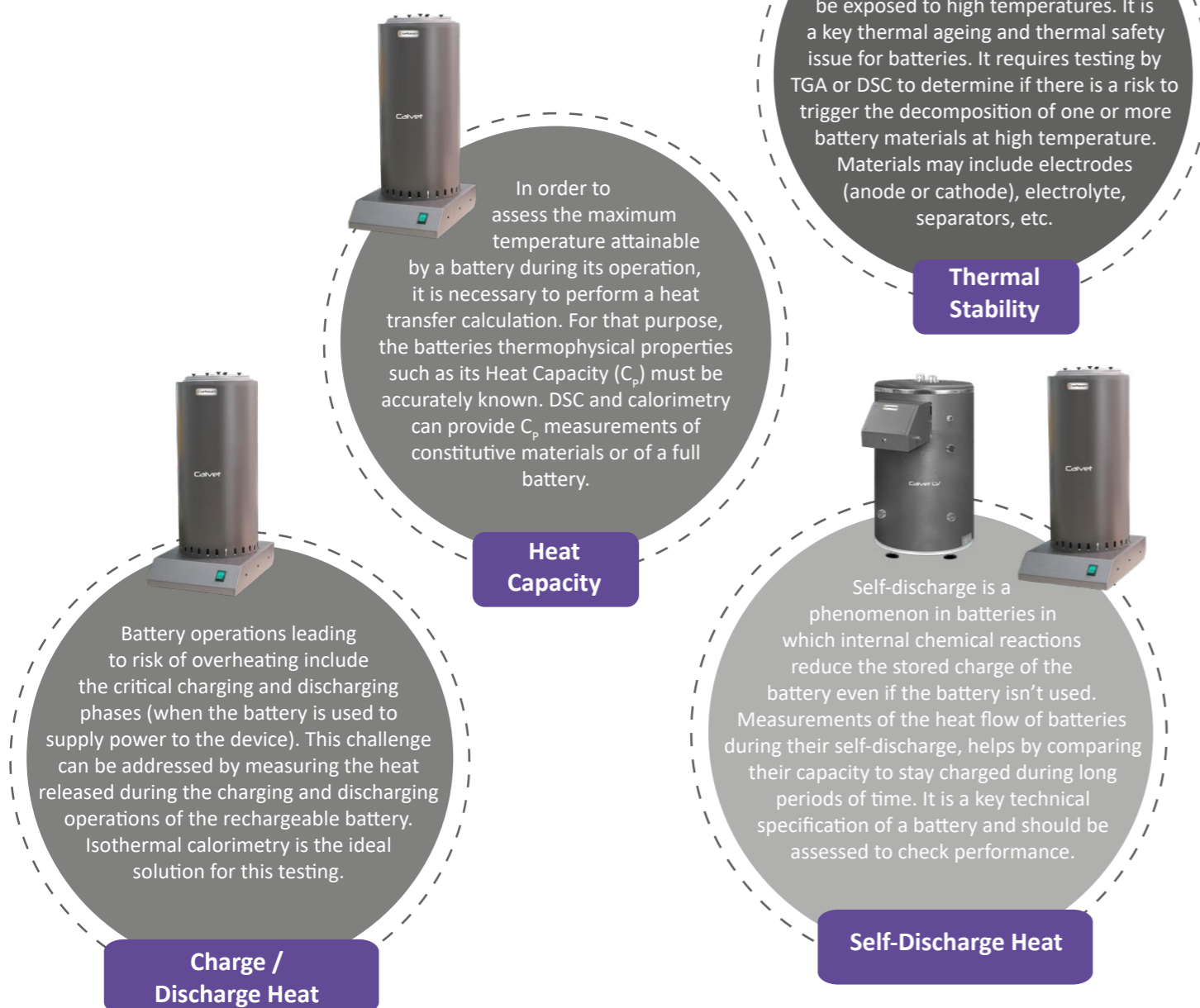
As the development of these new technologies is linked with the autonomy of devices, one key element which makes a great difference is their battery. The demand for new, more powerful, higher energy density and faster charging batteries is growing quickly. In addition, so are the requirements for new materials and thermal safety testing.

Thermal analysis and calorimetry, including traditional TGA and DSC as well as isothermal calorimetry techniques, provide critical insights into batteries and battery components.

KEP Technologies understands your challenges and offers a choice of solutions that provide experimental control, instrument versatility and quality results.

## COMMON BATTERIES - STUDIES & SOLUTIONS

This brochure presents some of our solutions in this field and we encourage you to contact us for more information.



## CUSTOMER TESTIMONIAL

"We chose the C80 due to its multi-modes and availability to use large number of specialized cells for a wide range of applications. There is the possibility of direct heat capacity measurements of solid materials in a wide temperature range. Also C80 has a possibility to use gas circulating cells (to work in controlled atmospheres). It will be used for measurements of specific heat capacity, phase transition temperatures and heat effects in lithium nickel dioxide material."

**Mr. Oleg Bolotov - Scientific-research electrodynamics laboratory, LLC "Proton-21"**

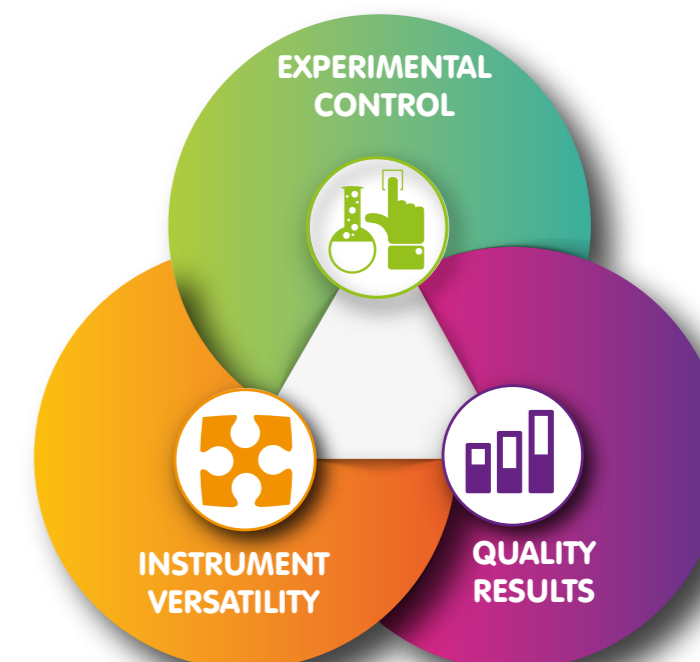
## THE KEP TECHNOLOGIES ADVANTAGE

KEP Technologies is addressing its offerings to the battery market by making available the widest and most versatile choice of solutions. Now you can consult with one company, KEP Technologies, to address your challenges across the broadest number of battery studies on the market.

Each solution embodies our "Reimagine Material Characterization" value proposition by delivering the three core customer benefits of **Experimental Control, Instrument Versatility and Quality Results.**

We believe solutions that provide these benefits will deliver the highest value to our customers.

In addition to our core customer benefits, we are able to provide **customized solutions** by harnessing the engineering and project management of our highly skilled organization.



## CUSTOMIZED SOLUTIONS

Modular design allows for upgraded and tailored functionality  
Access to all previous non-proprietary custom requests  
Open access to our engineering development team

## INSTRUMENT

### THEMYS ONE



- **HIGH SENSITIVITY BALANCE FOR THE DETECTION OF SMALL MASS VARIATIONS** specifically designed for TGA analysis.
- **CONVENIENCE OF ONE FURNACE** to reach temperatures as high as 1600°C.
- **PLUG AND PLAY INTERCHANGEABLE RODS** to perform TGA only, TG-DSC, TG-DTA, and 3D high sensitivity/Cp measurements.
- **EXTERNAL COUPLING CAPABILITY** including evolved gas analyzers



## SPECIFICATIONS

Temperature range (°C)	room temperature to 1600
Isothermal and temperature scanning (°C/min)	0.01 to 100
Sample volume (ml)	up to 1 in TGA
Evolved gas analyzers (FTIR, MS, GCMS, MS-FTIR, or FTIR-GCMS) for performing qualitative and quantitative gas characterization	

For more information on specifications please consult the product information and brochures available on our website : [www.setaramsolutions.com](http://www.setaramsolutions.com)

## APPLICATION

### THERMAL STABILITY OF ANODE MATERIALS COMPARED BY TGA

#### INTRODUCTION

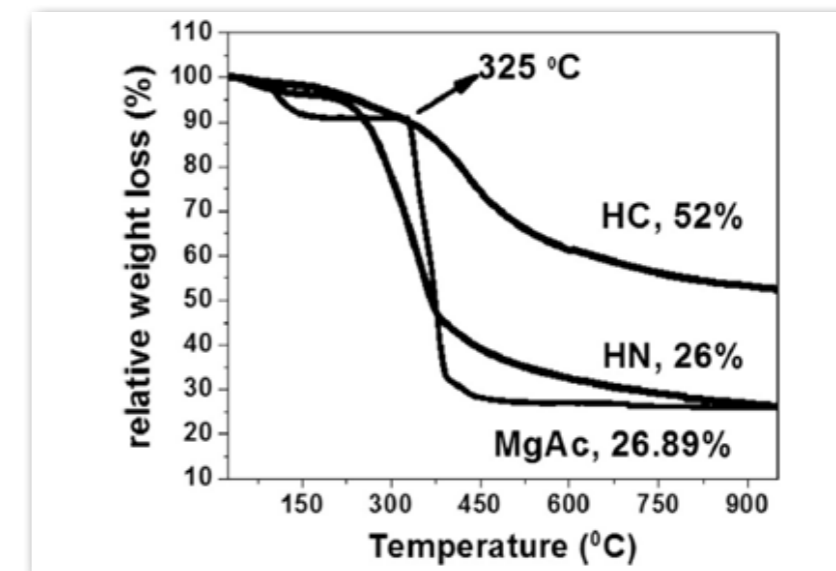
In the field of battery materials characterization, TGA is primarily used to determine the thermal stability of materials used to make cathodes, anodes, electrolytes or separators. TGA will measure the temperature at which a mass loss is detected, which corresponds to the temperature of decomposition of the material. For instance, different cathode materials tested using the same conditions can thus be compared in terms of their stability against thermal decomposition.

#### EXPERIMENT

- Instrument: LABSYS evo TGA 1600
- Sample : Anode materials  
Nanoporous Carbons from hydrothermally treated biomass (Hazelnut shell, HN : Powderer, HC : Hydrochar)
- Method: Heating under N<sub>2</sub> to 950 °C at 10°C/min.

#### RESULTS AND CONCLUSION

HN: starts to decompose at 200° C and final mass 26% at 950°C.  
 HC: more stable as major decomposition starts at 330°C and final mass 52%.  
 Other heat-treated or KOH-activated samples further improved the stability.



Ece Unur et al, *Microporous and Mesoporous Materials* 174 (2013) 25–33

# THERMAL STABILITY / HEAT CAPACITY CHARGE-DISCHARGE HEAT / SELF-DISCHARGE HEAT

## INSTRUMENT

### CALVET



**ISOTHERMAL OR TEMPERATURE SCANNING MODES** for increased flexibility

**HEAT MEASUREMENT ACCURACY** with Calvet 3D sensor capturing 93-95% of all heat forms. The highest level on the market

**CONVENIENT, INTERCHANGEABLE CELLS** to perform even the most demanding experiments using one instrument :

- Heat capacity and Self-discharge with standard cells
- Thermal stability with pressure measurement cells
- Charge / discharge with cells allowing the use of electrical wiring and connections to battery cycler

**WIDE TEMPERATURE RANGE** with low temperature version CALVET CRYO and high temperature version CALVET HT

## SPECIFICATIONS

	CALVET	CALVET CRYO	CALVET HT
Temperature range (°C)	Ambient to 300	-196 to 200	Ambient to 600
Temperature accuracy (°C)	+/-0.3 *	+/-0.5 **	+/-1*
Temperature precision (°C)	+/-0.15*	+/-0.25**	+/-0.5*
Programmable temperature scanning rate	0.001 to 2°C/min	0.01 to 1°C/min	0.01 to 2°C/min
Enthalpy accuracy	+/-0.4 *	+/-0.2 **	+/-1*
Calorimetric precision (%)	+/-0.4*	+/-0.5**	+/-1.5*
Cells (ml)	Up to 12.5 (standard cell)	Up to 12.5 (standard cell)	Up to 7
Battery sizes (mm)	Up to 15 diameter, 70 height. C80-22 (on request) up to 20	Up to 15 diameter, 70 height	
Pressure measured and controlled (bar [psi])	350 [5,075]; 600 [8,700]; 1000 [14,600]	100 [1,450]; 600 [8,700]; 1000 [14,600]	100 [1,450]; 300 [4,350]; 400 [5,800]

\* Based on indium melting tests \*\* Based on naphthalene melting tests

For more information on specifications please consult the product information and brochures available on our website : [www.setaramsolutions.com](http://www.setaramsolutions.com)

## APPLICATION

### THERMAL STABILITY OF AN ELECTROLYTE UNDER TEMPERATURE SCANNING CONDITIONS

## INTRODUCTION

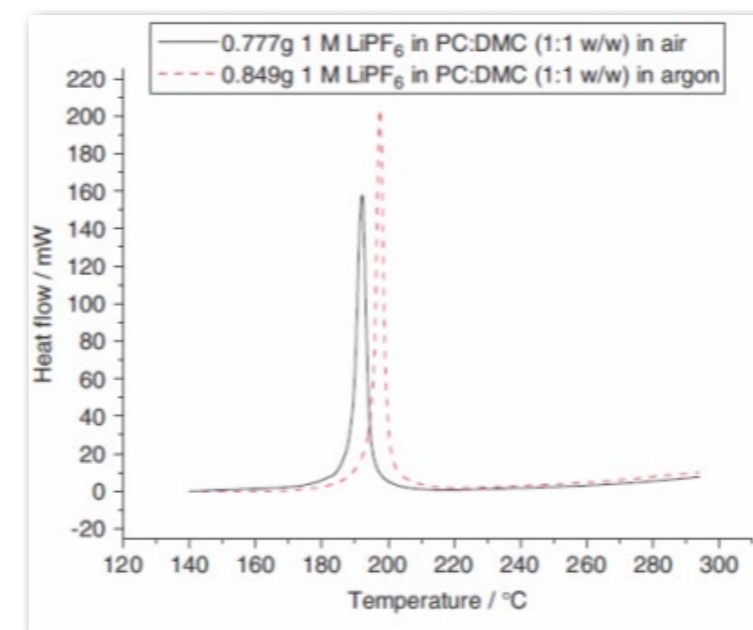
When the temperature of a Li-ion battery increases because of abusive conditions (e.g., short circuit, overcharge, heating) self-heating may be initiated. Various exothermic and endothermic reactions involving both the solution and the electrodes can occur inside the battery. The CALVET calorimeter is used with high pressure stainless steel vessels to study the thermal stability of several commonly used organic solvents and electrolytes and to investigate the kinetic process of the related reactions.

## EXPERIMENT

- Instrument: CALVET.
- Vessel : High pressure stainless steel.
- Sample : Electrolyte LiPF<sub>6</sub> in PC:DMC. Ethylene carbonate (EC), propylene carbonate (PC), dimethyl carbonate (DMC) and diethyl carbonate (DEC) are the most widely used solvents and LiPF<sub>6</sub> is the dominant solute used in practical lithium ion batteries.
- Method: Heating at 0.2°C/min to 300 °C in air or argon.

## RESULTS AND CONCLUSION

A strong exothermic reaction starting above 160°C is seen. That reaction is shifted to higher temperatures under argon, meaning an improved thermal stability.



from Qingsong Wang and coll., Journal of Loss Prevention in the Process Industries - 19 (2006) 561-569

## MEASUREMENT OF THE HEAT CAPACITY OF A FULL BATTERY

## INTRODUCTION

Heat capacity measurements of a battery, together with additional measurements of heat source factors and heat transfer coefficients using other techniques, makes the calculation of the battery's temperature rise possible as well as to compare calculations with measured values.

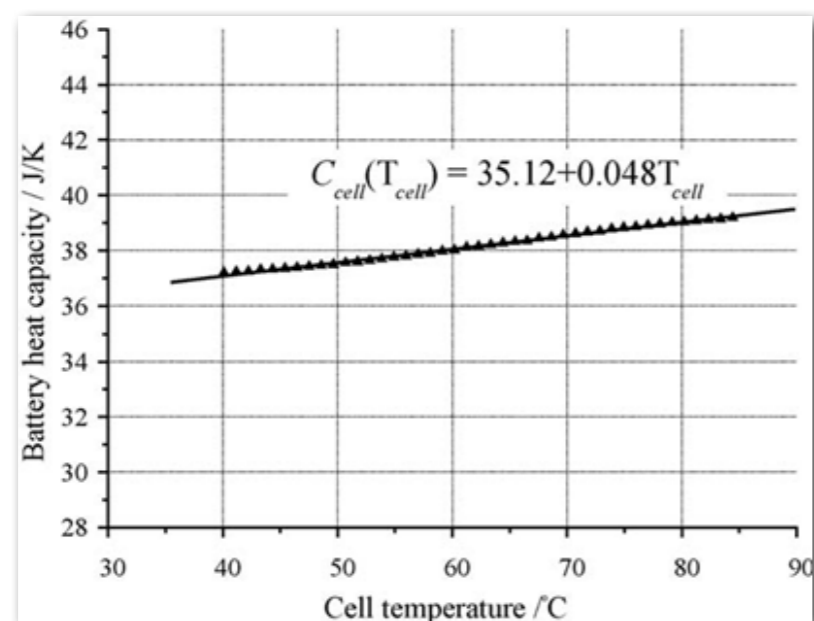
## EXPERIMENT

- Instrument: CALVET.
- Vessel : Standard stainless steel vessel.
- Sample : A commercially available, cylindrical lithium-ion battery.
- Method: A temperature ramp from 20 to 90°C at 0.4°C/min.

## RESULTS AND CONCLUSION

The heat capacity of the cell can be approximated by a linear function of temperature  $C_{cell} (J/°C) = 35.12 + 0.048T (°C)$ .

Thanks to the CALVET measurements and the application of this method, the authors were able to plot the battery's heat capacity against temperature over the tested range and observe that the variation was almost linear.



Kazuo Onda et al, *Journal of Power Sources* 158 (2006) 535–542

## MEASUREMENT OF THE HEAT PRODUCED BY A BATTERY WHEN IT IS CHARGING/DISCHARGING

## INTRODUCTION

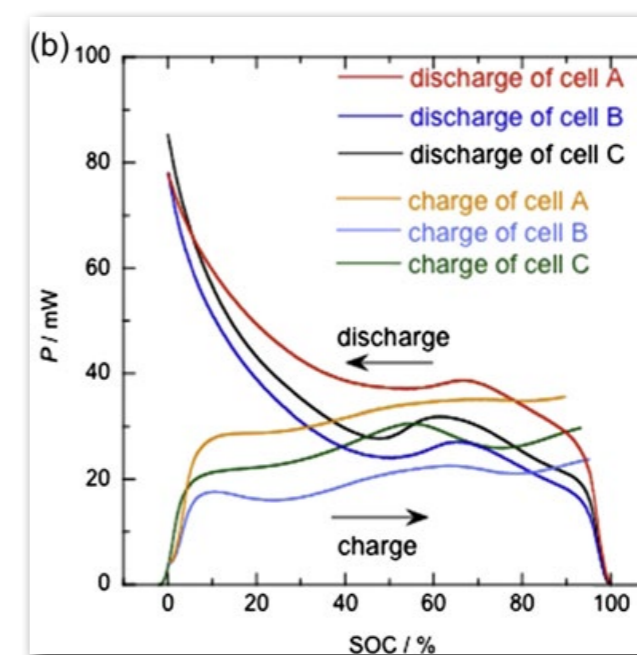
Isothermal calorimetry can measure the heat produced when a rechargeable battery is charged and discharged. The idea here is to assess the risks of damage to the battery if overheating occurs. The battery is connected using thin wires to an electrical cycling system before being placed in the sample holder.

## EXPERIMENT

- Instrument: CALVET.
- Vessel : Customized vessel
- Sample: Cylindrical Li-ion batteries, State of Charge ranging between 50 and 90%. Stored between 436 and 652 days. Storage temperature ranging between 20 and 50°C.
- Method: Isothermal testing at 25°C. The battery is fitted in the calorimeter, connected to an external battery charging/discharging system through lead wires (<1% heat loss) up to high current (450 mA).

## RESULTS AND CONCLUSION

At a high rate of charging and discharging (on the chart: 450 mA), the cell stored at 50°C (cell A) shows larger heat generation probably because of leakage of the electrolyte solution. To limit the temperature increase and thus preserve safety, improving the sealing would be an effective measure.



Saito et al, *Journal of Power Sources* 244 (2013) 294-299

## SELF DISCHARGE OF LITHIUM BATTERIES

## INTRODUCTION

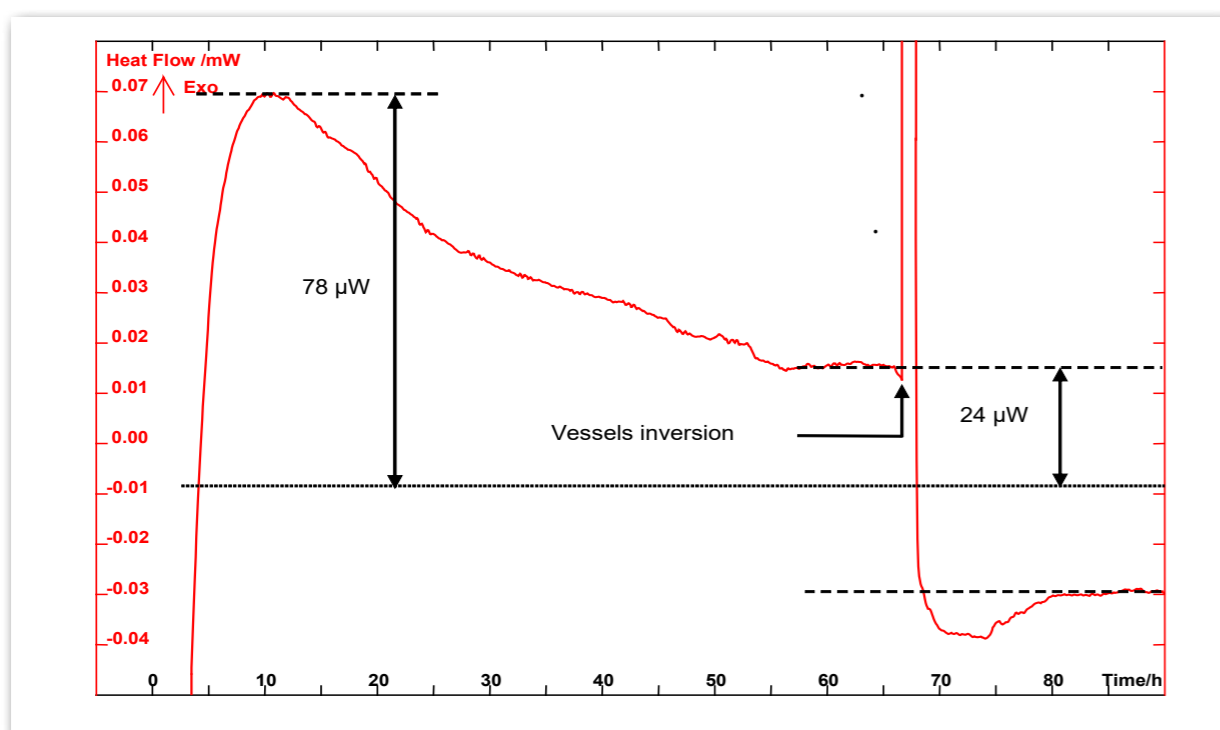
Self-discharge is a phenomenon in batteries in which internal chemical reactions reduce the stored charge of a battery even if the battery isn't used. Measurement of the heat flow of a battery during its self-discharge thus helps compare its capacity to stay charged during long periods of time versus other batteries. The method of measurement of self-discharge heat using an isothermal calorimeter consists of placing a sample holder containing a battery in the reference side of the calorimeter where the self-discharge heat is produced. The two sample holders are swapped and the difference of calorimetric signal level before and after swapping is used to determine the self-discharge heat.

## EXPERIMENT

- Instrument: CALVET.
  - Vessel : Standard vessel stainless steel.
  - Sample : Batteries of type CR1220 are studied.
- The temperature of the calorimeter was maintained at 70°C.
- Method : 9 batteries were superimposed inside the vessel. Between adjacent batteries a layer of paper is inserted to electrically insulate the batteries from each other.

## RESULTS AND CONCLUSION

- After the introduction, the calorimetric output passes by a maximum of 78  $\mu\text{W}$ . (or  $78/9 = 8.7 \mu\text{W}$  per battery).
- After 65 hours the two vessels : measure and reference are inverted. The mid-point between the trace obtained before inversion and the trace after 80 hours gives the position of "calorimetric zero".
- It means that after 55 hours the 9 batteries dissipate 24  $\mu\text{W}$  which corresponds to  $24/9 = 2.7 \mu\text{W}$  for each battery.



## INSTRUMENT

### CALVET LV



- HIGHEST HEAT MEASUREMENT ACCURACY**  
 with Calvet 3D sensors capturing 93-95% of all heat forms, the highest level on the market
- MODIFIABLE TEMPERATURE CONDITIONS**  
 for increased flexibility and replications of real life conditions
- CONVENIENT, INTERCHANGEABLE CELLS**  
 to perform varied experiments in one instrument:
  - Self-discharge with standard cells
  - Charge/discharge with cells allowing the use of electrical wiring and connections to battery cyclers
  - Single or twin measurements
- LARGE CELL VOLUME**  
 to test battery sizes up to 33mm diameter and 100mm height, including the standard 18650, 21700 or D-type batteries

## SPECIFICATIONS

	CALVET LV 17mm	CALVET LV 35mm
Temperature range (°C)	ambient to 200	
Temperature accuracy (°C)	+/-0.4 *	
Isothermal and temperature scanning rate (°C/hour)	< 2 between two isotherms	
Enthalpy accuracy	+/-0.2 *	
Cells (ml)	Up to 12.5	Up to 100
Battery sizes (mm)	Up to 15 diameter, 70 height	Up to 33 diameter, 100 height

\* Based on indium melting tests

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## APPLICATION

### SELF DISCHARGE OF BATTERIES

#### INTRODUCTION

Self-discharge is a phenomenon in batteries in which internal chemical reactions reduce the stored charge of a battery even if the battery isn't used. Measurement of the heat flow of a battery during its self-discharge thus helps compare its capacity to stay charged during long periods of time versus other batteries. The method of measurement of self-discharge heat using an isothermal calorimeter consists of placing a sample holder containing a battery in the reference side of the calorimeter where the self-discharge heat is produced. The two sample holders are swapped and the difference of calorimetric signal level before and after swapping is used to determine the self-discharge heat.

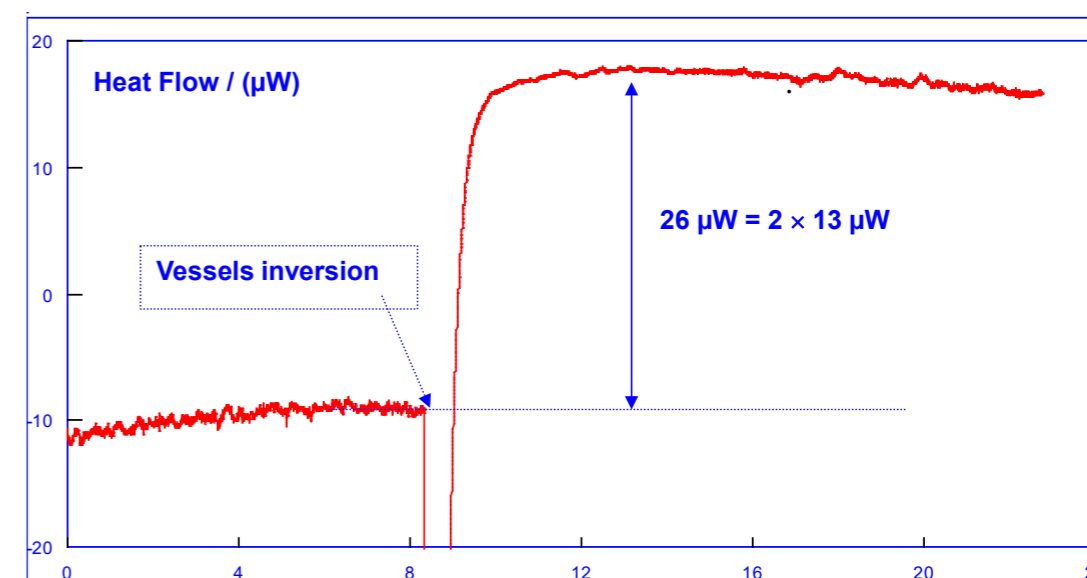
#### EXPERIMENT

- Instrument: CALVET LV.
  - Vessel: Standard vessel in stainless steel.
  - Sample : 6 watch batteries of Li-I type.
- Available space for the sample inside the vessel :
- diameter : 32.7 mm
  - height : 111.2 mm
  - volume : 93.3 ml
- Atmosphere : air.
- Method : Isotherm at 27.4°C for 24 hours.

#### RESULTS AND CONCLUSION

During the first 8 hours, the CALVET LV monitors the heat flow dissipated by the batteries. After this time, the two vessels (measure and reference) are inverted. After another 4 hours the heat flow is stable again.

- The deviation of heat flow before, and after the inversion ( $26 \mu\text{W}$ ) is twice as high as the heat flow dissipated by the 6 batteries :  $13 \mu\text{W}$  ( $= 26 \mu\text{W} / 2$ ).
- Each battery dissipates an average heat flow of  $2.2 \mu\text{W}$  ( $= 13 \mu\text{W} / 6$ ).





Switzerland – France – China – United States – India – Hong Kong  
For contact details: [www.setaramsolutions.com](http://www.setaramsolutions.com) or [setaram@kep-technologies.com](mailto:setaram@kep-technologies.com)

