

ARC[®]

The World Benchmark
Battery Testing Calorimeter Systems

Accelerating
Rate
Calorimeter
The World Benchmark

thermal hazard technology

Offices in ENGLAND, USA, CHINA, INDIA
Representation Worldwide

The ARC[®] Battery Systems

Battery Safety, Efficiency, Lifecycle, Performance,

Lithium Batteries & Heat...

Lithium batteries are recognised as hazardous - it is important to determine both the effect of heat on lithium batteries and the heat that results from their use and abuse.

ARC Calorimetry will give vital thermal data to areas of battery development, battery safety, battery performance efficiency and lifecycle.



Battery Safety and Battery Performance

The Accelerating Rate Calorimeter was devised by the Dow Chemical Company in the 1970's and was commercialised in 1980. This technology was developed to simulate exothermic runaway reactions from hazardous and reactive chemicals safely in the laboratory. For such a simulation an Adiabatic Calorimeter is appropriate and ARC technology embodies the best adiabatic control. In operation, the calorimeter temperature is controlled to always track or follow the sample temperature. Therefore as a sample self-heats and its temperature rises, so does the calorimeter temperature. Worst Case evaluation is made and a Real Life Scenario simulated. Pressure can also be measured and calorimeters can take very large batteries. In addition, the ARC will operate in isothermal mode with exceptional sensitivity and stability. The unique dynamic range allows for detection and measurement of very small heat release as well as the ability to quantify runaway explosive decompositions.

Such requirements are important for battery work. Uniquely the ARC has robustness and ruggedness to withstand damage should an explosive reaction occur and thus THT systems are designed to be safe in such circumstances.

Unrivalled Specification



® 'ARC' is a registered Trade Name
of Thermal Hazard Technology

The other key point necessary for many battery applications is the size of the calorimeter. Five options are available; of those cylindrical in their internal shape, the smallest measures 10cm in diameter by 10cm in depth and is suitable for testing battery components in metal holders, coin cells, small prismatic, 18650 and other smaller 'domestic' batteries. The largest 65cm by 50cm will take large battery modules and packs used, for example, in applications from power tools to satellite and automotive applications.

In use to detect heat release the system does not scan in temperature. Successive small heat steps are applied and after a wait period for isothermal equilibration, there is a seek period to detect heat release by temperature rise. When this occurs the system automatically switches to the Exotherm mode and tracks the heat release, by accurately following the temperature rise, storing Time-Temperature-Pressure data.

Such studies are routine for groups studying battery components in order to develop chemistry that optimise specific power requirements and increase their inherent safety. But ARC technology can allow much more to be achieved. Batteries like reactive chemicals or explosives will also release heat – they will react and decompose when heated, internal pressure may cause them to rupture and disintegrate. Accompanying this is typically smoke and fire as

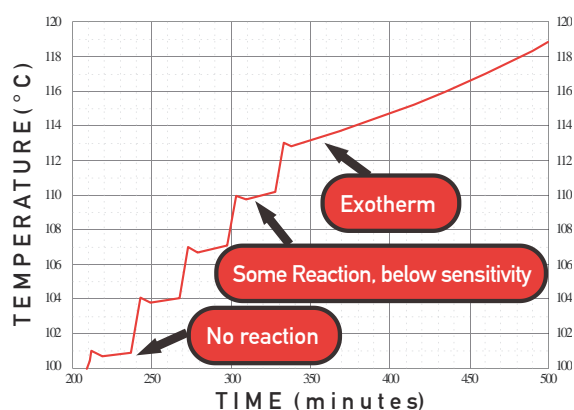
severe oxidation reactions occur between battery components and oxygen in the air. Because of the volume of the calorimeter it is simple to connect the battery to leads, allowing in-situ electrical measurement. Also with large batteries it is possible to apply multiple thermocouples to allow temperature distribution measurement over the battery surface. Connecting the battery to an appropriate cycler or battery test system allows vital temperature and pressure data to be obtained under conditions of charge, discharge (including of course the very fast discharge needed for automotive applications). Also successive cycling and abuse testing such as short circuit, overvoltage and nail penetration and crush (internal short circuit) can be performed within the ARC calorimeter. The ARC is therefore an ideal tool to evaluate both performance and safety aspects of lithium batteries, not forgetting its initial role in evaluating new battery chemistries. 30 years after the technology was first available the ARC remains the world benchmark calorimeter for scientists and engineers focusing in the area of chemical hazards. Today the latest generation ϵ sARC with large volume calorimeters is the No.1 choice for those researching battery safety and development of safer batteries and to evaluate the performance, efficiency and life-cycle of those batteries.

The esARC



For safety studies and accurate detection the Heat-Wait-Seek protocol of the ARC and automatic detection of exothermic reaction is shown below. For full details of the esARC, its mode of operation data and analysis, please request the 28 page ARC brochure or visit www.thtuk.com, www.thtusa.com or www.thtchina.com.

Heat-Wait-Seek Protocol to Detect Self-Heating



ARC key aspects are:

- Excellent Adiabatic Control to 0.01°C
- Ultimate Sensitivity to 0.002°C/min
- Measurement of Pressure and Temperature
- Choice of Calorimeter Size to 65cm by 50cm
- Resultant Gas Collection facility

Ease of Use

- Simple Hardware Configuration
- Rapid Set Up; 10 Minutes
- Intuitive Labview™ Software
- 1-3m³ Working Volume

Versatile and Flexible Operation

- Any Chemical / Battery Type
- Many Battery Holders
- Quantifies Exotherms and Endotherms
- Isothermal and Isoperibolic Modes
- Air: Inert Gas Atmosphere, Vacuum

Multiple Built-In Safety Features

- Rugged Robust Construction
- Explosion Proof Containment Vessel
- 3mm Reinforced Steel
- Proximity Switch Shut Down
- Software Shut Down Facilities
- Automated Fume Extraction
- Fireproof and Explosion Containment

Calorimeter Choice



Batteries come in all shapes and sizes. Often it is necessary to test both small and large batteries.

Standard esARC

The standard ARC Calorimeter has an internal size of 10cm diameter by 10cm depth. This restricts use to battery components and smaller batteries, from Coin Cells to AA and 18650 to 26650, prismatic and smaller lithium polymer batteries.

EV

To facilitate safety testing of large batteries, EV batteries and modules, THT developed the large volume calorimeter, the EVARC. The internal size of this EV calorimeter is 25cm diameter and 50cm depth. Using all electronics and software of the ESARC, the EVARC can be operated with either the EV calorimeter or the standard calorimeter allowing full functionality of both instruments.

EV+

The EV+ calorimeter has larger volume and is designed for safety and performance testing of EV cells and small modules.

It has been designed to fulfil requirements of SAND 2005-3123, SAE J2464. USABC / FreedomCAR, UN & UL tests.

The EV+ calorimeter is a low-pressure tight sealed unit. The lid to base seal is maintained by electromagnets. It is designed to vent with a modest over pressure, but this is unlikely as gas generation is led to collection facilities.

There is in built capability for gas collection, video monitoring, battery clamping, multifunction and cryogenic operation.

BPC

For battery performance studies and research of large format cells appropriate in the automotive and power tools industries, THT has developed the battery performance calorimeter (BPC).

The BPC is also likely to be the calorimeter of choice in areas of Stationary Applications; for storage and 'peak shaving'. The BPC is 65cm diameter by 50cm depth and is housed in the EV containment vessel ensuring maximum safety in operation. The BPC uses the same electronics hardware and software as other THT ARC systems and can be acquired packaged together with the other calorimeters – or as a multi calorimetry system.



A THT Battery Test system; the esARC, EVARC Double System is shown

Battery Specific ARC Options

A range of options and kits are available to allow testing for Battery Chemicals to Abuse tests and EV battery modules. Many options are suitable for all calorimeters, some are specific to particular calorimeters.

Options can be acquired with the unit or added at any later date.

Battery Materials & Chemical Components Kit

To test effect of heat upon materials, the standard (small size) calorimeter is typically used. Sample size may be 100mg to 5g. The kit contains appropriate sample holders and modified pressure lines to facilitate such studies.



Battery Safety Holders & Canisters

THT have available a full range of battery holders. There are suitable for all shapes and sizes from small coin cells, 1850 to prismatic to large format cells and modules. In addition THT provide pressure certified canisters where cells can be isolated in smaller calorimeters, gas can be isolated and then taken for analysis.



Integrated Battery Cycler - KSU Option

THT, KSU option, is an integrated single channel battery cycler. The voltage and current range are user specified. Fully integrated with appropriate software to give a single turn-key instrument to allow rapid charge/discharge cycling i.e tests under conditions of battery use.

Further to this interfacing of other units to THT ARCs can be requested or the user may implement other available cyclers and testers, loads and charges with stand alone software.



Battery Abuse Tester (BSU)



calorimeters.

The BSU allows abuse tests to be carried out such as over voltage charging and discharging and short circuit testing. The options currently requires manual operation but links with ARC software for data analysis. Such tests can be achieved in the standard and EV

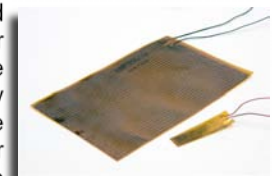
Battery Connection Kits

EV size cells and modules tested with high power discharge require low impedance cables and connections. THT supplies standard and customer specified connection kits to allow appropriate testing.



Heat Capacity Option (CPO)

There is need to measure heat capacity for 'Thermal Management' studies. If the battery overall heat capacity is known the temperature and temperature rate data can be converted to units of heat in Enthalpy (Joules) and heat release rate i.e Power (Watts). The option can be provided as semi or fully automated form. To measure the heat capacity two or more cells are heated with a 'heater mat' by a precise power supply. In the fully automated form the power supply is integrated to the ARC system and the data is analysed with the ARCCal+ ARC software.



Surface Area Heat Measurement Multipoint Option (MPO)



Heat release for batteries is not uniform. Typically heat may conduct through metal components or collector plates and appear at the terminals. For applications such as Thermal Management there is the need to understand the variation of heat release over batter surface.



Surface Area Heat Measurement Multipoint Option (MPO) - *continued*

THT provide both semi and fully automatic options to allow surface area heat measurement. 8, 16 and 24 thermocouple options are available.

The fully automated option integrates both with the ARC hardware and software. The same option is available for all THT battery calorimeters.

Cryogenic Operation



Isothermal, Thermal Management, Multipoint and Heat Capacity testing is often needed over the full range of ambient temperature - this could possibly be to -40°C . To facilitate such tests THT provide a manual cooling option for the EV and EV+. The option using liquid nitrogen allows rapid cooling and short term cryogenic isothermal stability.

Nail Penetration & Crush Option

THT provide Nail Penetration & Crush option for differing requirements; smaller and larger batteries. For the standard and EV calorimeter the option is pneumatic and has capabilities to crush 18650 cells. The interchangeable nail and crush head can be selected and easily replaced. A range of size and shapes are possible.

Manual Nail Penetration and Crush can be carried out simply with all calorimeters but can be hazardous to the operator and gas release to the environment is possible.

THT offer two options; pneumatic and motorised: The pneumatic system has been designed to automate Nail Penetration and Crush with smaller batteries. This option does not have controlled speed but can give high power. When the batteries are small, speed variation gives little difference in result. Small batteries (e.g. 18650) may be very rigid and this pneumatic method will give nail penetration and appropriate crush. This option is available for the standard and EV calorimeters.

The motorised option allows controlled speed nail penetration. This is needed in 'standard test methods' and is valuable for larger batteries or modules when speed variation can give difference in result. The controlled speed unit has been developed for the EV+ calorimeter which is itself designed to carry out testing to standard methods.



Video Monitoring

Video monitoring is standard in the EV+ calorimeter again fulfilling the need in 'standard test methods'. The high resolution camera is air cooled for close proximity filming. The special glass window used in the EV+ calorimeter allows a gas tight seal and easy cleaning.



Gas Collection

Gas collection is achieved in the standard and EV calorimeters by use of sealed canisters. These are placed internally within the calorimeter. The canister allow for cables and thermocouples to pass through via sealed ports. Other ports allow for inerting the atmosphere, pressure measurement and gas collection. The versatility of this approach means that a variety of external collection methods are possible (cylinders or bags). This method eliminates the need for closed battery holders. Closed holders were originally used but explosion of holders with associated hazard is common with this approach.

The EV+ calorimeter is of sealed construction but with a 5cm port integrated for gas collection. As standard the unit is supplied with gas collection bags though alternative configurations can be simply made. The EV+ is designed to facilitate gas measurement and collection with the gas being taken for external analysis and fulfil the requirements of standard test methods



Battery & Battery Components Testing

The application of the ARC to lithium batteries may be categorised into six groups:

- 1 Battery components testing and development of new battery chemistries; a research area where much work has been done within University & Academic environments
- 2 Complete batteries and packs for safety studies; testing and for performance and safety; typically carried out by battery manufacturers and bulk purchasers or battery specifiers
- 3 Battery heat output under normal conditions of use for heat output; cycling for heat release to determine battery efficiency and life-cycle studies; testing carried out in academic or industrial laboratories
- 4 Fast discharge, battery performance studies important for EV, HEV, PHEV, battery packs & modules where the temperature distribution over the battery varies, typically MultiPoint measurement important for power tools and automotive applications
- 5 Battery heat output under abuse conditions; implementing tests where the battery is subjected to misuse and quantifying heat output (shorting & overvoltage); again typically carried out by battery manufacturers and bulk users or specifiers.
- 6 Stationary applications; important in storage and peak shaving. These are typically with very large batteries that are potentially subjected to a full range of tests; and is of value to power generator, and municipalities.

There may be the need to measure pressure and collect resulting gas for analysis; this is possible for all batteries. Also there is ability to measure heat capacity of batteries of any size. This is uniquely available with THT ARC technology.

Battery Components

Evaluation of battery components to study new battery chemistries is key to enhanced battery performance and safety. Also heat capacity of batteries of any size can be measured - this is uniquely available with THT ARC technology.

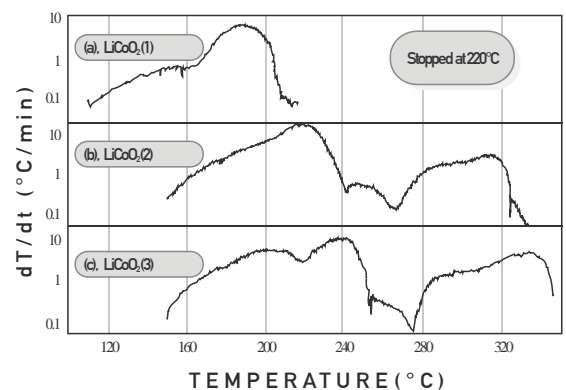


It is the reaction or an interaction between components that can lead to heat release. The first reaction is decomposition of SEI surface layers, anode reaction then internal release of oxygen from electrolyte or cathode that

can self-heat or violently react with the lithiated anode. Pressure measurement is important, disintegration of the battery will lead explosively to fire and smoke.

THT has worked with battery companies to develop the technology and application in this area. The 'Battery Component Kit' contains low mass silver tubes and these can connect to a 'side branch pressure tube'. In this way 'low Φ ' testing with measurement of pressure can be achieved using small quantities of battery component material. Of course where large quantities are available, the ARC can be used without modification using standard 'ARC Bombs'.

Effect of Increasing Component Particle Size LiCoO_2 (a), (b), (c)





Safety Testing of Batteries

Batteries of varying shape and size may be tested in the ARC; they may be accommodated by suspension from the lid section (as is usual) or may be supported directly in the base of the calorimeter. The simplest test is effect of heat upon the battery. It is possible to test batteries at any State of Charge, or age, and it is possible to connect cables to the battery terminals to measure voltage during the test. Open or closed sample holders are available – but as with all samples that undergo significant gas generation, rupture of a closed holder might occur.

A key difference between chemicals and batteries is that batteries have their own 'holder'. Also initial pressure generation is contained. THT offer two possibilities to study pressure associated with batteries, internal pressure measurement or the pressure upon gas release from battery after disk rupture or disintegration.

Aside from onset, the ARC test will determine self heating at all temperatures – and thus gives much more information than Hot Box and other empirical tests. The final potential of the battery to contain pressure or to disintegrate in such an event is important; ejection of battery components will be associated with fire as the lithium reacts with air and the release of potentially toxic smoke.

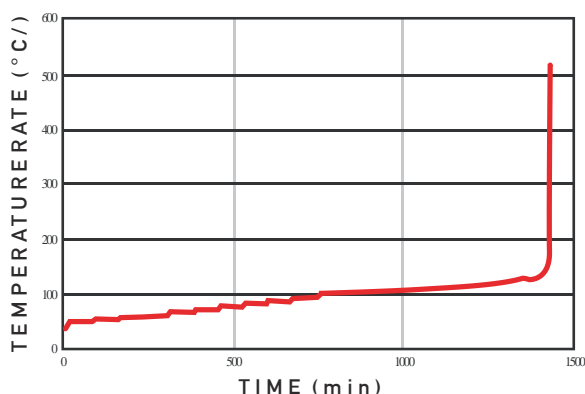
To facilitate pressure measurement THT has developed tanks that will accommodate the battery and be gas tight. These still allow for thermal and electrical measurement. The chamber maybe evacuated or filled with inert gas. In addition to measurement of pressure it is possible after the test to sample gas for analysis. If connected to external analytical instrumentation and with gas flow there is the potential to get real time gas analysis.

The exothermic reactions shown from a fully charged 18650 battery are typically SEI, anode, separator (endotherm) and cathode reacting with electrolyte – as shown. Above 200°C the battery may disintegrate or the reaction may go to completion without disintegration of the battery.

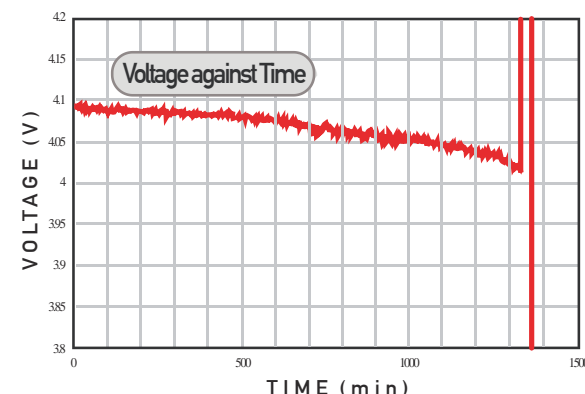
Batteries at different States of Charge or different age will give different heat release profiles. Voltage measurement often shows that batteries will retain their voltage until well into exothermic decomposition as illustrated.

Internal pressure measurement is simply achieved by attaching (in a glove box) a sealed fine pressure line to the battery. Data shown here indicates pressure increase to 4bar prior to the exothermic decomposition commencing. As decomposition proceeds the internal pressure increases and it is not until above 12 bar that the battery disintegrates and there is gas release.

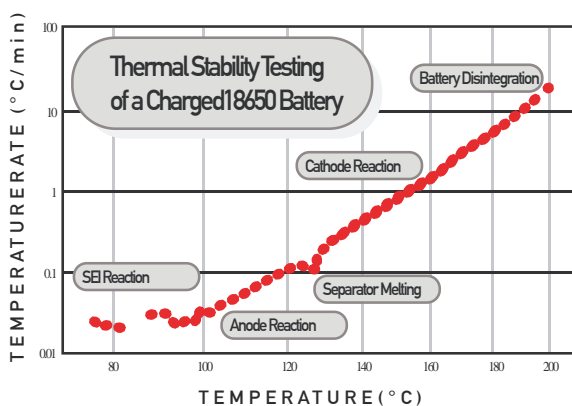
Battery Thermal Stability Test



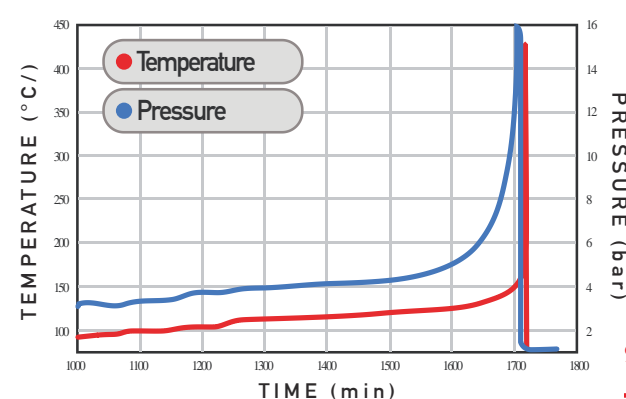
Voltage Against Time Graph



The Exotherm Portion has Overlapping Reactions



Internal Pressure During Test



Use & Abuse Testing of Lithium Batteries

Testing of Batteries under Abuse conditions

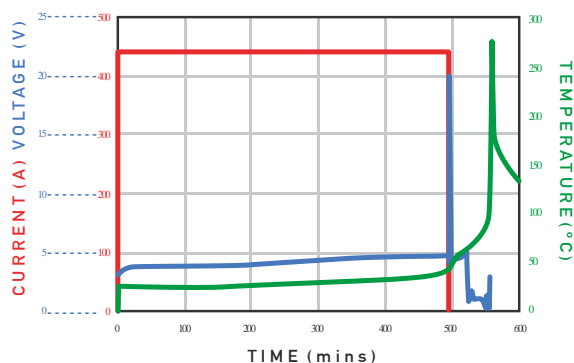
Lithium batteries can fail dramatically when overvoltage charged or when physically abused. Many such abuse tests have been proposed and several are detailed as standard tests required by regulating authorities. Prescribed tests typically give an empirical pass or fail answer. The ARC has potential here to accomplish a range of tests in which abuse conditions are simulated, generating quantitative thermal information. Such tests may be carried out with smaller batteries in the standard ϵ sARC system or with large batteries in the ϵ vARC system.

Several esoteric tests have been achieved within an ARC calorimeter, for example water immersion. However here overvoltage, external short circuit and nail penetration are illustrated.

Options are available from THT to allow either manual or automated (computer controlled) abuse tests on batteries to be carried out within the ARC calorimeter.

Lithium batteries if overcharged are known to self heat and can lead to disintegration. The battery here was overcharged. The battery was subjected to charging at 450mA on a 20V supply. After 200 minutes the battery voltage increased above 4V with associated temperature rise. After 500 minutes there was rapid temperature rise (and cyclor shutdown), the exothermic reactions continued and increased until battery disintegration occurred.

Overvoltage Test

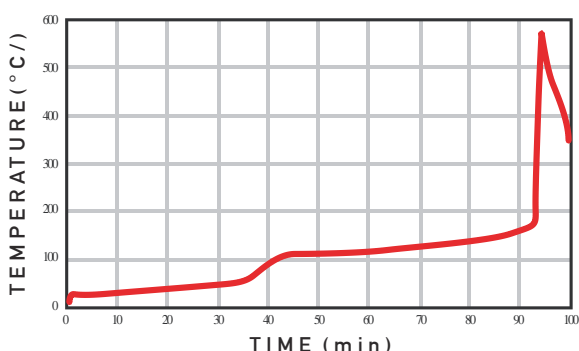


External shorting of a battery within the ARC is simply achieved by joining two low impedance wires connected to the battery terminals. The test is rapid (1-2 hours) and carried out with the instrument in an isothermal mode.

Shorting gives heat output that is tracked by the calorimeter and the amount of heat released can be quantified together with an understanding that this temperature rise can lead to battery disintegration.

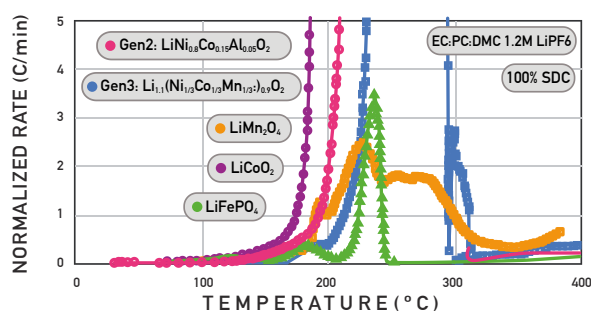
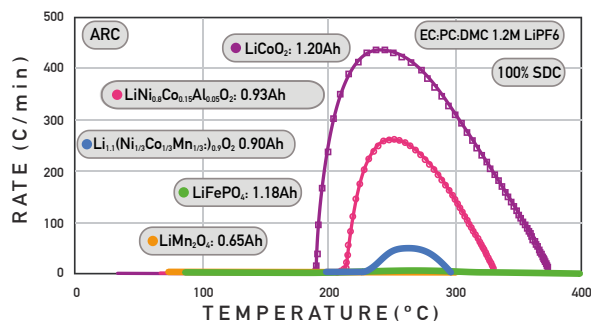
The result shown is from a fully charged battery and shorting leads to a temperature rise of 100°C and then to disintegration. This will not happen for all battery chemistries and does not happen for this battery type if it is fully discharged. In the discharged state, a temperature rise of 30°C occurs. This temperature rise is not sufficient to lead to runaway and Heat-Wait-Seek steps occur.

External Short Circuit



As battery development has progressed; variation in chemistry has led to batteries that are thermally stable to higher temperatures and undergo much smaller exothermic reactions i.e. giving less heat release. These batteries are SAFER, it is naive to say they are SAFE! Data shown for Generation 1 to 5 of cathode material has been published by Dr E. Peter Roth at Sandia National Laboratories and is shown with his permission.

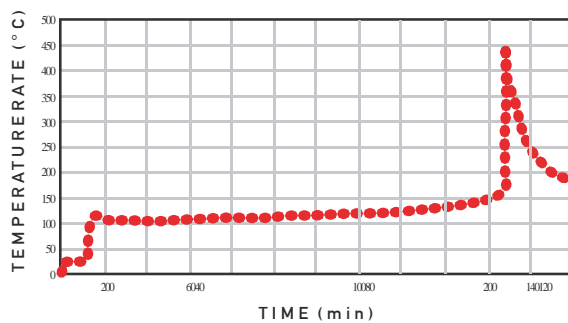
Comparison of Cathode Materials and Reduction in Heat Output





Nail penetration testing considers the effect of such abuse and of internal short circuit. This test can be done within the ARC in a manual or automatic mode. The battery is held on a support and the nail, on the end of a rod is driven through the battery, the thermal effect is measured. Of key importance is to know if the battery temperature will be raised to initiate a disintegration reaction. In the example shown nail penetration results in a temperature rise of near 100°C and there was further temperature rise that led slowly to battery disintegration.

Nail Penetration



Such tests are illustrated here with 'model' 18650 batteries. These are often the choice in development studies. However using the eVARC and BPC calorimeters these same tests can be carried out with larger, indeed very large battery packs and modules. To facilitate these tests Battery Abuse Kits for manual operation and the Battery Safety Unit (BSU Option) for automated operation are available.



Testing of Batteries under Use Conditions

Quantifying the heat generated by lithium batteries during conditions of use allows for an understanding of their efficiency and gives information that is important in determining their use and any thermal or safety issues that may result during normal operation. Variation in heat release from batteries as they age will indicate life cycle.

Heat release relates to internal resistance and though heat generation may not be significant in applications where discharge is slow and gradual, in applications such as electric vehicles or power tools

where rapid and large discharge is needed, this can result in a much greater heat release and temperature rise.

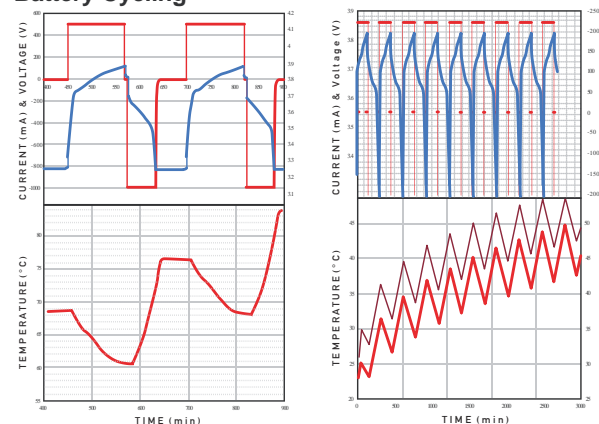
The KSU Option for the ARC is a single channel cycler and several versions are available with differing voltage and current ranges. This may be used or testing can be achieved with a stand-alone customer supplied cycler. In the latter case there will be two sets of data that need to be synchronised.

Often in cycling tests, the battery within the ARC is surrounded by a jacket of insulation. This reduces heat loss and better data can be obtained from tests carried out either isothermally or adiabatically.

Repeated cycling is often implemented. Tests may be carried out with a few cycles in the ARC (when the battery is fresh), the battery removed and repeated cycling performed outside the ARC. Then the test is repeated (with the aged battery) inside the ARC. The change in thermal effect and speed of charge discharge will give a measure of capacity change with time, a change of internal resistance. The efficiency and lifetime characteristics of the battery are determined.

The data below illustrates, left, two cycles of a fresh 18650 battery and, right, several cycles of an aged battery. The associated thermal change, shows charging to be endothermic and discharging exothermic. However the overall trend is for a temperature increase, the magnitude of this relates to the internal resistance of the battery.

Battery Cycling



Again such application can be realised not just with smaller (e.g. 18650) batteries but with large batteries, packs and modules. Cycle life is one key advantage of lithium batteries over different chemistries; cycle life is important in satellite and space application where there may be a day – night charge – discharge rotation; it is similarly important in stationary applications designed for peak shaving and storage.

Calorimeter Choice

EV+

EV+ Calorimeter

The EV+ Calorimeter has been designed for EV cells and small modules and to make the needs of testing necessary with EV batteries.

Many prescribed 'standard' tests call for Calorimetry and also call for use of abuse testing (eg SAND, SAE). Tests call for video monitoring, gas detection/analysis, nail penetration and crush under controlled conditions. The EV+ calorimeter from THT has been designed to accommodate many of these tests - and more whilst gaining quality information on heat release.



Key features of the EV+ are its size and its ability to:-

- Have good calorimetric performance
- Incorporate the range of appropriate options

The EV+ is an cylindrical calorimeter 40cm in diameter and 44cm depth. Unlike other THT adiabatic calorimeters the lid seals to the base unit. This seal is restricted to below 1 bar and is maintained by electromagnets. This allows for inertion of the environment and the ability to collect products of battery disintegration. A gas collection line (leading to gas collection bag) is standard.

Also standard on the EV+ calorimeter are:-

- Integrated cables for current & voltage measurement
- Video camera (with light)
- Ability for battery pressure and temperature measurement
- Ability for purging, evaluation, inerting

The calorimeter is ready for addition of further options:-

■ Sub Ambient Operation, Cryogenic Option *Calorimeter Ready.*

Pressure tight ports provided in calorimeter which can be blanked off or used if cryogenic (LNFO) option is available operates to -50°C.

■ Heat Capacity Measurement Cp Option *Calorimeter Ready*

Pressure tight ports provided in calorimeter which can be blanked off or used if Heat Capacity Option (CPO) is available.

■ Surface Area Heat Distribution, Multipoint Option *Calorimeter Ready*

Pressure tight ports provided in calorimeter which can be blanked off or used if either the 8 thermocouple, 16 thermocouple or 24 thermocouple Multipoint Option (MPO) is available.

■ Controlled Speed Nail Penetration & Crush Option *Calorimeter Ready*

Pressure tight ports provided in calorimeter which can be blanked off or used if the NP option is available. The latest THT Nail Penetration option allows functionality to SAND 2005-3123 & SAE J2464 specification. (EC-CS-NPCO). The NP option is pressure tight to the calorimeter (it allows in operation in situ gas collection and video monitoring). The NP option allows for nail penetration at controlled speed and crush with movement stop at voltage drop.

In built safety features are standard to the EV+ calorimeter and it is housed within the THT 'EV Containment Vessel'. Multiple shut down features add to the fail safe mode of operation.

The EV+ links to THT hardware and software to allow modulating and upgrade possibilities at moderate cost.

Gas analysis options can be supplied by THT or THT can advise on specific options for gas analysis; real-time MS or GC-MS.

Calorimeter Choice BPC & IBC



The Battery Performance Calorimeter (BPC) *Thermal Management, Efficiency & Lifecycle Studies.*

The Battery Performance Calorimeter (BPC) is a large volume calorimeter developed for research studies of larger (EV) cells and modules. The BPC utilise ARC electronics and software and is modular with the standard, EV and EV+ calorimeters. It can be housed in the standard THT EV containment vessel.



The BPC is designed to quantify heat changes during charge and discharge – at conditions that simulate use of the battery.

The BPC is not appropriate for stability, safety and abuse studies where battery disintegration is possible. The BPC has an upper temperature limit of 200°C but operates down to -40°C. Cryogenic operation is possible by linking to a refrigerated circulating bath.

A key and unique feature of the BPC is the 'Thermal Diode' heating system that allows current flow through the calorimeter. This eliminates need for conductive leads or cables to carry current. At high power operation such loads do come major heat loss and data error in calorimeters with no 'thermal guard'. (The EV+ uniquely does have thermally guarded leads and these do minimise error)

The calorimeter has a depth of 50cm but its cross section is oval (50-65cm) maximising useful volume for large batteries.

With heat detection sensitivity at 0.005°C/min the BPC has stable specific detection for heat release. In conjunction with the THT surface area (multipoint) heat measurement option, the BPC at well below 1 w/g detection, is ideally suited for gaining information appropriate for Thermal Management.

The BPC used with THT ARC software and electronics can be used within the adiabatic isothermal or isoperibolic modes. The choice of mode relates to the studies undertaken.

Key options used with the BPC are the surface area (multipoint) option and the heat capacity option. The BPC complements the other calorimeters available from THT.

The Isothermal Battery Calorimeters (IBC)

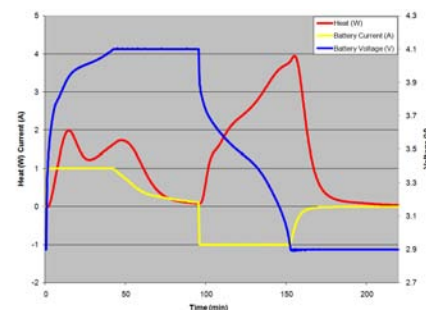
THT have a range of isothermal battery calorimeters. These complement the ARC-based calorimeters however they do not use ARC electronics or software.



The need for true isothermal calorimeters is when charge discharge or self discharge isothermal testing is appropriate.

The isothermal battery calorimeters are fully described in their specific brochure.

Isothermal calorimeters have higher sensitivity than adiabatic calorimeters but have a limited range of applications. Their temperature range is limited and they are not appropriate for safety abuse testing when battery disintegration or high temperatures may result.



The THT range of isothermal calorimeters includes size specific calorimeters that have been built in cyclor capacity. The high sensitivity of these units allows the measurement of small coin cells. The IBC for prismatic batteries can have integrated

multipoint heat measurement. This allows the variation of heat release over the surface area of the battery to be measured.

High sensitivity self-discharge calorimeters (IBC-SD) are available. The chamber size (typically Double-D) means that a wide variety of small cell may be accommodated. Accelerated self-discharge tests may be accomplished of long shelf-life batteries. Special build multi-well (4 sample) self discharge calorimeter (IBC-SD4) are available with chamber size being cell specific.

EV+ Options: Sub-Ambient Testing & Thermal Distribution

EV+ Calorimeter

Larger batteries and batteries / modules / packs have application requirements that extend from the application of smaller batteries.

With large batteries there is still the need stability and safety testing, use and abuse testing however larger batteries applications often need high power. This maybe discharge of 10-100°C and potentially charge in minutes rather than hours. The applications focus on destructive abuse and thermal management under use scenarios. The size of the batteries and applications has led to the need to devise additional application options.

Cryogenic Applications

There is the need to evaluate battery performance and thermal aspects of its operation at all environmental temperatures. These may be to a temperature of -30°C or below. Temperatures where electrodes could freeze!

The THT CryoCool option to accommodate such testing at modest cost. The CryoCool option simply allows testing to begin at sub-zero temperatures by cooling with a flow of ultra cold nitrogen / liquid nitrogen. The CryoCool option 'plugs in' to the EV or EV+ calorimeter.

Surface Area Heat Determination - MultiPoint Option

For larger batteries and modules it is key to determine where heat release under use or abuse is focused – how the temperature rise varies through the unit.

The MultiPoint option provides a multiple thermocouple facility to achieve measurement of thermal distribution over the surface of the battery, pack or module. The MultiPoint is available with 8,16 or 24 thermocouples to be positioned where appropriate. The temperature at all points is recorded and control can be at any of these positions.

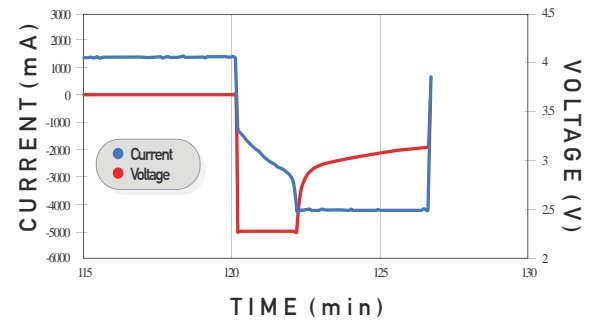


MultiPoint calorimeter tests obtain data more accurately than open bench tests. In the calorimeter the environment is controlled and unknown and un-quantified heat loss is minimised. Conditions are worst case and the final equilibrated battery

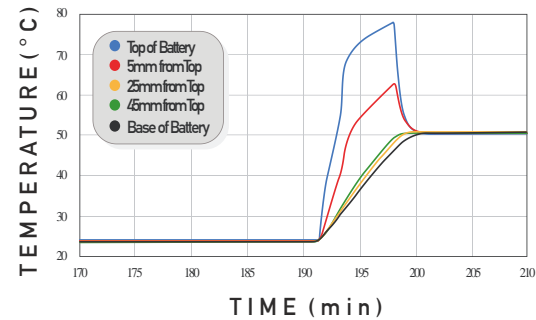
temperature is recorded. Heat effects using such calorimeters are carefully quantified.

Multipoint data is illustrated for a small battery (18650).

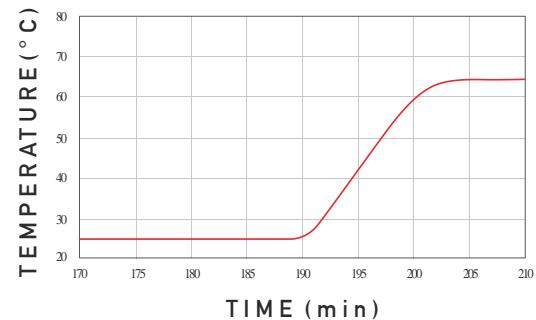
Cycler Data



Spatial Temperature of Battery



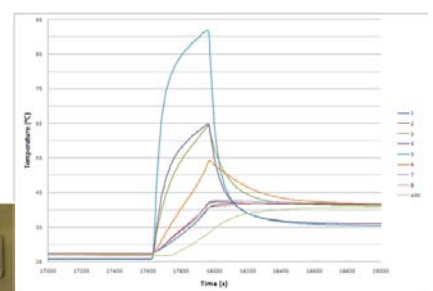
Control Temperature as a Function of Time



Note the time scale of the test, the speed of heat release and temperature increase – and that this is primarily at the anode collector. The speed of battery thermal equilibration is illustrated. The discharge profile is shown and also the calorimeter temperature, controlled by the 'average battery temperature'.

With a single large pouch cell the data might be more complex.

Data here illustrates a 100 amp discharge experiment.



EV+ Options: Heat Capacity, Thermal Management & Abuse Testing

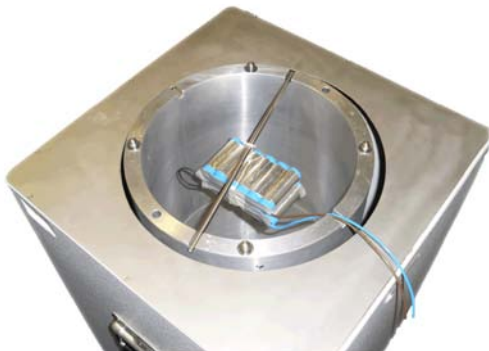
Heat Capacity

A value for the overall heat capacity of the battery is needed to allow conversion of standard calorimeter data to react with units of joules (heat) and watts (power or speed of heat release).

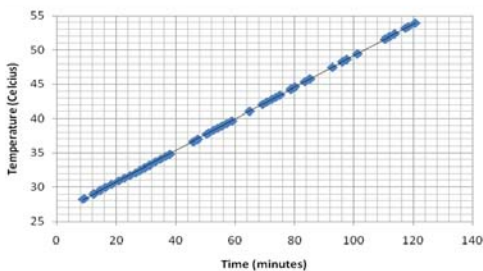
There are many methods by which heat capacity can be determined through typically these involve an additional heater. The heater is in contact with 1 or more batteries, power is supplied and the temperature rise of the battery relate to the heat capacity.

The THT Heat Capacity Option is supplied with heaters appropriate for batteries of the size to be measured. The unit utilises aluminium reference samples. The method is automatic and leads directly to determination of the 'overall' heat capacity.

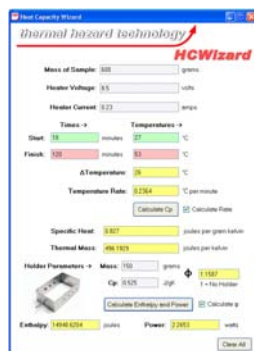
The THT ARC data analysis software has ability to take in sample heat capacity and generate automatically Enthalpy and Power graphs.



Battery pack wrapped in aluminium tape suspended within the calorimeter



The raw data is shown in above. THT has a Wizard to calculate the specific heat and heat capacity at any temperature (or over any temperature range). Taking the value for the temperature rate (at or averaged over a temperature range), and with knowledge the mass of the battery pack and the voltage and current supplied to the heater, we can then calculate the Cp value and heat capacity using the heat capacity wizard software.

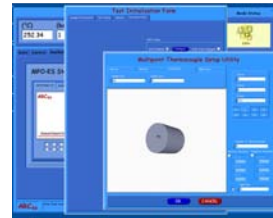


From this test the wizard calculated an average (mean) Cp value over the entire temperature range of the experiment of 0.83 J/gK.

Thermal management of EV Batteries

Utilising the THT EV options, key information is available for thermal management of EV batteries. The battery may be subjected to EV use conditions by charge/discharge with an EV test system (eg Bitrode XXX, dSpace coupled to high power charge and load units or stand alone high power charge and load units). The test system might apply repeated charge-discharge cycles or a prescribed drive scenario.

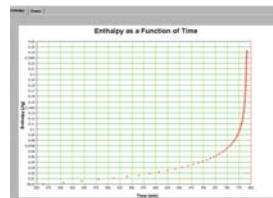
The ARC/EV+ calorimeter equipped with Multipoint and Heat Capacity options will give the thermal management information. Initially the specific heat capacity must be available. This value is simply put in to a MultiPoint Test allowing for generation of Enthalpy and Power graphs.



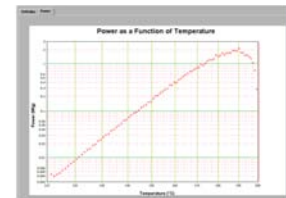
Software Set Up for MultiPoint



Screen Display During Test



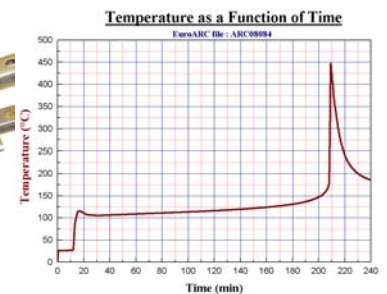
Enthalpy at One Point



Power at One Point

Nail penetration and crush – controlled speed

Tests call for nail penetration at defined speed and crush to be terminated at voltage drop. This presents challenges though the THT option fulfils all requirements. The THT NPCO can be added to the EV or EV+ calorimeter.



Data from a nail penetration test, shown above. Initially the system was held isothermally before nail penetration commenced. Following the penetration the cell led into complete thermal disintegration (in fully adiabatic conditions).

History / Users

History

The Accelerating Rate Calorimeter has a long history of being the favoured technology to study lithium batteries. Clearly this is down to the ARC's...

- Ability to accommodate large samples
- Rugged and Robust construction
- Possibility to connect cables in-situ to allow charge and discharge
- Quality adiabatic control

Lithium and sulphur dioxide batteries were being investigated over 30 years ago – the first publication known to THT being *Eber W. B & Ernst D. W, Power Sources Symposium June 1982; Safety Studies of the Li/SO₂ system using Accelerating Rate Calorimetry*. The Li/SO₂ battery being a primary cell considered for defence applications.

However the major impetus for Lithium battery use was triggered by the advent of the Li-ion secondary 18650 cells pioneered from the mid 1990's by Sony. Sony was the first company to buy a THT ARC system for battery studies.

Initial studies were using 18650 cells and centred on stability and safety – and improvements to stability with changes in chemistry. This work either at cell level or at component level was the primary work carried out in the later 1990's and early 2000's.

The focus on safety at this time was crucial due to applications in cell phones and laptop computers – and well documented incidents that led to hugely expensive 'recalls'. The manufacturers were predominately Japanese companies.

From the year 2000 application areas expanded rapidly; large format (prismatic and pouch cells) appeared. The potential to use the ARC to study cells and small modules in situ under a variety of use and abuse conditions was realised. Within the period of 2000 - 2010 THT worked with organisations around the world to implement the options described in this brochure.

From 2005 and until today large format cells have become more established for high power applications. This led to challenges that THT has met with the large format calorimeters EV+ and BPC.

Key users of THT arc systems now are Tier 1 automotive OEM's, their suppliers and specifiers.

Going into the 2010's it seems likely that most application areas have been addressed though in the future new challenges will no doubt arise where THT will aim to fulfil.

Users of THT ARC

Sony	ITRI
Nokia	Samsung
NREL	Panasonic
Mitsubishi	Lishen
NASA	BAK
Sandia National Labs	Lion Cell
ATL	All Cell
CEA	Hyundai - Kia
LG	Shin Kobe
Nissan	Kokam
Sanyo	Tianjin Institute of Power Sources

4 Key Tangible Benefits from THT

- Latest Hardware and latest Labview Software
- Highest Sensitivity, Widest Performance Features
- Large Volume EVARC and BPC Calorimeters
- Integrated Cyclers & Battery Abuse Options

4 Key Intangible Benefits

- Largest Global Customer Base
- Most Experienced Technical Personnel
- Lifetime FREE Phone & -E-Mail Support
- Worldwide Offices & Support

Specification



ARC Common Components

- Safety; 1-3 cubic meter containment vessels (allows options); reinforced 3mm steel; proximity switch, door interlock.
- Electronics 3kVA or 7kVA power supply system
- Workstation with Microsoft Windows and NI Labview™; flat screen monitor, keyboard and mouse
- Real Time software in NI Labview; on-the-fly conditions change and full control; remote operation
- Remote User; ability to transfer operation of system to any allowed PC over network or internet
- Virtual Technician; ability to set up multiple tests in one method
- Modes; Adiabatic; quasi Isothermal; true Isothermal; Iso-peribolic, Ramping
- Operation in air, vacuum, inert gas, reactive gas, flowing gas
- Adiabatic control to 0.01°C
- Pressure resolution 0.005bar; precision 0.02%; accuracy 0.05%
- Sample holders; ARC Bombs, low phi holders, tube bombs, special open or closed holders for any battery type
- Data Analysis software in Labview with ability that includes
 - Graphical and tabulation of raw data including Phi Corrected tmr plots
 - Data Conversion to Enthalpy, Power, Gas Generation
 - Kinetic Modelling for thermodynamic and kinetic data analysis
 - Phi Correction through kinetic modelling
 - Report generation in Microsoft Word, Excel, html
 - Analysis of 9 data sets; 3 analyses on each data set, 5 merge datasets
- Temperature resolution 0.001°C; precision 0.01%; thermocouples external and internal
- Vacuum to 200 bar pressure range (10-2000 bar with alternative transducers)
- Lifetime email and phone support, 1 Year warranty
- CE, UL, VCCI, CSA test certification

Standard Calorimeter

- Fully compliant to ASTM E1981 E27
- Calorimeter design to Dow Patents of 1980 and 1984
- 10cm diameter 10cm depth calorimeter
- Temperature range 0-600°C (-40°C with cryogenic system)
- Sensitivity: 0.002°C/min to 200°C, 0.005°C/min to 400°C; 0.010°C/min to 500°C
- Tracking Rate to 20°C/min
- Gas Collection via canister
- Slotted base for thermally guarded cables
- Pneumatic Nail Abuse Testing

Fast Tracking

Designed for Vent Sizing Applications, not appropriate for abuse Battery work

- 10cm diameter 10cm depth calorimeter
- Temperature range 0-400°C (-40°C with cryogenic system)
- Sensitivity 0.02°C/min
- Tracking rate to 150°C/min
- Size and shape similar to standard calorimeter

EV Calorimeter

- 25cm diameter 50cm depth
- Temperature range 0-400°C (-60°C with cryogenic system)
- Sensitivity 0.02°C/min
- Gas collection via canister
- Collar for abuse testing
- Thermally guarded cable insert
- Pneumatic or Control Speed Nail Penetration Crush option

EV+

- 40cm diameter 44cm depth
- Temperature range 0-300°C (-60°C with cryogenic system)
- Sealed lid designed for integral gas collection (Tedlar bags or cylinder)
- Automatic electronic safe lid lift
- Sealed lid pressure limits 0-2 bar
- Integrated video monitor
- Integrated Inert gas purging facility
- Integrated Battery Cable Connectors

BPC

Not designed for Vent Sizing Applications, abuse Battery work

- 40x60 elliptical x 40cm depth
- Temperature range -35 -200°C (with refrigerated circulating bath)
- Sensitivity 0.01°C/min
- Integrated Battery Cables



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