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Technical Information Sheet No 50

Accelerating Rate Calorimeter and Options Overview and Current Status (last modified, March 1999)

The Thermal Hazard Technology Accelerating Rate Calorimeter is the second generation Accelerating Rate Calorimeter system. The current development team incorporate over 50 man years of experience, part of this the experiences of the original CSI International Office staff. The THT instrument was developed in principle on basis of Continuity, Enhancement and Expansion:

(1) Continuity

The THT Accelerating Rate Calorimeter is designed to perform in an identical manner as the original CSI system, ensuring data continuity and direct comparison with original system data. Many of the control algorithms have been retained and improved upon, such that for an identical sample and identical test setup parameters, the data sets from the original CSI system and the THT system are almost indistinguishable. The THT instrument on the other hand is designed to operate at sensitivities down to 0.005°C/min, and has an identical operating range as the original system. This 'continuity' aspect has been proven using 'standard' samples – request data from THT.

(2) Improvements (Enhancement)

Improvements have been made to the system in a number of areas as follows :

Low Phi Tests – A higher volume sample container is available. This can be used as a stand alone though would also be used with Options (eg the Vent Size Unit and the Stirring and Dosing Unit). In using this there is further hardware – the calorimeter through lid pressure adapter is modified and a Burst Disc Assembly must be used. The Burst Disc Assembly sits on the lid of the calorimeter, it has a catch-pot and though a variety of discs are available to allow rupture at differing pressures there is a standard disc to ensure safe operation of the unit. With this add-on improvement it is possible to use the THT Accelerating Rate Calorimeter at ϕ -values to below 1.1. Isothermal Tests - The first involves a smarter isothermal run algorithm which reduces

any small drift with time, holding a steady temperature with a fraction of the reheat cycling experienced with the original system when negative drift was encountered. The elimination of any gradual positive drift to higher temperatures once again present in the original system, has also been implemented. The isothermal mode may be truly isothermal (control of the calorimeter) or quasi-adiabatic (control of the sample).

Ramp Tests - The standard system also incorporates the capability of ramp testing a sample. A ramp-wait-seek cycle is implemented, with the use of a specially designed shield that allows the system to detect self-heating rates below the temperature ramp rate being used (for instance ramping at 1°C/min and detecting at 0.2°C/min). When an exotherm is detected during ramping between the test start and end temperatures, the system executes a wait and then confirms the exotherm during a seek step. If confirmed, the exotherm mode is implemented. This approach is designed to speed up the testing procedure, and may be used as a precision screening method.

Specific Bomb Types – Battery Bombs – THT has collaborated with major companies in the area of battery research and safety. This has led to development of containers for batteries of various size and shape (AA, 18650, prismatics...). These holders have been both 'open' and gas-tight. In addition gas-tight battery holders have been developed that allow the battery to be connected with leads via gas-tight fittings. This allows charging, discharging etc of the battery to be done during the test.

(3) Auxiliary Options (Expansion)

System expansion has also been a primary driving force in the design of the next generation instrument. A number of specially designed add-on modules have been developed. Currently the following are available :

(i) Gas Scrubbing Unit (GSU) - This system is designed to handle particularly toxic vapourous and gaseous products, purging the test cell of its volatile components and passing them through an integral scrubber unit within the blast shield. In addition to this a gas sampling port enables sampling of the volatile products for subsequent FTIR and/or G-C Mass-Spec analyses. This again is done automatically, ensuring total user safety.

(ii) Vent Sizing Unit (VSU)- Thi option provides the user with all the necessary equipment needed to conduct vent sizing experiments as prescribed in the DIERS methodology. These include :- closed test; low- ϕ closed test, tempering test, top vented blow-down test, bottom vented blow-down test. 65ml low- ϕ test cells are used, capable of withstandinging up to 150 bar (2000 psi) of pressure and in the case of the closed tests a special burst-disk catch-pot assembly is used to prevent the test cell

from rupturing, The Thermal Hazard Technology ARCCal[™] analysis package supplied with the instrument handles all necessary calculations based on the Fauske relationships.

(iii) Battery Safety Unit (BSU) - this unit is designed for the testing of rechargable (mainly Lithium) batteries under standard charging/discharging conditions as well as battery abuse. Specially manufactured battery test cells for both closed and open tests are available and may be designed to the user's specifications. See further specific information.

(iv) Fan Extraction Unit (FEU) - This unit is a system add-on which provides the THT Accelerating Rate Calorimeter system with an automated draughting facility, eliminating the need for user supplied draughting equipment. In addition it is pulsed during the operation of the VSU to ensure that any ejected volatile components are removed from the user-instrument environment..

(v) Cryogenic System Unit (CSU) - which will enable sub-ambient operations

(vi) System Stirring Unit (vii) Dosing System Unit

Thermal Hazard Technology is committed to developing additional units. In the pipeline are; Stirrer System Unit (SSU) which will provide direct-link agitation of sample; Dosing System Unit (DSU) which will provide a means of adiabatically dosing materials into the test cell at high temperatures and pressures.

(4) PC Interface

The system interface is Windows 98/Windows NT based, and provides the user with an easy-to-use front end. Test status is clearly shown in text, graphical output and clip-art forms. User implemented changes are executed from this interface, as well as manual overrides. Real time data (.DAT) is stored to the hard disk throughout the test, whilst during an exotherm fast-data is stored to the system memory for saving to disk after the test has been completed (.EXO). Although the PC front end is the primary user interface, a provision is made on the main instrument control system where a basic keypad interface for implementing mode changes and diagnostic tests is provided.

ARCCal[™] is the standard analysis package supplied with the system, and allows the user to handle data and analyse it manually, semi-automatically and automatically. Thermodaynamics, thermokinetics, and safety parameters (such as TMRs and temperatures of no return) may be determined. The package can also automatically generate a finished report in Microsoft Word prompting the user for input when

necessary. A GLP (Good Laboratory Practice) approach has been adopted conforming to the requirements of most medium to lartge scale companies. This ensures that test parameter and data integrity are maintained up to the finished report stage of an analysis. ARCCalTM is also capable of implementing vent sizing based on the DIERS methodology, and is primarily designed for use with the VSU, although if data was reformatted correctly, could be used with the Fike VSPTM and/or the HEL Phi-TecTM. Network Connection: An add-on option is available to allow the THT Accelerating Rate Calorimeter PC to be fully networked. A data window can be called up from any PC on the network and the system PC can be accessed from any linked system.

(5) Upgrades

Thermal Hazard Technology will release updated versions of the system firmware and PC based software, incorporating improvements and new routines. All our users recieve these upgrades which may be performed within 20 minutes using the Thermal Hazard Technology Check-Up software.

(6) Support of all Accelerating Rate Calorimeters

Thermal Hazard Technology are committed to supporting not only their own systems but provide support to many users of the original CSI system. We manufacture calorimeters and parts for the original design, and provide back-up with regards to servicing and data analysis where needed.

(7) Synergistic Technology

Thermal Hazard Technology is committed to assist engineers and scientists whose work and responsibility involve reactive chemical hazards. THT therefore work with otheres providing highest class synergistic products. THT cooperate with and help distribute and support the Cheminform Thermal Safety Software (CISP-TSS). Recognised as the world leading software for the complete analysis of calorimetric data this allows full thermokinetic evaluation and plant simulations of any material from original data from any type of calorimeter.

(8) International Standards and Safety

Recognising the need for instrumentation to meet all current regulatory requirements, safety and other issues, the THT Accelerating Rate Calorimeter is fuly tested to meet its legal and moral obligations. The insrtument and all options are CE tested and

marked, comply to VCCI rules (Japan) and other standards. The system is PAT tested and Year2000 compliant. This is not the case with some competitive instrumentation – that cannot legally now be sold in certain geographic regions!

Stop Press

The EVARC^m.

Thermal Hazard Technology is currently in an advanced stage of design and development of the $EVARC^{TM}$ option.

This major development is to assist those developing larger scale batteries suitable for either Electric Vehicles (EV) Hybrid-electric Vehicles or for Storage applications.

The option can be provided as a separate unit – to link into the THT Accelerating Rate Calorimeter such that tests can be carried out with either 'normal' sample types or with the EVARCTM option installed larger batteries can be tested. Alternatively the EVARCTM can be bought as a stand-alone unit solely for testing large batteries.

The EVARC^{TM} option consists of a redesigned (larger) calorimeter and a larger and much more strongly constructed containment vessel. The system is designed to be fail-safe in operation due to the potential risk of explosion with large batteries.