

ADIABATIC CALORIMETRY

For DIERS Vent Sizing Data, Advanced Thermal Stability Analysis and Runaway Reaction Characterisation, the assessment of runaway reactions and thermal decompositions is a critical part of the safe operation of a chemical process or drying operation. Obtaining laboratory data that can be directly scaled-up to the production size of most plant operations requires that both the heat losses and thermal inertia (phi factor) of the plant be replicated. Adiabatic calorimetry is designed to provide data that can be used for consequence assessment, emergency relief system design (using DIERS methods) and accurate thermal stability assessment.

WHEN IS ADIABATIC CALORIMETRY REQUIRED?

An adiabatic calorimeter is required when data is sought under low heat loss, low thermal inertia conditions. This may be when a direct simulation of a process deviation requires characterisation or when thermal stability screening tests indicate a narrow margin between normal processing conditions and the onset conditions of a hazardous event (e.g. an undesirable decomposition). Sizing an emergency relief vent for a runaway exothermic reaction demands adiabatic test data and is the primary application of the technique. However, the apparatus is also used for other forms of consequence analysis and designing safety systems to protect against a runaway process.

WHAT IS THE DATA USED FOR?

Adiabatic calorimetry data is typically required to characterise:

- the onset temperature of thermal instability of a process mixture or material under sensitive conditions (which requires provision of a negligible safety margin)
- the consequence of a process deviation (or deviations) under pseudo-plant scale thermal conditions
- the rates of runaway reactions
- the data required for emergency relief system design calculations using best practice methodologies (i.e. DIERS) for runaway reactions
- the nature of flow from the venting of a runaway reaction.
- the time to maximum rate of a runaway reaction from inception, or the definition of the temperature from which it takes a reaction 24 hours to reach its peak rate (often referred to as the T_{D24})

WHAT IS ADIABATIC CALORIMETRY?

As the name implies, adiabatic calorimetry involves the quantification of energy release from a reaction or decomposition under zero heat exchange (adiabatic) conditions. Large scale process vessels are typified by very low heat losses to the environment and a low thermal inertia (i.e. the reaction vessel absorbs little of the heat generated by an exothermic process). In adiabatic calorimetry, reactions are allowed to proceed under zero heat loss conditions and, in most cases are designed to have a very low thermal inertia. The data obtained can represent a direct simulation of the large scale process vessel.

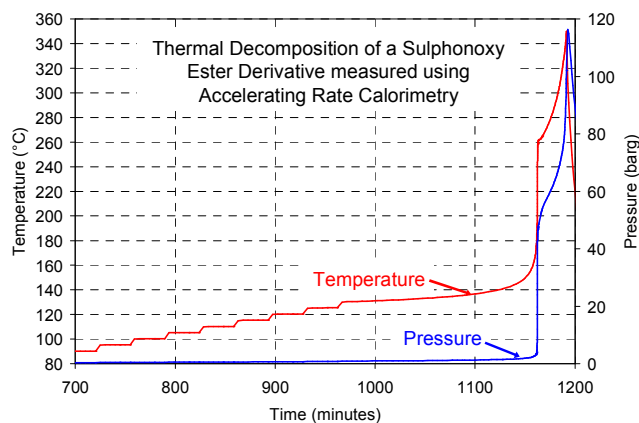


ADC II from Chilworth Technology

WHAT METHODS OF ADIABATIC CALORIMETRY ARE AVAILABLE?

Methods for adiabatic calorimetry generally fall into two categories: pressure resistant adiabatic calorimetry (such as Accelerating Rate Calorimetry (ARC) and Adiabatic Pressure Dewar Calorimetry (ADC II)), or pressure compensated adiabatic calorimetry (such as the Vent Sizing Package (VSP II)). Whilst fundamentally operating in a similar manner and producing comparable data, each method has its advantages and disadvantages.

Accelerating Rate Calorimeter (or ARC), is typically used for determining the onset temperature of decomposition for unstable materials and to provide kinetic data for energetic processes which cannot be obtained in less robust calorimeters of this type. The ARC is a fully adiabatic calorimeter which has a moderate thermal inertia. This type of test is not normally directly scalable but data can be mathematically corrected to account for the thermal inertia. The ARC has a very high pressure and temperature capability (typically up to 500°C and 200 barg) and is an ideal technique for advanced thermal stability testing or testing of highly energetic compounds which may prove dangerous in larger scale calorimeters. Since the ARC is not easily agitated, its application for reaction simulation is limited. Similarly, the small scale nature of the bomb makes reliable semi-batch addition studies prohibitively difficult.



Adiabatic Pressure Dewar Calorimeter system (or ADC II) has a lower pressure capability (typically to 40 barg) than the ARC but the thermal inertia is much lower and reactions can be performed under heat loss and thermal inertia conditions which are directly applicable up to 25 m³ (without any mathematical adjustment). Compared with other adiabatic calorimeters, the ADC II system offers several distinct advantages:

- exceptional thermal stability and heat retention (even at elevated temperatures and pressures)
- mechanical agitation (via anchor or impeller) with torque and speed measurement
- semi-batch additions can be made readily and in a well controlled manner
- the potential to rapidly change from temperature controlled conditions (using internal cooling coils) to adiabatic operation, whilst maintaining a low thermal inertia
- the study of runaway reaction mitigation strategies. For example, assessing the efficacy of reaction inhibition

as a basis for arresting incipient runaway reactions by chemical inhibitor injection.

The instrument is extremely versatile and is readily tailored to most applications. The larger test cell (1 litre) readily facilitates controlled semi-batch additions of liquids and gases whilst maintaining low phi factors down to 1.05 in most configurations. The apparatus can be used for differentiating between gas generating and vapour pressure controlled reactions through the conduct of runaway tempering trials. It can also be used to establish flow regime characteristics (single or two-phase) and fluid flowability/viscosity via discharge trials. Simulation of runaway reaction dynamics under fire engulfment conditions is simple to undertake with the ADC II.

Vent Sizing Package (VSP II)

The Vent Sizing Package, was originally developed during the DIERS project in the mid-1980's to collect data for vent sizing calculations on runaway exothermic reactions and decompositions, and later commercialised as the VSP II. Unlike the Adiabatic Dewar and ARC systems, which use thicker wall test cells to withstand high pressures, the 120 cm³ VSP test cells are thin walled with lower phi factor. The apparatus is capable of studying reactions to high pressures by using pressure balancing, whereby the lightweight test cell is placed in a high pressure chamber where the containment pressure is regulated close to that inside the cell. Maintaining a low differential pressure permits reactions to be studied up to 100 barg. The test is fully adiabatic and operates with very low phi factors typically down to 1.05.



VSP II Apparatus

The apparatus is agitated either through magnetic or mechanical stirrers and reactions can be conducted under standard sealed cell conditions or to examine the tempering

or flow regime characteristics of reaction runaways. The study of semi-batch additions is possible using pressure injection systems. This unit is one of the most well known adiabatic calorimeters across the world and is particularly popular in the USA for the study of runaway reactions.

WHAT METHOD OF ADIABATIC CALORIMETRY DO YOU NEED?

Each adiabatic calorimetry method has its relative merits for specific applications. For exceptional thermal stability performance, for example studying the thermal instability of a mixture over a prolonged period, the Adiabatic Dewar technique excels. For the study of highly energetic materials and mixtures, the ARC is a safer yet equally reliable tool. For studying reaction systems that generate high pressures by gas generation or use of volatile solvents, the VSP II has an excellent peak pressure capability combined with good agitation performance.

In having access to all three calorimeters within our laboratories, it is possible to select the most appropriate technique for the specific application – resulting in the highest quality data for safety studies or protection system design.

WHAT CAN CHILWORTH PROVIDE?

Our fully equipped laboratories and consulting staff are available to assist you to select the most appropriate test (or tests) for any specific application. Once we understand your needs and specific plant situation, we will tailor an experimental programme to your material or reaction. We ensure that unnecessary tests are not performed. We do not just provide data – we always provide a complete technical report which outlines the rationale behind each test, the safety margins that should be applied (if any) and concludes with recommendations regarding safe processing conditions (made by one of our Process Safety Specialists). Our customer support service is second to none.

Our consulting team can readily utilise the experimental data to calculate emergency relief system requirements (for example using methods developed by the DIERS project) or to assist in the specification of a safety system and basis of safety for a plant operation.

faxback

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IEC61508/11 and SIL Determination.....	<input type="checkbox"/>	ATEX / DSEAR Audits	<input type="checkbox"/>
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CHILWORTH DIRECTORY OF SERVICES

With the recent expansion of services at Chilworth it is useful to review what is now available. The list below is an overview of the specialist areas where we are able to provide expert advice.

Consulting Capability



Organisation & compliance

- Safety Management
- Regulatory Compliance Support

Technical Specialist Areas

- Hazard Assessment and Risk Analysis
- Explosion Prevention & Protection
- Modelling
- Chemical Reactions
- Energetic Materials
- Electrostatics
- Fire Prevention & Protection
- Occupational Health & Safety
- Reliability

Protection & Equipment

- Inert Gas Protection
- Instrumentation and Equipment
- Pressure Vessels

Project Engineering

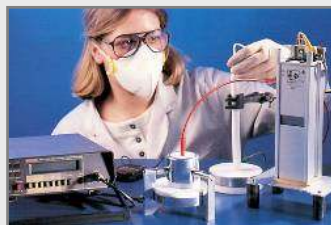
Incidents & Support

- Incident Investigation
- Litigation Support

Insurance Risk

Environment

Testing Capabilities



Process Safety

- Special Testing
 - Laboratory Testing
 - Field Tests (Large Scale)
- Explosion (Deflagration)
 - Dust
 - Gas / Vapour
 - Hybrid
 - Aerosol
- Thermal Stability / Chemistry
 - Chemical Reaction Hazards
 - Powder Thermal Stability
 - Chemical Process Optimisation
- Explosion (Detonation)
 - Propellants / Pyrotechnics
- Explosives / Fire
 - Mattress / Furniture
 - Custom Tests
 - Full Scale Simulation

Electrostatics

- Process Problems
- Applications
- Safety Test

Regulatory

- UN / DoT Transportation
- Classification Packaging Labelling (CPL)
- MSDS
- Notification Registration Evaluation
- REACH

Training Capabilities



Organisation & Compliance

- Process Safety Management
- Process Safety Culture
- COSHH
- OSHA Dust Explosion Prep Training
- ATEX 137 / DSEAR
- Environment / Integrated Management Systems
- Process Hazards Analysis

Technical Specialist

- Dust Explosion Prevention & Protection
- Control of Static Electricity
- Gas & Vapour Explosions
- Chemical Reaction Hazards / Thermal Stability
- Hazardous Area Classification
- HAZOP

Protection & Equipment

- IEC 61508/11 SIL Levels
- ATEX 94/9
- Hazardous / Electrical Area Classification
- Vent Design (Explosion, Pressure, Reactor Protection)

Instrument / Equipment Supplies (Chilworth Systems & JCI)



Process Safety Laboratory Equipment (Chilworth Systems)

- Special Equipment
 - Large Scale Explosion
 - High Pressure / Temperature
 - Custom Design
- Explosions
 - Dust / Gas / Vapour
 - Explosion Testing
- Thermal Stability Chemistry
 - Reaction Hazard Screen Tools
 - Adiabatic Calorimeter
 - Powder Thermal Stability
- Fire
 - i-Cal (Fire Calorimeter)

Electrostatic Equipment (JCI)

- Laboratory Equipment
 - Electric Field Meter / Volt Meter
 - Charge Relaxation Time
 - Charge Measurement
- Field Test Equipment
 - Lightning Warning
 - Adverse Conditions Equipment

Regulatory (Systems)

- Physical or Chemical Properties Measurement