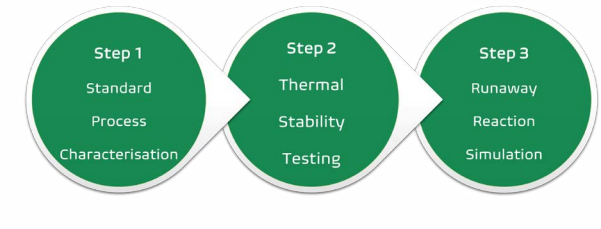
Chemical Reaction Hazards

The 3 Key Areas of Process Safety Testing

The understanding of chemical reactions and material reactivity is a critical element of safe processing.

When working with any manufacturing process it is always necessary to establish the hazards associated with its operation. The identification, assessment and characterisation of both intended and, more importantly, unintended exothermic reactions, are critical for ensuring the safe scale-up and operation of a chemical process. DEKRA are world leaders in Process Safety and this datasheet provides an overview of the key tests we undertake in a laboratory setting to ensure the safety and protection of personnel and plant from runaway reactions.

The DEKRA 3 Step Assessment Procedure



Step 1 Standard Process Characterisation

Characterisation is key for scaling up any exothermic process. Equipment to cool the exotherm and data on the cooling capacity required (cooling jacket, reflux condenser etc.) is essential.

RC1 Reaction Calorimeter

A simulation of the controlled plant process. Essential data produced during testing for scaling up exothermic / gas generating reactions includes;

- Heat of reaction
- Accumulation (maximum remaining energy if cooling is lost, e.g. immediately after completion of an addition)
- Heat capacity
- Gas evolution
- Predicted adiabatic T rise (maximum achieveable temperature for the reaction)
- Criticality Class (if thermal stability data is also available, e.g ARC)

Step 2 Thermal Stability Testing

DEKRA uses 3 thermal screening techniques, which is used depends on the data and answers required.

Differential Scanning Calorimeter (DSC)

This test provides the thermal stability trace of raw materials, intermediates or products and is very good at establishing decomposition energy. It uses a small mg sample size, so great when there isn't much sample availability. Interpretation of the evaluations can sometimes be challenging, analysis of exothermic or endothermic events rely on getting a stable baseline and can be ambiguous. DSC can provide accurate heat of decomposition values.

Carius Tube Test

This test is not generally for performing reactions but a very economic way to study raw materials, intermediates and products. The test looks at the decomposition profile and defines the onset and severity of any reactions as well as measuring gas evolution. This test differs from the DSC in that it measures pressure (vapour and gas). The decomposition energy is not accurate, but it gives a good general profile of thermal events.

Accelerating Rate Calorimeter (ARC) Test

The ARC provides a decomposition profile for raw materials, intermediates or products. The ARC works on a Heat/ Wait/ Search method, whereas the Carius is on a constant ramp, the ARC is programmable to search for the exothermic event and its sensitivity is very good, typically 0.02K/min sensitivity. When the onset of an exothermic event is detected, it switches into adiabatic mode and tracks the sample producing an exotherm profile.

Uses for ARC analysis

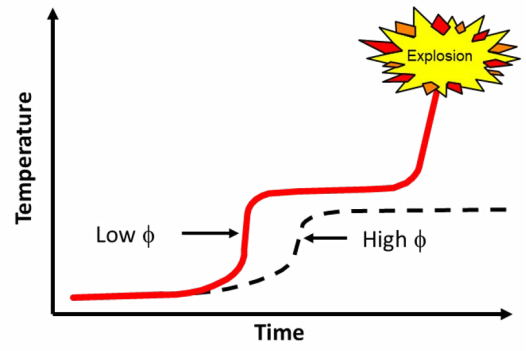
- Gathering data on the exothermic onset and severity of the decomposition
- TMR (Time to Maximum Rate) calculation from any temperature and T_{D24} (a common maximum allowable operating temperature used by industry)
- Self-accelerating decomposition temperature (SADT) estimation (used for material transport classification as well as in normal plant processes to assess long-term stability of materials)
- Uses pressure measurement to garner gas evolution data. Cm^3 of gas production per gram of sample can then be calculated

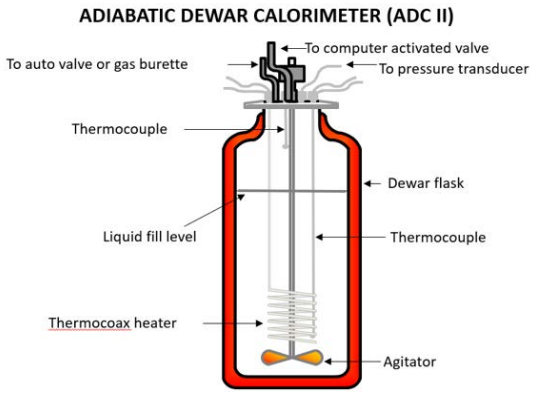
Step 2 Runaway Reaction Simulation

An exothermic reaction can lead to thermal runaway, which begins when the heat produced by the reaction exceeds the heat removed. DEKRA use 2 main tests to analyse runaway reactions; the Adiabatic Dewar Test (ACD II) and Adiabatic Vent Sizing Package (VSP) Test.

Phi Factor Definition

This defines heat loss to the reactor vessel, i.e. the heat absorbed by the vessel that is removing energy from the system. It's important to know the Phi Factor of any process testing and understand it changes based on the vessel size. Phi has an influence on the temperature and pressure rates seen in process chemistry. A laboratory glassware test may show significantly less severe conditions than a large plant reactor.





Adiabatic Dewar Test (ADC II)

In this test, large-scale plant vessels are mimicked in small scale using the adiabatic Dewar calorimeter, a 1L stainless steel vessel with a vacuum jacket. The vacuum means minimal heat is lost from the reaction and the exothermic energy heats the sample as it would in situ in a plant vessel. This instrument is good for mechanical agitation meaning it is better when testing highly viscous materials.

This low phi test is used to establish the worst-case scenario so our Consultants can then form vent sizing calculations on the data to establish TMR data, TD24 data and gas evolution. You generally don't need to correct this data as you would with ARC, Carius or DSC as it is already simulating a larger scale vessel up to about 25M^3. For vessels larger than 25M^3, a phi correction calculation would be needed on the data for it to be accurate.

Adiabatic Vent Sizing Package (VSP) Test

In principle, this test gives the same data as the Dewar test mentioned above, just via a different method. A 110ml vessel inside a chamber with a heating element on the outside is used. An advantage of this test in comparison to the Dewar test is pressure equalisation with outer vessel so tests at much higher pressures can be carried out (typically 70-100barg).

Learn More

Read more on DEKRA's Testing Services here

DEKRA provide an in-depth 2-day training course on **Chemical Reaction Hazards (CRH) & Safe Scale-Up**. Find more information and registration here

DEKRA Organisational & Process Safety

DEKRA Organisational and Process Safety are a behavioural change and process safety consultancy company. Working in collaboration with our clients, our approach is to assess the process safety and influence the safety culture with the aim of 'making a difference´.

In terms of behavioural change, we deliver the skills, methods, and motivation to change leadership attitudes, behaviours and decision-making among employees; supporting our clients in creating a culture of care and measurable sustainable improvement of safety outcomes is our goal.

The breadth and depth of expertise in process safety makes us globally recognised specialists and trusted advisors. We help our clients to understand and evaluate their risks, and work together to develop pragmatic solutions. Our value-adding and practical approach integrates specialist process safety management, engineering and testing. We seek to educate and grow client competence to vide sustainable performance improvement; partnering with our clients we combine technical expertise with a passion for life preservation, harm reduction and asset protection.

We are a service unit of DEKRA SE, a global leader in safety since 1925 with over 45,000 employees in 60 countries and 5 continent. As a part of the world's leading expert organisation DEKRA, we are the global partner for a safe world.

We have offices throughout North America, Europe, and Asia.
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