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THERMAL STABILITY TESTING **Datasheet**

Do you know the thermal limits of your materials? Almost all materials will undergo some form of thermal oxidation, decomposition or self-reaction at elevated temperatures. It is therefore a prerequisite of safe chemical manufacture and processing that the thermal stability limits of the materials are understood when working at elevated temperatures.

WHAT ARE THERMAL OXIDATION, DECOMPOSITION AND SELF-REACTION?

A material can undergo one (or more) of the following thermal transformations when it is heated:

- Oxidise (i.e. react with oxygen in the atmosphere and start to glow, inflame or self-heat)
- Decompose (i.e. molecules fragment into smaller molecules, with the evolution of heat)
- Self-react (i.e. molecules react with one another to evolve heat)

The degree to which these events occur can be affected by process methods. Therefore, when assessing the thermal stability of a material, it is important to consider process conditions especially when working at elevated temperatures or when considering bulk storage after a drying operation. The selection of a suitable thermal stability test will depend greatly on these process factors.

WHY ISN'T ONE TEST METHOD GOOD ENOUGH?

Decomposition and self-reaction are generally assessed using the same tools. Oxidation requires the use of specific, tailored tests to simulate the specific conditions of air availability and geometry – this is especially true for finely divided solids (dusts and powders). A study was performed by Chilworth Technology to assess the thermal stability of four powders using 'small scale' and powder specific methods. The comparison of onset temperatures (°C) for two of the materials studied, citrus peel and azodicarbonamide provides a dramatic demonstration of the effect that the test method can have on the detected onset temperature.

THERMAL STABILITY TEST RESULTS FOR DRIED CITRUS PEEL AND AZODICARBONAMIDE

Test	**Citrus Peel (°C)**	**Azodicarbonamide (°C)**
Carius Tube	115	145
Differential Scanning Calorimetry (in air)	223	179
Differential Scanning Calorimetry (in nitrogen)	>250	179
Accelerating Rate Calorimetry (ARC)	165	135
Diffusion Cell Test	114	169
Aerated Cell Test	101	168

For Azodicarbonamide, the onset temperatures are relatively closely grouped (within the expected sensitivity of the test methods). This substance is known to thermally decompose (i.e. is unaffected by the environment). For citrus peel on the other hand, the substance oxidises in air at moderate temperatures. This activity goes undetected (or poorly detected) in the closed cell tests and is only well characterised in tests in which air is available (Diffusion Cell Test and Aerated Cell Test).

If a decision on the safe drying temperature in air was made using Accelerating Rate Calorimetry data, normally a very sensitive technique used for the study of chemical systems in closed environments, then it is possible that potentially hazardous manufacturing could have been specified. This demonstrates the importance of selecting the correct test method for the processing environment employed.

TESTING FOR THERMAL STABILITY

A flow diagram for test selection is provided in Figure 1. The thermal stability of all materials exposed to elevated temperatures must be known to assure safe handling conditions. This includes raw materials, intermediates (including reaction mixtures) and final products.

If a material either has a low melting point, is only exposed to elevated temperatures under an inert (or vacuum) atmosphere, or is present as a solution or solvent based mixture, then 'small scale' thermal stability tests are appropriate. This will include (in increasing order of sensitivity):

- Differential Scanning Calorimetry (DSC)
- Differential Thermal Analysis (DTA, e.g. the Carius Tube technique)
- Accelerating Rate Calorimetry (ARC)
- Adiabatic Calorimetry (e.g. the Adiabatic Dewar Calorimeter)

The first two tests (DSC / DTA) are employed to determine, crudely, the onset temperature and magnitude of any exothermic (energy releasing) events. Considerable safety factors are applied to these crude tests (typically, between 50 and 100°C to the onset of an event). More sensitive tests are performed if the predicted onset temperature (minus safety factor) is close to a maximum possible process or exposure temperature.

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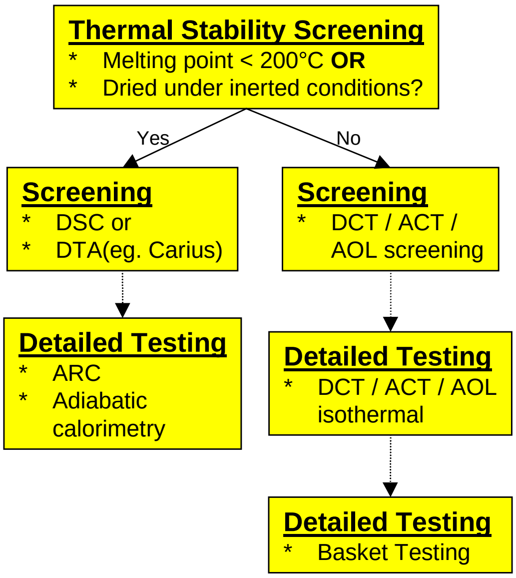

Figure 1.

For materials exposed to air at elevated temperatures (e.g. for powder drying or large scale storage applications), particular tests are performed which are designed to simulate specific exposure conditions. These tests include:

- Diffusion Cell Test (DCT). A test in which natural diffusion of air is permitted around and through the sample during a temperature ramped or isothermal test. This test simulates bulk powder situations such as tray dryers and the base of spray dryers.
- Aerated Cell Test (ACT). A test in which air is forced through the sample during a temperature ramped or isothermal test. This test simulates fluid bed drying and tumble type dryers.
- Air Over Layer Test (AOL). A test in which pre-heated air is circulated over the sample during a temperature ramped or isothermal test. This test specifically simulates the tops of spray dryers, flash dryers or conditions where thin layers exist.
- Basket Tests. The ultimate thermal stability test for powders is the Basket Test series. Onset conditions are measured for a minimum of three different small scale basket sizes allowing the extrapolation of safe drying or storage conditions for any size and shape of powder accumulation.

What Chilworth Can Provide

Our specially equipped laboratories and professional consulting staff can help you to select the most appropriate test (or tests) for any specific application. We will tailor a package of tests to define safe storage, drying or handling temperatures for any material (liquids, solids or mixtures). We will ensure that no unnecessary tests are performed and provide you with a complete technical report which outlines the rationale behind each test, the safety margins that should be applied and concludes with recommendations regarding safe storage, handling or drying conditions.

As well as providing process safety data, we also have test equipment available for the provision of transportation information including testing for UN Class 4, divisions 4.1 and 4.2 (pyrophoric, self-reactive and self-heating substances and determination of self-accelerating decomposition temperature).

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***My particular interests are**:-*

Chemical Reaction Hazards... ☐ Hazardous Area Classification...................................... ☐

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I would like a free talk to a consultant about a process safety matter... ☐

I would like a free visit from a consultant next time one is in my area... ☐

For further information phone Systems at Chilworth Technology on +44 (0)23 8076 0722

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