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Thermal Stability: A Review of Methods and Interpretation of Data

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Abstract:

The assessment and understanding of thermal instability associated with process materials is a prerequisite for safe manufacturing. This is particularly true for materials exposed to elevated temperatures during processing. Information gathering on thermal stability should commence as early as possible in the process development lifecycle such that, initially, highly energetic substances can be identified and handled accordingly. Further studies to accurately define safe exposure temperatures should then be performed using equipment and methods specifically appropriate to the plant or processing technique selected, or envisaged, for scale-up. A simplified procedure for thermal stability assessment is proposed which takes into account decomposition and oxidation activity which are both commonly observed with organic substances. The strengths and weaknesses of individual test methods are reviewed, and information is provided on correct application of the resulting data.

Introduction

A wide variety of techniques are available for assessment of the thermal stability hazards of process materials. It is a prerequisite of safe manufacture that the thermal conditions under which a material or mixture will degrade are well defined. Often, decomposition of a material will impact only on quality. However, in many other cases, a decomposition may pose a significant hazard by virtue of gas generation, inflammation, or in extreme cases, rapid deflagration/detonation.

This report appraises the methods most commonly employed for thermal stability assessment for different operating environments and provides general guidance on the selection of test methods and interpretation of data.

Experimental thermal stability test methods can be grouped as follows: (i) small scale thermal screening techniques (DSC/DTA/TGA), (ii) adiabatic calorimetry techniques, and (iii) test methods specifically developed for powders.

Each group of methods (together with preliminary desk screening methods) is discussed with examples of test data obtained and conclusions that can be drawn. A definitive strategy for assessing thermal stability is proposed. This report does not cover (in detail) regulatory or transportation classification (although some of the test methods described are used for this purpose). In addition, this article is presented for use by organisations who are not specifically involved in munitions, explosives, or pyrotechnics manufacture. Special legislation and provisions exist for the manufacture, handling, and storage of such materials that are not within the scope of this article. In addition, it should be recognised that this article is not intended to provide full details of the experimental methods employed (although references are provided for that purpose). The intention is to provide an overview of the data that should be obtained and, more importantly, how the data should be applied when assessing the safety of a process.

Thermal instability generally arises through one of two mechanisms: (i) molecular decomposition or self-reaction or (ii) reaction of the molecule with the surrounding environment [in practice, this case generally refers to oxidation (burning) events].

Most thermal analysis performed on materials tends to focus on the potential for decomposition/self-reaction. However, for large-scale processing, the potential for oxidation (when drying, storing, or transporting a material) requires the application of specific test methodologies. This article is intended to provide a balanced overview of the assessment procedure for both reaction mechanisms. Data are provided which clearly demonstrate how application of the wrong test method can give rise to erroneous results. Interactions between process materials (either through desired or undesired chemical reactions) are not specifically considered within this article. However, procedures to assess the thermal properties of mixtures should clearly be in place when scaling up a chemical process. This article is not designed to review the theory of thermal explosions. It is intended as a "hands-on" guide to what tests should be performed and when and how the data should be interpreted.

Desk Screening Methods

Prior to undertaking any laboratory studies on a new substance, a significant amount of data can be obtained on the likely thermal stability characteristics of the material. An examination of the molecule for highly energetic functional groups should be the starting point for any such evaluation. Table 1 provides a nonexhaustive list of functional groups which are known to impart thermal instability within a molecule.[1]

In addition numerous thermochemical calculation techniques are available for predicting exothermic decomposition potential (one such commonly used method is CHETAH

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(1) Grewer, T. *Thermal Hazards of Chemical Reactions*; Elsevier: New York, 1994.

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Table 1. **Highly Energetic Functional Groups**

name/structure[a]	range of decomposition energies[b] ($kJ\cdot mol^{-1}$)
alkenes ($R_2C{=}CR_2$)	50 → 90
alkynes/acetylenes ($R{-}C{\equiv}C{-}R$)	120 → 170
epoxides	70 → 100
organic/inorganic peroxides/hydroperoxides ($R{-}O{-}O{-}R/R{-}O{-}O{-}H$)	230 → 360
peracids ($R{-}CO{-}O{-}O{-}H$)	240 → 290
organic sulfoxides ($R_2S{=}O$)	40 → 70
organic sulfonyl chlorides ($R{-}SO_2Cl$)	50 → 70
hydrazines ($R{-}NH{-}NH{-}R$)	70 → 90
diazo/diazonium ($R{-}N{=}N{-}R/R{-}N{\equiv}N^+$)	100 → 180
azides ($R{-}N_3$)	200 → 240
oxime ($R_2C{=}NOH$)	110 → 140
N-oxides ($R_2N{:}O$)	100 → 130
nitroso ($R_2C{-}N{=}O$)	150 → 290
isocyanate ($R{-}N{=}C{=}O$)	50 → 75
nitro ($R_3C{-}NO_2$)	310 → 360
N-nitro ($R_2N{-}NO_2$)	400 → 430
acyl nitrates ($-O{-}NO_2$)	400 → 480

[a] R, in most cases, represents an organic fragment. [b] Not exhaustive.

(published by ASTM)[2]). In general terms, the heat of formation of a compound provides a strong indication of whether exothermic decomposition potential exists (highly endothermic compounds are almost always found to possess energetic decomposition potential).

The scientific literature should always be interrogated to identify whether data on the compound, or a close analogue, already exists.[3−7]

Desk screening should always provide an indication of whether energetic decomposition potential may exist. In some cases, a material for which no hazards are predicted may be found in practice to present a significant hazard. In almost all cases, the desk screening exercise is unlikely to yield accurate information on the thermal conditions under which activity may occur. For this reason, small-scale thermal screening is generally always required on materials for which thermal stability data does not exist.

Small-Scale Thermal Screening Methods

The strategy for any planned testing regime should always start small and increase in scale (since only small quantities may be available at early-stage process development and, more importantly, any unexpectedly energetic activity is likely to have lesser consequences on a smaller scale). A proposed procedure for assessing the thermal instability hazards of a material is provided in Figure 1. This is an intentionally condensed procedure although following this route will generally provide satisfactory data for defining thermal limits for a material-handling process. The procedure aims to: (i) screen for highly energetic activity, (ii) define thermal decomposition limits through small-scale screening, and then (iii) more accurately define thermal decomposition limits using more sensitive and appropriate test apparatus.

Three types of test methods are commonly employed for preliminary investigation of thermal stability:[8,9]

(i) Differential thermal analysis (DTA): the sample is (usually) ramped at a pre-set rate and the sample temperature monitored with respect to a reference temperature. Methods include the commercially available Carius tube and RADEX systems. The test systems are usually enclosed such that coincident pressure measurement is permitted.

(ii) Differential scanning calorimetry (DSC): the sample is (usually) ramped at a pre-set rate. Heat flow from the sample is either measured through power compensation applied to an inert and independent reference cell (power compensation DSC) or through thermal resistance measurements made between the coupled sample and reference cells (heat flow DSC). The tests can be performed in sealed or open pans.

(iii) Thermogravimetric analysis (TGA): the sample is (usually) ramped at a pre-defined rate whilst accurately recording its mass. The test is not commonly employed for thermal stability safety applications and is most commonly employed for mechanistic elucidation studies.

When assessing the test data from these methods, a number of important test criteria should be examined:

(1) Ramping Rate. Optimum sensitivity is provided by lower ramp rates ($0.5{-}2\ K\cdot min^{-1}$).

(2) Sample Mass. Potential for unrepresentative sampling exists for small-scale tests such as DSC (where typically 1−20 mg of material are tested).

(3) The Range of Test. The test must cover the potential range of temperatures (including applicable ΔT_{safety}) which may be encountered during plant processing.

(4) Materials of Construction and Potential Contaminants. It is often important that the thermal stability of the material is assessed not just in pure form but also in the presence of materials of plant construction and potential, foreseeable contaminants.

(5) Test Cell Configuration. Open-cell tests can yield potentially misleading results since exothermic events may be masked by endothermic phase transitions (e.g., boiling/evaporation of volatile components). Closed-cell tests are generally preferred for preliminary thermal stability analysis.

The DSC is advantageous in that it provides a direct measure of decomposition energy (this is not obtained in DTA). However, DTA methods generally employ larger samples, thus giving greater detected onset sensitivity, and normally afford coincident pressure measurement (indicating

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(2) *The ASTM Computer Program for Chemical Thermodynamic and Energy Release Evaluation*; ASTM Data Series DS51C, version 7.3; American Society for Testing and Materials: Philadelphia, PA, 2002.

(3) Bretherick, L. *Handbook of Reactive Chemical Hazards*, 6th ed.; Butterworth-Heinemann: Oxford, 2000.

(4) Sax, N. I. *Dangerous Properties of Industrial Materials*, 9th ed.; Reinhold Publishing Corporation: New York, 1996.

(5) Lewis, R. J. *Hazardous Chemicals Desk Reference*, 3rd ed.; Van Nostrand Reinhold: New York, 1993.

(6) Davis, D. J.; Davis, J. A. *Hazardous Materials Reference Book*; Van Nostrand Reinhold: New York, 1996.

(7) Carson, P. A.; Mumford, C. J. *Hazardous Chemicals Handbook*; Butterworth-Heinemann: Oxford, 1994.

(8) Cronin, J. L.; Nolan, P. F. The Comparative Sensitivity of Test Methods for Determining Initial Exotherm Temperatures in Thermal Decompositions of Single Substances. *J. Haz. Mater.* **1987**, *14*, 293−307.

(9) van der Plaats, G. *The Practice of Thermal Analysis*; Mettler Toledo AG: Schwerzenbach, Switzerland, 1992.

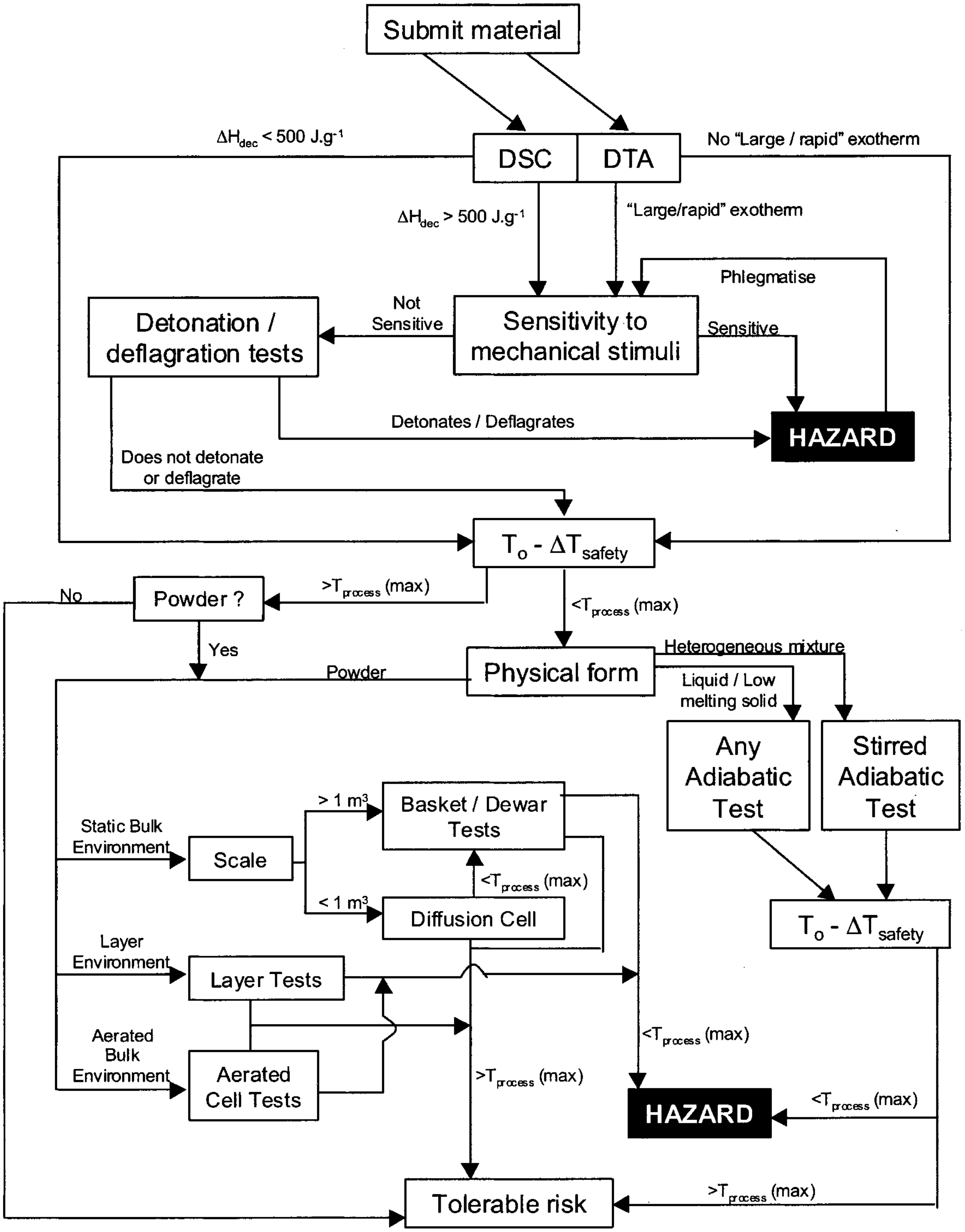

Figure 1. **Strategy for assessing thermal stability. The terms are: T_o = the detectable onset temperature of an event from a specific test. It should be noted that the "onset" temperature for an event is not a constant. It is totally dependent on the detection limits of the specific test. For this reason, in all but the most sensitive tests, considerable safety factors are required. ΔT_{safety} = the safety margin appropriate for the specific test. $T_{process}(max)$ = the maximum temperature to which the substance may potentially be exposed during a specific stage of processing.**

the occurrence of a low energy transformation which may liberate significant permanent gas).

Before progressing to larger-scale test methods, it is necessary to interrogate the small-scale DSC or DTA data obtained on a material. For DSC, this generally entails examination of the onset conditions of any activity as well as an examination of the observed decomposition energy. As depicted in Figure 1, a material with a decomposition energy of >500 J•g^{-1} should be subjected to further small-scale testing to determine if the material is sensitive to mechanical ignition sources (i.e., friction and impact) and, if not, whether the material undergoes rapid deflagration or detonation when heated under confinement or subject to a high-energy initiating source.

Defining potentially hazardous properties from DTA data is usually a much more subjective exercise. It can be loosely

Table 2. **Safety factors proposed for application to thermal stability test data**

test method	mode of test	safety factor
DSC	ramped (>5 k.min^{-1})	100 K
	ramped (<5 k.min^{-1})	75 K
Carius tube/radex (DTA)	ramped (>5 k.min^{-1})	75 K
	ramped (<5 k.min^{-1})	50 K
ARC	heat-wait-search	30 K
adiabatic, low ϕ factor method	heat-wait-search	10 K
bulk powder diffusion cell test[a]	isothermal	50 K
bulk powder aerated cell test[a]	isothermal	30 K
air over layer test	isothermal	20 K
basket tests[b]	isothermal	10% on extrapolated onset

[a] Applicable up to 1 m^3 powder volume only. [b] Applicable to all scales of handling and storage.

defined that if a material undergoes “rapid” or “large” exothermic activity in a DTA test, further explosivity testing should be progressed prior to larger-scale thermal stability analysis. Formal procedures for explosivity testing are provided in the United Nations *Recommendations on the Transport of Dangerous Goods. Manual of Tests and Criteria*.[10]

If a material is sensitive to mechanical stimulation or presents a significant detonation/deflagration hazard, consideration should be given to whether scale-up should proceed with that specific material. Only if confidence exists that such hazards can be reliably controlled on scale-up should further testing proceed. One approach to reducing any such hazards is to phlegmatise (dilute) the material with an inert and compatible diluent. Again, however, care must be taken to confirm that the phlegmatising agent remains intimately mixed (in the correct proportions) with the material. Use of a volatile component that will readily evaporate, for example, would only provide limited mitigation of hazardous properties.

Assuming that a material passes through the initial screening procedure, an analysis of decomposition onset temperatures should be performed. By definition, small-scale screening tests are usually high heat-loss methods and not adiabatic. Significant safety factors must therefore be applied to the measured T_o. Application of the appropriate ΔT_{safety} is, again, a rather subjective area of thermal stability testing with few published literature sources available for specific guidance. The main reason for this lack of published recommendation is the inherent variability in the mechanism and kinetics of reactions (this is discussed in greater detail in a work by Hofelich[11]). However, conservative safety factors, which may be considered for all tests discussed in this report, are presented in Table 2. These factors are intended to be applied to correctly specified and conducted tests (e.g., appropriate sample size employed, appropriate mode of test, etc.) but may require review when autocatalytic events are observed or suspected.

Once the small-scale screening has been completed, there are two approaches which can be taken: (i) define the maximum allowable sample exposure temperature through further testing and (ii) define the maximum potential processing temperature ($T_{process}$(max)) from examination of the large-scale plant in which the material is likely to be processed and compare with the onset of activity (minus safety margin) obtained in the screening test.

Option (ii) is not always known (particularly at the research and development stage of a new process). However, if this option can be followed, a significant reduction in the test program can be achieved.

For materials that may be handled as powders, irrespective of whether events are detected in the DSC or DTA screening tests, some form of powder thermal stability test is strongly recommended. All too often, the results of DSC or DTA tests are considered to be sufficient information on the thermal stability of a material. This is simply not the case since oxidative events may occur which are not observed in the contained and oxygen-starved environment of the small-scale tests. Few companies possess the relatively simple equipment required to perform specific powder tests or, in fact, consider the necessity for tests of this type.

For liquids (or solids with a melting point below $T_{process}$(max)), if it is seen that $T_o - \Delta T_{safety}$ is significantly above $T_{process}$(max), then further testing is not usually required—the risk posed by the material is considered to be tolerable under the specific processing conditions. However, if this is not the case (i.e., if $(T_o - \Delta T_{safety}) < T_{process}$(max)), then some form of adiabatic calorimetry is required to more sensitively assess the thermal stability of the material.

Adiabatic Calorimetry Data

Adiabatic calorimetry provides a heat loss environment which closely approximates that which exists on the large scale (i.e., approaching zero). Accordingly, this type of test allows safety factors to be reduced considerably (see Table 2). Systems available for conducting adiabatic studies on liquids or low-melting solids include:

- Small-scale adiabatic calorimeters (e.g., accelerating rate calorimetry (ARC),[12] advanced reactive system screening tool (ARSST)[13]). Small-scale calorimeters typically employ test cells of small volume (ca. 10 cm^3) and are usually adiabatic with ϕ factors (thermal inertia) of typically in the range 1.2 to 3. The often large ϕ factor compromises the direct application of the data and infers the need for a fairly substantial safety margin (see Table 2). (The ϕ factor expresses the ratio of the heat capacity of the sample plus that of the test cell to that of the sample only. For large-scale vessels, this value usually approaches 1.0, and test systems should ideally replicate this situation (i.e., minimal effect of the test cell).

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(10) *Recommendations on the Transport of Dangerous Goods. Manual of Tests and Criteria,* 3rd revised ed.; United Nations, Committee of Experts on the Transport of Dangerous Goods; United Nations: New York, 1999.

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(12) Townsend, D. I.; Tou, J. C. Thermal Hazard Evaluation by an Accelerating Rate Calorimeter. *Thermochim. Acta* **1980**, *37*, 1−30.

(13) Fauske, H. K. *The Reactive System Screening Tool (RSST): An Easy, Inexpensive Approach to the DIERS Procedure*; International Symposium on Runaway Reactions, DIERS User Group, AIChE, New Orleans, March 1998; American Institute of Chemical Engineers: New York, NY, 1998; pp 51−63.

- Adiabatic Dewar calorimetry (using metal-sealed or glass open Dewar vessels[14]). These large-scale methods usually provide a low ϕ factor environment (less than 1.3). Open (glass) or sealed (metal) systems can be employed for highly sensitive tracking of low-rate (and high-rate) adiabatic events. The systems can be agitated with (or without) pressure measurement (as required). Vessel sizes can vary from 250 cm^3 up to 2 L.
- Pressure compensated adiabatic calorimeters (e.g., VSP II,[15] APTAC,[16] Phi Tec II[17]). As the name suggests, these systems use fairly weak sample containers but maintain the integrity of the cells by placing the cells in a high-pressure containment system, the pressure of which is controlled to remain essentially the same as that within the sample container. Maintaining a low differential pressure allows thin-walled (low ϕ factor) cells to be used. The systems can be agitated (normally with magnetically coupled stirrers) with, or without, pressure measurement as required. Vessel sizes are normally around 100 cm^3.

Sensitive tests of this type are usually performed in heat−wait−search or isothermal mode whereby the environment around the sample accurately tracks the sample temperature. The detection limits of each test method are usually around 1.0 $K{\cdot}h^{-1}$, although the Dewar methods can be lower. The use of low ϕ factor apparatus normally yields results which can be directly applied with minimal safety margins. Where the ϕ factor is low kinetic data is likely to be valid without mathematical correction or uncertainty.

Tests of this type can be performed on liquids, agitated heterogeneous mixtures, and solids. In the case of solids, the most appropriate methods are ARC, ARSST, and glass Dewar calorimetry. When testing solids, however, these methods will only detect self-reaction or decomposition. Combustion/oxidation events will not be satisfactorily monitored. For solids, the limiting event, that is, the event which shows the lowest onset temperature, should be studied to determine the thermal limits of the material.

In addition to these purely adiabatic test methods, there are a number of derivative tests which are well described in the United Nations *Recommendations on the Transport of Dangerous Goods. Manual of Tests and Criteria*.[10] These tests (including the heat accumulation storage test, adiabatic storage test, and isothermal storage test) are used in combination with vessel/package heat loss data to provide information on the onset of thermal activity under the prevailing heat loss conditions of processing/storing. These methods are commonly used in the determination of the self-accelerating decomposition temperature (SADT) for given package or vessel sizes and are used predominantly for transportation classification.

Table 3. **Powder test methods and applicability**

test name	applicable plant situations
diffusion cell test	bulk drying/storage (up to 1 m^3) rotary drying slumped fluid bed or spray drier bed tray drying (thick layers >30 mm)
aerated cell test	fluid bed drying
air over layer test	band drying tray drying (thin layers <30 mm) spray/pneumatic conveying (flash) drying layer deposits in/on equipment
basket tests	bulk storage (any scale) bulk drying (any scale)

Thermal Stability Tests Developed for Powders

Almost all pharmaceutical and fine chemical final products are powders. In most cases, these will be dried at elevated temperature in a potentially wide variety of drying equipment. Powders are generally produced with a small particle size to improve and facilitate handling and conveying. This small particle size produces a very large surface area-to-volume ratio which will promote any oxidative events.

Although some drying takes place under an inert or vacuum atmosphere, other operations are performed in an air environment where oxidative events may become possible. Even if the drying operation itself is performed under an inert atmosphere, discharge of hot powder, storage of hot/warm powder, or inadvertent loss of the inert atmosphere during drying needs to be considered so that maximum safe exposure temperatures can be identified. This can only be achieved by using the appropriate test methodology.

In most cases, conservative drying temperatures are selected for pharmaceuticals and fine chemicals. However, from conducting appropriate tests, it may be possible to increase drying temperatures and achieve greater throughput and productivity.

Most test methods devised specifically for powders are based on ensuring that the oxygen availability of the plant-scale operation is reflected in the laboratory test. Table 3 highlights the available test methods and plant procedures to which they would relate.

Further information on the design of the tests is provided in a publication by Abbott.[18] In summary:

- The Diffusion Cell test utilises a cylindrical test cell (volume ca. 150 cm^3) with a sintered glass base. The open cell is filled with powder and equipped with four temperature-measuring thermocouples positioned throughout the bulk of material. Air is allowed to diffuse naturally through the powder.

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(14) Rowe, S. M.; Middle, K. V. *Advances in Adiabatic Dewar Calorimetry*; International Conference on Runaway Reactions, St. Petersburg, Russia, June, 1999.

(15) *DIERS Reports: Bench Scale ERS Sizing Tools: Equipment Details and Test Procedures*; American Institute of Chemical Engineers: New York, NY, 1984.

(16) Chippett, S.; Ralbovsky, P.; Granville, R. *The APTAC: A High Pressure, Low Thermal Inertia, Adiabatic Calorimeter*; International Symposium on Runaway Reactions, DIERS User Group, AIChE, New Orleans, March 1998; American Institute of Chemical Engineers: New York, NY, 1998; pp 81−108.

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(18) Abbott, J. A. *Prevention of Fires and Explosions in Dryers: A User Guide*, 2nd ed.; Institution of Chemical Engineers: Rugby, UK, 1990.

Table 4: **Test results for thermal stability using different techniques**

test	ramp rate ($K{\cdot}min^{-1}$)	milk powder (skimmed) onset (°C)	citrus peel (dried) onset (°C)	azodicarbonamide onset (°C)	sodium perborate (monohydrate) onset (°C)
DSC[a] (air)	0.5	214	223	179	106
DSC (N_2)	0.5	>350	>350	179	114
Carius tube[b]	0.5	106	115	146	89
ARC[c]	H−W−S	85	165	135	90
diffusion cell	0.5	108	114	169	N/A
aerated cell	0.5	94	101	168	N/A

[a] DSC tests performed using Perkin-Elmer DSC-7 (open aluminium pans) under both air and nitrogen initial atmospheres. [b] Carius tube is a sealed DTA method using a 35 cm^3 glass tube sealed to a pressure measurement system. Sample is charged to a fill level of ca. 30%. [c] Columbia Scientific ARC apparatus (low ϕ titanium bombs) used in heat−wait−search (H−W−S) mode with 5 K heating steps and a detection threshold of 0.02 $K{\cdot}min^{-1}$.

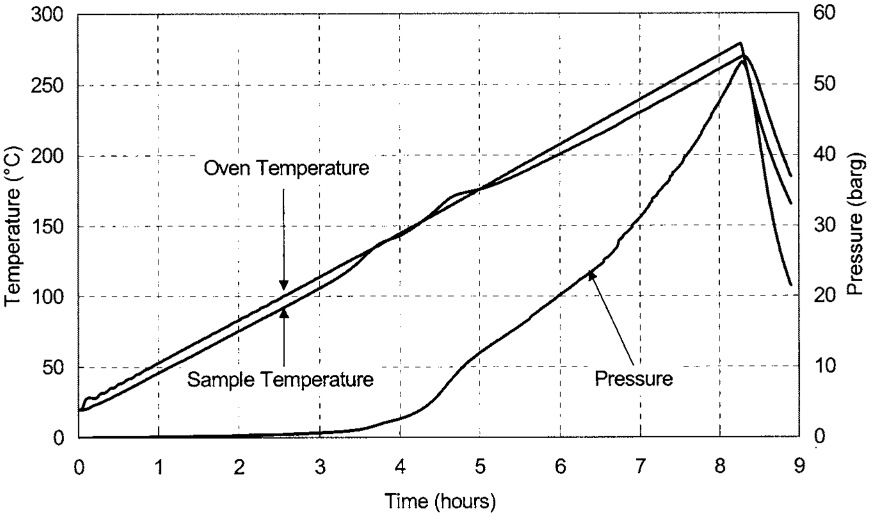

Figure 2. **Carius tube screening test data for skimmed milk powder.**

- The Aerated Cell test is performed using the same equipment except that a closure is placed on the Diffusion cell and a flow of preheated air (0.6 $L{\cdot}min^{-1}$) is continually purged through the sample for the duration of the test.
- The Air Over Layer test, as the name implies, uses a thin layer of material (normally 15 mm deep) over which preheated air is continually purged.

In all of these tests, an initial temperature-ramped test is usually followed by a series of isothermal tests to provide optimum onset sensitivity and simulate process exposure times.

The results from the diffusion cell and aerated cell tests can be applied to powder masses of up to 1000 kg. Above this, more sensitive methods for extrapolating safe conditions are required, the most sensitive of which is the basket test method. In essence, ignition/no-ignition boundary conditions of the powder are closely defined in a range of different cubic baskets (usually, 15, 125, and 1000 cm^3). A plot of log(volume/surface area) versus 1/temperature should provide a straight line which can be extrapolated to large bulks and any shape of container. This allows accurate prediction of maximum allowable storage temperature conditions.

When studying any powder, potential large-scale handling and drying conditions should be examined to identify the most appropriate test method.

An unpublished study performed by Chilworth Technology clearly demonstrates the criticality of assessing the oxidation potential of powders. The thermal stability of four powders was assessed using conventional closed-cell techniques and specific powder thermal stability techniques. The results (Table 4) produce a startling demonstration of the value of powder-specific tests.

For the decomposing substances (azodicarbonamide and sodium perborate), the range of onset temperatures determined is relatively narrow (135−179 and 89−114 °C, respectively). In contrast, for the oxidisable citrus peel and milk powder materials, the range of onset temperatures varies significantly (101 → 350 and 85 → 350 °C, respectively).

Some of the sealed tests are seen to be effective at determining the onset temperature even for the oxidisable substances. These tend to be larger-scale tests in which the fill volume is relatively low (e.g., in the Carius tube test). This provides a significant amount of air to be available to support the onset of the oxidation event (such that the event is detectable). The magnitudes of the events in these tests are seen to be much smaller than those for the powder-specific tests. This is illustrated in Figure 2 where the scan result from the Carius tube test is presented for skimmed milk powder.

If a powder exhibits a low melting point, the powder-specific tests are rarely appropriate as the material tends to seep out of the sample chamber once molten. For such materials, tests for liquids should be applied.

Conclusions

Preventing thermal decomposition or oxidation of raw materials, intermediates, and final products is a prerequisite for any chemical process. A strategic assessment procedure is required such that the thermal limits of all materials are known, and can be safely controlled, during processing. Ideally, these data should be available at the earliest possible opportunity in process development such that safety can be in-built to subsequent scale-up steps.

A simplified procedure has been proposed for the assessment of thermal stability hazards which initially aims to screen materials for highly energetic activity and then moves on to look at accurate definition of maximum safe exposure temperatures. The test method selected for any study must be appropriate for the handling conditions prevailing during plant processing. Most importantly, provision of appropriate tests for materials handled as powders should be made. These specific tests are often overlooked in favour of "classical" thermal methods such as DSC, DTA, or ARC.

When interpreting data from any test, there are a wide variety of factors which should be taken into account including:

- Mode of test. In order of sensitivity, adiabatic, isothermal, heat−wait−search, and screening tests may be performed.
- For ramped tests, the sample ramp rate is critical. Optimum sensitivity is provided by lower ramp rates (0.5−2 $K \cdot min^{-1}$)
- Sample mass. Potential for unrepresentative sampling exists for small-scale tests such as DSC (where typically 1−20 mg of material is tested).
- The range of test. The test must cover the potential range of temperatures (including test safety factors) which may be encountered during plant processing.
- Materials of construction and potential contaminants. It is often important that the thermal stability of the material is assessed not just in pure form but also in the presence of materials of plant construction and possible, foreseeable contaminants.
- Test cell configuration. Using an open-cell test can cause any evaporative losses to mask thermal activity (and will not allow any information on gas generation to be determined). Conversely, oxidative events may not be identified in sealed-cell tests.

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