SAFETY GUIDE

A strategic guide to characterization and understanding
Handling Dusts and Powders Safely
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Chilworth, The backbone of Process Safety within the DEKRA
Risk assessment for the safe handling of dusts and powders can be far more challenging than that of flammable liquids and gases which is often viewed as relatively straightforward. This is generally because the relationship between the operating process Basis Of Safety and the physical properties data for safe handling of dusts and powders are not always fully understood. Historically, there is also a greater awareness amongst operating staff of the risk of fire and explosion from flammable liquids and gases.

Many people are surprised when they first hear that there can be a significant risk of fire and explosion from processing apparently innocuous materials such as powdered sugar, aluminium powder and even aspirin. Process engineers, however, have long been aware of the hazards and risks involved with processing these types of materials and the measures, procedures and training that must be in place in order to operate a safe site. As new materials are developed, engineers must constantly challenge safety assumptions and provide sound risk assessments on how material (and any dust / fines generated during processing or mixing) should be handled and stored. The basis of any risk assessment must be an understanding of the physical properties of the material being handled.

The aim of this guide is to provide a firm foundation to the evaluation of flammable powders and dusts across all industries, with the objective of helping operating companies minimize fire & explosion risk. In the following pages we will develop a strategy for the evaluation of fire hazards, dust explosion hazards and thermal stability hazards associated with these materials.

This methodology can be applied to most foreseeable process situations. The booklet begins by explaining the conditions for a fire and what conditions may arise in the workplace to convert this process into an explosion. We go on to describe what consideration should be given to establishing a safe operating environment by establishing an operating Basis Of Safety and then what safety data is needed to verify and confirm the key assumptions that underpin the chosen Basis Of Safety.

Some of the data required to verify a Basis Of Safety may already exist in the form of Material Safety Data Sheets (MSDS or SDS) or existing test reports at the operating site. If this information is not available, then a suitable strategy needs to be put in place to determine the explosive properties of any materials being handled. One such strategy developed by Chilworth Technology over many years of working with the process industry is outlined within this booklet.

Many of the most common tests referred to in this guide are described in detail within the Appendix of Test Descriptions section at the back of the booklet.

This booklet is published by Chilworth Technology Inc. and written by a team of process safety specialists. Their joint experience provides over 150,000 man-hours of industrial process safety expertise distilled into clear concise guidance on the safe handling of combustible dusts and powders.

Fires in layers of powder, dust explosions and the decomposition of thermally unstable powders can have dramatic and catastrophic consequences when encountered in industrial situations. If not effectively identified and adequately assessed, with the risk either prevented or controlled, these hazardous situations can lead to major loss of containment with resulting impact on the manufacturing company. For example:

- Loss of life or personal injury
- Loss or devaluation of assets
- Negative publicity
- Loss of shareholder confidence and devaluation of the company
- Loss of production capacity and potentially market share, and
- Losses from regulatory authorities / Compensation claims from individuals.

When working with any manufacturing process, it is always necessary to establish the hazards associated with its operation. This is most prominent with issues such as machine guards, tripping or slippery floors etc. but there could also be flammable gases which is often viewed as relatively straightforward. This is generally because the relationship between the operating process Basis Of Safety and the physical properties data for safe handling of dusts and powders are not always fully understood. Historically, there is also a greater awareness amongst operating staff of the risk of fire and explosion from flammable liquids and gases.

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Most people are familiar with the "fire triangle". This encapsulates the message that for a fire to occur, three components must be present simultaneously. The fuel can be a gas (e.g. ethylene), a vapor (e.g. methanol) or a dust (e.g. flour). The oxidant is most usually oxygen in air although other oxidants can be effective in supporting combustion (e.g. chlorine or nitric oxide). The ignition source can be a spark, a naked flame, or elevated temperature (causing "autoignition").

If the fuel is intimately mixed with the oxidant (e.g. in a gas cloud or in a finely divided dust cloud), the rate of combustion increases significantly and the combustion process can be complete within 10’s of milliseconds. This phenomenon is responsible for the marked difference between the burning of a lump of coal and the rapid combustion associated with a coal dust cloud ignition.

If the final ingredient of "confinement" is added to the equation, with intimately mixed fuel and oxidant, then the conditions exist for an explosion – the rapid release of stored energy. Under confined conditions, this can lead to elevated pressures of typically up to 10 bar(g) (although some powders are capable of attaining higher than this).

In a fire, the fuel and the oxidant are separate and need to be present at appropriate concentrations (i.e. within the flammable range) and the ignition source must have sufficient energy to ignite the prevailing fuel / oxidant mixture. The duration of a fire, in an open air environment, is typically dictated by the time taken for the fuel to be consumed (i.e. completely oxidized). In the combustion of solid materials such as coal or wood, the rate of combustion is dictated by diffusion processes which restrict the rate that oxygen can access the remaining fuel. Depending on the size of the fuel, the combustion process can take hours to complete.

The safe processing of flammable materials (dusts, gases or vapors) is facilitated by either preventing the conditions required for an explosion to occur, or protecting the process and personnel from the effects of any such explosion. The combination of measures required to achieve such safe conditions is referred to as the "Basis Of Safety".
Establish a Basis Of Safety

In order to define a suitable Basis Of Safety for any industrial process that may have the potential to produce explosive dust atmospheres, it is necessary to evaluate the flammability of the dust, its potential to form a dust cloud, its sensitivity to ignition from the full range of ignition sources that may exist, its explosion limits and explosion severity. Potential bases of safety that can then be considered for dust explosion risks include:

> **Prevention**
> - Avoidance of the formation of flammable atmospheres
> - Operation outside of the flammable range (normally below the Minimum Explosive Concentration, MEC)
> - Operation in an environment where the oxygen content is below the lowest oxygen concentration necessary to support combustion (‘inerting’)

> **Operation**
> - Avoidance of ignition sources

**Protection**
- Explosion venting
- Explosion suppression
- Explosion containment

The overall strategy for assessing the dust explosion hazards of a powder is summarized in Figure 1. Each of the boxes is described in more detail throughout this section of the guide.

**Figure 1. Strategy for Dust Explosion Testing**

![Diagram of the strategy for dust explosion testing.]

A complete explanation of the figure is given on page 13

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**What do you Already Know?**

When assessing the flammability or thermal stability hazards of any potentially combustible powder, the most appropriate starting point is to evaluate what is already known about the material. Substantial information can be gained from:

> **Material Safety Data Sheet (MSDS or SDS)**

This should include data on physico-chemical properties such as melting point, boiling point, decomposition behavior, vapor pressure, oxidising capability, explosive properties and flammability. However whilst most of the gas and vapor flammability hazards are routinely addressed by the contents of an MSDS, the properties of dust strongly indicate that the material will exhibit thermal instability. In terms of dust explosion hazards, it would be expected that any organic powder (i.e. any powder containing significant carbon and hydrogen) would be potentially flammable.

A strategy for determining dust explosion, fire risk and thermal stability characteristics of powders1 is developed through this guide. The initial starting point is to evaluate whether the powder might exhibit explosive characteristics (i.e. be capable of detonation or rapid deflagration). Any such material clearly requires identification at the earliest possible stage of assessment and be subject to rigorous process safety considerations.

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1. For this document the words "powder" and "dust" are synonymous.
The preliminary evaluation of explosive properties can (and should) be undertaken well before large scale manufacture commences.

A flow diagram for evaluating explosive properties is provided in Figure 2. Initial screening methods are set out in Box 1 and include:

- An examination of the molecular structure looking specifically for functional groups which are known to impart explosive behavior (e.g. nitro-, peroxy-, chlorate-, azide, etc.),
- Thermodynamic calculations and predictions (such as oxygen balance, CHETAH [computer program for Chemical Thermodynamic and Energy Release Evaluation], etc.), and/or
- Small-scale screening of the material.

The small-scale screening need not, in the first instance, be a high precision determination. It is usually sufficient to test a small quantity of material in an ignition tube or on a spatula and rapidly heat the material to qualitatively assess the decomposition potential of the material. In order to protect laboratory personnel and equipment all powder samples submitted to a test laboratory for any type of physical property determination should be subjected to this type of small-scale screening analysis.

Figure 2. A Process Safety Strategy for Powders

1. Explosive Properties Evaluation
   - Functional group examination
   - Small scale screening tests
   - O2 Balance / CHETAH calcs

2. Explosivity Analysis
   - DSC screening
   - UN Sensitivity testing (See page 10)

3. Dust Explosion Analysis
   - See page 13

Fire Analysis
   - See page 19

4. Thermal Stability Analysis
   - See page 21
If a material is shown to exhibit significant explosive properties, this is not necessarily the end of the road for use or synthesis in your company. The following options can be considered:

- Subcontracting synthesis and handling to a specialist third party for the "hazardous" phases of the process,
- Handling the hazardous substance in a sufficiently diluted form so that its explosive properties are reduced to an acceptable level,
- Considering alternative substances or chemical routes which do not include explosive compounds.

Providing that the material is not unduly sensitive (or capable of detonation), then further process safety testing may proceed, i.e. thermal stability and dust explosion analysis.

If a material is not classified as explosive, it may still create an appreciable fire or dust explosion risk.

STOP! – Where do we go next?

If the material is potentially explosive (i.e. if any of the tools above indicate a potential for explosive hazards), then a more detailed investigation of explosive properties is required (Figure 2 Box 2). This should start with a more detailed thermal stability analysis using Differential Scanning Calorimetry (DSC). If the DSC results indicate a decomposition energy of >500 J/g, then explosion sensitivity tests as given under the United Nations (UN) Transportation of Dangerous Goods Recommendations, Tests Series 3 should be conducted to confirm that the material is not too sensitive to handle under normal processing conditions.

For transportation and classification purposes, tests to evaluate the severity of a detonation or rapid deflagration may need to be completed. These tests are again, normally undertaken using the UN Transport of Dangerous Goods Recommended test methods.

Explosivity Analysis

Differential Scanning Calorimeter

Explosive Properties Evaluation
In the first instance (Box 3), the critical question that must be answered is "Can the dust form a cloud?". This should not be interpreted as "Does the dust form a cloud in the workplace or equipment?".

Under normal operations, it may not be expected that the powder will form a cloud. However, if it is possible that the dust could form a flammable cloud then a Basis Of Safety must be selected and applied, which reduces the risk of a dust explosion to one that is as low as practicable. Certain process operations such as spray drying, charging of powders into reactors / hoppers / bags / storage containers, pneumatic conveying, milling and blending, etc., inherently involve the formation of dust clouds. In other operations, the potential may exist through abnormal operation (even though the dust does not usually exist in a cloud). An example might include dust which accumulates in layers and is disturbed, forming a cloud.

If the dust cannot exist as a cloud (for example, it is sufficiently wetted by water or solvent\(^2\) so that it cannot physically form a dust cloud at any stage in both normal and foreseeable abnormal circumstances, then no dust explosion hazard exists. In this case, further evaluation may be unnecessary.

Particle size has a huge impact on the flammability, sensitivity and severity of dust cloud explosions. As a very rough guide, particles below 500 μm should be considered as particularly flammable. When granules or pellets are handled, the potential for attrition to form fines must be considered. Even if only a few percent by weight of a powder is fine, this could still be enough to pose a serious dust explosion risk. If the bulk material is disturbed or transferred, the fines will remain suspended long after the larger particles have deposited. It is crucial that this concept is encompassed in the assessment of flammability hazards. All subsequent tests should be performed on the "finest material" that can accumulate in the process situation. Testing of unrepresentative samples will undoubtedly compromise the validity of the data.

\(^2\)For solvent damp powders, the flammability properties of the solvent must be considered (even if the dust cannot form a cloud). A Basis Of Safety for the flammable atmosphere should still be specified. Data for the dust will be required for processes where the dust is dried and is capable of forming a cloud.
Dust Explosion Screening

If a powder is capable of forming a combustible dust cloud then there are numerous ways of protecting process and personnel. In order to determine whether a powder is flammable when dispersed as a dust cloud as opposed to a powder layer (see Box 4) it is usual in the USA to perform a Group Go/NoGo Classification test (see appendix A.2.i), where a material may be classified as Go (flammable) or NoGo (non-flammable). In the EU a dust Explosibility test conducted in the 20l sphere apparatus following the kuhner method is more widely used to assess dust cloud flammability (see appendix A.2.ii).

However for most organic materials it can be expected with a reasonable degree of confidence that the powder will be flammable. The above screening tests are therefore sometimes bypassed for mixtures of flammable (organic) and non-flammable materials this assumption is not necessarily true and the test should be conducted.

If it is decided to by-pass the Group Go/NoGo screening test we would confirm the assumption of flammability by performing a Minimum Ignition Energy (MIE, see appendix A.3) test. This data is typically used as part of the process safety assessment for the identification of potential ignition sources especially electrostatic and mechanical discharges (see Figure 4).

In the initial stages of the MIE test if it is found that the material cannot be ignited with a high energy spark discharge, then the test procedure will revert to the Group Go/NoGo test. If after exhaustive testing, the material proves to be NoGo (i.e. non-flammable as a dust cloud), then no further dust cloud testing is required, although consideration must be given to thermal stability and fire risk.

The Minimum Ignition Energy (MIE) is an important parameter when assessing electrostatic or mechanical discharge incendivity hazards. There are two ways to determine the MIE – the purely capacitive method or by the addition of an inducer into the circuit (the inductive method). The inductive method uses a 1 MΩ inducer in the ground loop which results in the extended duration of the spark discharge. The IEC 61241-2-3, EN 13821 and ASTM E2019 standards for determining the MIE of dust / air mixtures allow both options depending on the application of the data.

Generally, the inductive method produces lower MIE’s than the capacitive method. MIE’s determined by the inductive method may be used in combination with the Minimum Ignition Temperature of the cloud [MIT, see appendix A.4] for determining the hazards associated with various types of impact spark arising from mechanical action. However, the purely capacitive discharge method must be used for electrostatic hazard assessment. The MIE method employed should be specified and agreed prior to commencement of testing.

Interpretation of MIE data is critical in assigning an appropriate Basis Of Safety. Figure 4 sets out the generally accepted pre-cautions that might be expected to be employed based on ignition energy alone.

Where powders have a high MIE, then it may be possible to implement ‘avoidance of ignition sources’ as a sole Basis Of Safety, providing supplementary data are obtained on, for example: the Minimum Ignition Temperature (MIT) and the Minimum Ignition Temperature Layer (MIT Layer, see appendix A.5) and a thorough hazard and risk assessment is undertaken.

If the MIE of a powder is very low (typically less than 25 mJ), then consideration should be given to measuring further electrostatic properties of the powder. The three main parameters to investigate are:

- **Powder volume resistivity**
  This gives an indication of how conducting or insulating a powder is. Data can also be used for the selection of electrical equipment.

- **Charge relaxation time**
  This is a measurement of how long electrostatic charge will take to relax to safe levels and is used with resistivity and chargeability data for an overall electrostatic hazard and problem assessment.

- **Chargeability**
  This identifies how easily a powder can accumulate electrostatic charge. It provides data on the electrostatic charge magnitude and polarity when the powders are conveyed along standard materials such as steel and plastic pipes. The data is useful for identifying high charging scenarios that may lead to ignition sources being present during a conveying operation. The information can also be used to solve non-safety related conveying problems such as powder hang-up, sticking or poor mixing as a result of excessive electrostatic charge generation.

It should always be the case that ignition sensitivity information for the powder is known for powder handling operations both inside process plant and the workplace. Only these data will allow identification of all ignition sources that are capable of igniting the cloud and precautions taken accordingly. As well as MIE, the sensitivity of the dust to electrostatic and mechanical discharges (including mechanical and electrical equipment) should be determined.

Two tests are performed to evaluate the sensitivity to ignition from hot surfaces - the Minimum Ignition Temperature (MIT) of a dust cloud and the Minimum Ignition Temperature Layer (MIT Layer) of a layer of powder (nominally 5 mm). Under the latest USA standards both tests are required to specify the maximum surface temperature of enclosure for electrical and non-electrical equipment that can be used in potentially hazardous areas.

If a material is found to be sensitive to ignition (i.e. low values of MIE, MIT or MIT Layer), then avoidance of ignition sources may not be acceptable as the ultimate Basis Of Safety. In such cases, ignition sources are identified and minimized as far as possible but some additional form of explosion prevention or protection may be required as the ultimate Basis Of Safety.
Flammable Limits (Dust Cloud)

For powders, it is particularly difficult to work outside of their flammable concentrations due to their ability to form powder layers that could regenerate a dust cloud and the inherent non-uniformity of concentrations in a dust cloud. The lower flammable limit of the powder or Minimum Explosible Concentration (MEC) is therefore considered to be of limited use for most practical situations. However, for truly steady-state dust forming processes, ventilation rates can be applied to ensure that powder concentrations are below the MEC.

Normally working under an oxygen depleted atmosphere is the most conventional way of avoiding the presence or formation of flammable atmospheres. Again, this option is only applicable to contained situations (i.e. the inside of vessels) where the oxygen level can be readily controlled and monitored. When this Basis Of Safety is employed, it is necessary to establish the Limiting Oxygen Concentration (LOC) below which a dust cloud becomes non-flammable. Without this necessary data, very low levels of oxygen must be established, controlled and monitored. These data are usually generated using the T-180 apparatus.

If the flammable atmosphere cannot be prevented then the avoidance of ignition sources and use of protections (ignitabilities) becomes the Basis of Safety (see Box 6 of Figure 3).

Explosion Severity

Where preventative measures alone cannot be used as a Basis Of Safety some form of dust explosion protection is required to ensure the safety of equipment and process operations.

Explosion protection can be achieved by using:

- **Containment**
  - The equipment must be of suitable strength to prevent rupture of the vessel in the event of an explosion.

- **Explosion pressure relief venting**
  - The provision of a weak panel that is sized adequately to prevent over pressurization of a vessel in the event of an explosion.

- **Explosion suppression**
  - A protective system which detects and quenches an explosion before hazardous pressures are attained.

Whatever of these cases is selected as the proposed Basis Of Safety the key design parameters are calculated using explosion severity data (see Box 7 of Figure 3). The 20-Litre Sphere test provides the necessary explosion severity data including the maximum explosion pressure (Pmax) and the rate of pressure rise (dP/dt) data expressed as a dust explosion constant (Kst). The maximum explosion pressure is used for containment design and the dust explosion constant is used for the specification of pressure relief venting or suppression system design.

A means of preventing explosion propagation (e.g. isolation valves) are always required with explosion protection measures and have to be considered in the hazard and risk assessment.

Special Cases

There are some situations which complicate the assessment of dust explosion hazards. These include (but are not limited to) the use of solvent damp powders and powders which normally have a high particle size (e.g. granules, pellets, tablets, etc.). A brief discussion on the testing strategy for such materials is provided below for guidance.

**Solvent Damp Powders**

For a solvent damp powder, the testing strategy will depend on the solvent level and physical nature of the powder. If there is sufficient solvent that the powder cannot form a dust cloud, then the properties of the flammable liquids dominate and the examination of dust explosion properties is only necessary for handling the dried powder. Therefore, the flammability of the solvent under processing conditions should be assessed and if necessary characterized and a Basis Of Safety applied accordingly.

**Note**

Although solvent damp powder may not be capable of forming a dust cloud, during processing powder may become dryer due to natural evaporation of the solvent especially when working at elevated temperatures. Pockets of dried dust may therefore occur in certain areas of the process.

Where a solvent damp powder remains capable of forming a dust cloud, then the following modifications to the normal procedure for pure dusts is proposed:

- **The MIE should be assumed to be that of the most sensitive component, usually the solvent** - if the powder is subsequently dried, then the MIE of the powder should be determined and used for powder handling and processing stages where “dry” material is present.
- **The MIT and MIT Layer of the dry powder should be determined and the results compared to the Auto-Ignition Temperature (AIT) of the solvent** - the limiting values should be the lowest temperature at which ignition could occur when applied to defining the maximum surface temperature of hot surfaces or enclosures for electrical equipment.
- **The LOD and explosion severity (Pmax and Kst) should be determined for the damp solid and compared to that of the dry powder**.

**Large Particle Size Materials and Blends**

Many materials whether they be raw ingredients, intermediates or finished products are often processed as granules or pellets rather than as finely divided powder. In such cases the question “can the material form a dust cloud?” becomes particularly important. The propensity to form a dust cloud should be based on a consideration of:

- **The friability of the powder i.e. how easily the material forms dust by attrition**.

- **The concentration of fine powder within the bulk of the material.** If only a small percent of fines to form a sizeable dust cloud. In addition it is the fines that will persist in an airborne state if the powder is poured, conveyed or disturbed.

- **Whether an accumulation of fine powder can occur during all or only specific operations**.

- **The potential of the processing operation to generate fine powder**.

If fine powder exists or can readily be formed, then the normal assessment route for dust explosion hazards should be followed. If a fine powder cannot accumulate “under all foreseeable processing conditions” then no further assessment of dust explosion hazards is required (providing the larger material has been found not to be dust explosive).

Dust cloud explosion test standards all dictate the maximum particle size of material that should be tested. For example, the EN method for determination of MIE dictates that the test should be performed on powder with a particle diameter of less than 63 µm.

In many cases, this requires some form of preparation (particle size reduction) prior to testing. This may involve mechanical milling followed by sieving to yield the correct particle size. Whilst this is relatively straightforward for single component powders, such procedures, when applied to blends, can lead to segregation of the various components. This may ultimately lead to testing of an unrepresentative sample. For this reason, particle size reduction for blended powders requires careful consideration.

**Charging Powders into Flammable Atmospheres**

When charging powders into flammable atmospheres (e.g. emptying material from sacks, IBCs or FIBCs into a solvent filled reactor), the electrostatic ignition sensitivity of the solvent usually predominates. Even if the solvent is operating below its flash point or below the Lower Flammable Limit (LFL), the ignition properties of the dust cloud can be affected. When a powder is outside the influence of the solvent vapor then the dust cloud ignition sensitivity values (MIE, MIT & MIT Layer) will be required to assess the ignition risk. Where solvent may be present in the powder, then the solvent influence requires separate consideration.

Even when a receiving vessel is pre-inerted, the oxygen concentration can rise rapidly during manual powder charging. Working below the LDC is therefore not considered an acceptable Basis Of Safety unless special powder charging procedures are employed which guarantee that the atmosphere in the vessel remains inert. Such measures may include the use of a double valve powder charging mechanism where the powder is pre-inerted prior to charging. For further information on the hazards of charging powders, please see the Chilworth document “A Guide to Process Safety”.

Minimum Ignition Temperature Layer Test (MIT Layer)
The ability of a powder layer to propagate flame or smoulder throughout its mass needs to be assessed especially where ignition could spread from a localized source of ignition. The fire properties of a material are assessed under laboratory conditions using either one of the following tests:

- **The Verein Deutscher Ingenieure (VDI) Burning Behavior Test**
  This test is performed at both ambient (20°C) and elevated temperature (100°C) and provides an indication (by way of a Combustibility Class (CC or German BZ) number, 1 - 6) of the extent to which a localized hot spot will propagate throughout a given mass of powder. This is the most appropriate test for process safety applications.

- **The EC and UN Flammability of Solids (Burning Rate) Test**
  This test provides data predominantly for regulatory purposes. This might mean the definition of a "Highly Flammable" classification under EC testing strategy or transportation classification as a Class 4, division 4.1, Highly Flammable Solid under the UN test methodology. The data can be also be used for process safety applications but does not give the depth of information as that from the VDI Burning Behavior test. Both the EC and UN tests are performed at ambient temperature only.

These tests can form a critical part of a hazard and risk assessment and the results may lead to the specification of fire suppression systems to mitigate the hazards posed by a high "fire" risk material.

<table>
<thead>
<tr>
<th>Combustibility Class Table - German &quot;Burning Number&quot; (BZ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 - No ignition</td>
</tr>
<tr>
<td>2 - Ignition and rapid extinction</td>
</tr>
<tr>
<td>3 - Local smouldering or burning</td>
</tr>
<tr>
<td>4 - Propagation of smouldering</td>
</tr>
<tr>
<td>5 - Propagation of open fire</td>
</tr>
<tr>
<td>6 - Flash fire</td>
</tr>
</tbody>
</table>

The VDI Burning Behavior Test Set-up

Table 1.
In the previous section, the flammability characteristics of dust clouds and powder layers have been considered. However, if products are to be processed at elevated temperature then consideration must be given to evaluating the safety implications from the bulk storage or drying of powders. Thermal stability data for powders are used to define safe operating temperatures for drying applications or where powders are otherwise subjected to elevated temperatures. The necessary risk assessment must include data that has been gathered experimentally in order to confirm a proposed Basis Of Safety for the drying or storage application.

It is not uncommon for powders to be processed at elevated temperatures and then stored often in very large quantities for prolonged periods of time. It is therefore essential to understand the thermal stability characteristics of the materials being processed and stored, together with the operating conditions under which they are handled; otherwise it may be possible to initiate a dangerous exotherm arising from self-heating or self-reaction.

Undesirable exothermic events in bulk powder handling and storage will normally be the result of either:

- **Pure molecular decomposition:**
  With this type of reaction the material decomposes, at a molecular level, usually generating heat and volatile and/or non-condensable decomposition products. This “self-reaction” does not require any additional component to proceed and is generally relatively unaffected by all environmental conditions other than temperature.

- **Oxidation resulting in self-heating:**
  With this type of reaction the material reacts exothermically with its environment. This is not a “self-reaction” and specifically requires an oxidant for the reaction to proceed. This type of process is highly dependent on environmental conditions such as material particle size, geometry of the process vessel and air availability. Ultimately, this reaction generally leads to burning, glowing or combustion (with flame) of the powder. Powders are particularly susceptible to this type of reaction owing to their small particle size and large surface area to volume ratio.

Note: Both of these forms of self-heating should not be confused with biological degradation as found in a compost heap.

When assessing thermal stability hazards, the specific processing operations should also be examined to identify whether or not, one or both of these reaction mechanisms can occur. This information will assist in selection of the correct testing methods to provide the necessary data.
Thermal Stability Analysis

Thermal Stability Screening

To ensure that the correct testing approach is selected, there are two questions which can be asked which will decide whether molecular decomposition properties, oxidation properties or both should be examined. Specifically, does the material have a low melting point (<200°C) OR is the material being processed at elevated temperatures in an inert environment? If the answer is ‘yes’ for either of these questions then only the molecular decomposition properties of the product require assessment.

If however the material has a high melting point and is processed in the presence of air or could be exposed to an oxidant, especially in the event of a process failure, then oxidation properties should be investigated. It should be noted that in this latter case even if the material exhibits molecular decomposition properties during testing, these effects will be identified and characterized in the oxidation test methods. The converse is not true. For example, oxidation events will not be identified in molecular decomposition specific tests owing to the limited availability of air.

The overall approach for assessing powder thermal stability is depicted in Figure 5. The strategy is discussed in more detail in the sections following.

Testing for Potential Oxidation Reactions

Screening

Where a material is exposed to elevated temperatures in the presence of air, special test methods are required to assess the oxidation potential (see Boxes 11 and 12 of Figure 5). Decomposition tests (as described in the previous section) are not designed to examine oxidation properties and may provide misleading and unsafe data if used for this purpose.

One or more of the following three tests are usually employed where oxidation reactions are being considered:

- Bulk Powder (Diffusion Cell) Test (DCT)
  - This test is selected when powders are processed or stored in “bulk” conditions and the data is directly applicable up to volumes of 1 m³. In this test the sample is heated through a known temperature profile, with air being allowed to diffuse naturally through the powder. The test simulates, for example, the air availability that bulk powder might expect to be exposed to in a storage silo or in the base of a ‘dirty’ spray dryer.
  - Aerated Cell Test (ACT)
    - This test is selected specifically for powder drying applications where pre-heated air is forced through the material. Fluidized bed drying is a good example where the Aerated Cell Test could be applied or a rotating drum dryer. The effect of varying air availability can alter the thermal oxidation onset temperature by as much as +/-50°C from that of the DCT, a margin that can significantly affect the safety of a drying operation if an incorrect test method is employed.
  - Air Over Layer (AOL) Test

With all of the above test methods, screening tests are initially recommended, where the sample temperature is ranged from ambient to nominally 400°C at a steady rate of temperature rise. If the estimated onset temperature is close to the drying temperature, i.e. within 50°C, OR is less than 200°C, then further testing may be required. In the first instance, this consists of a series of isothermal tests (sample is held at a constant temperature) to determine, more sensitively, the minimum temperature from which significant self-heating can occur. The duration of the tests must meet or exceed, the expected exposure duration of the large scale process.

Even with isothermal tests, the results are subject to volume restrictions (up to 1 m³ only) and require a suitable safety margin of between 20 and 50°C.

Large Scale Drying or Storage

In certain circumstances, for example where large bulks of material are being processed or stored it will be necessary to determine safe storage temperatures and duration times. These values are determined using the isothermal basket test (Box 13 of Figure 5) approach. These series of tests employ three wire mesh baskets of differing volumes. The minimum onset temperature and the maximum temperature where no reaction is observed are determined to a very close tolerance (typically 3°C). The data for the 3 basket sizes are then displayed graphically and can be extrapolated, taking account of the mass and geometry of the storage vessel, to the industrial scale. Safe working (storage) temperatures and maximum safe duration times can then be obtained for any given size of vessel.
The previous sections identify how data from powder testing can be applied in a risk assessment for most general manufacturing and processing operations. An alternative approach is to identify the types of data typically required for specific process operations. Table 2 gives an overview of the most common type of processes encountered in manufacturing environments, where powders could exist as a dust cloud. Not all of the suggested tests may be required for an individual operation as the selection of data will depend upon the proposed Basis Of Safety.

Table 2. Dust Explosion Test Data - Requirements for Specific Unit Operations

<table>
<thead>
<tr>
<th>Process Description</th>
<th>Test Data Required</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MIE</td>
</tr>
<tr>
<td>Charging of vessel</td>
<td>Yes</td>
</tr>
<tr>
<td>Discharging of vessel</td>
<td>Yes</td>
</tr>
<tr>
<td>Blending</td>
<td>Yes</td>
</tr>
<tr>
<td>De-agglomeration</td>
<td>Yes</td>
</tr>
<tr>
<td>Milling</td>
<td>Yes</td>
</tr>
<tr>
<td>Compaction</td>
<td>Yes</td>
</tr>
<tr>
<td>Compression</td>
<td>Yes</td>
</tr>
<tr>
<td>Capsule filling</td>
<td>Yes</td>
</tr>
<tr>
<td>Granulation</td>
<td>Yes</td>
</tr>
<tr>
<td>Tray drying</td>
<td>Yes</td>
</tr>
<tr>
<td>Microwave dryers</td>
<td>Yes</td>
</tr>
<tr>
<td>Fluid Bed drying</td>
<td>Yes</td>
</tr>
<tr>
<td>Flash or Ring dryers</td>
<td>Yes</td>
</tr>
<tr>
<td>Spray coating</td>
<td>Yes</td>
</tr>
<tr>
<td>Pneumatic conveying</td>
<td>Yes</td>
</tr>
<tr>
<td>Filters</td>
<td>Yes</td>
</tr>
<tr>
<td>Storage (silo etc.)</td>
<td>Yes</td>
</tr>
</tbody>
</table>

It should be recognized that even for a specific process, different methods of operation are possible thus requiring a different Basis Of Safety to be applied. This may result in a different test protocol being used to that set out above.

As previously discussed, the type of thermal stability test required will depend very much on the type of dryer employed and the environmental conditions. Table 3 shows the types of thermal stability tests that may be required for different types of dryer:

Table 3. Thermal Stability Test Data Requirements for Specific Drying Operations

<table>
<thead>
<tr>
<th>Type of Dryer</th>
<th>Air Over Layer</th>
<th>Aerated Cell</th>
<th>Diffusion cell</th>
<th>DSC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spray</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>Fluid Bed</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>Vacuum</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>Tray</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>Flash or Ring</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
</tbody>
</table>

Note: The above table shows the types of test that may be required. However, depending on the operating conditions of the equipment certain tests may not be selected. For instance in a small tray dryer where powder depth is <15mm and no explosive properties are suspected then only the Air Over Layer test would be selected. However, in deeper layers the Bulk Powder (diffusion Cell) test would be selected instead of the Air Over Layer test.
This document has provided a methodical approach to powder testing and can act as a guide to companies assessing the explosive potential, dust explosion risk, fire risk and thermal stability hazards posed by powders in any industrial operation. The overall strategy is depicted in Figure 6.

In summary, the following approach is recommended when considering powder/dust testing:

- Start with an appreciation of the potential explosive properties of the material. This assessment needs to be completed and signed-off before any large scale processing (or testing) is undertaken.
- A Basis Of Safety should be developed, documented and maintained for all unit operations. In order to confirm this Basis Of Safety it will be necessary to obtain specific flammability, fire risk and/or thermal stability data for each material used or stored on site.
- Any new material introduced to a manufacturing process must also be evaluated to confirm the adequacy of the existing Basis Of Safety for the new material.
- Testing must replicate the conditions in the full scale process as closely as possible. While many parameters can be obtained from standard tests, others may require innovative, customized testing solutions. Chilworth Technology regularly develops and performs unique testing solutions for individual situations.
Multi Powder Screening

The standard test procedures, as detailed in this document, are intended as a guide to provide a sensible testing strategy for a powder, whether it is a suspended cloud, powder layer or as a bulk material. However, in multi-product facilities (e.g. pharmaceutical, food industry or fine chemical companies), such detailed testing may not be cost effective. In these cases, it may be more desirable to undertake a more rapid and cost-effective screening exercise for a wide variety of properties. This screening approach may be used to confirm that a new product is well within the process design criteria for a range of flammability/thermal stability properties without having to perform a detailed analysis for each property.

- DustScreen™
  This highly tailored product identifies key safety parameters which must be known to support a specific Basis Of Safety. Each material is initially tested at a pre-selected level for each key parameter, for instance electrical equipment has a specified surface temperature of enclosure and therefore the MIT & MIT Layer tests are performed at the correct temperature to define whether a powder is suitable for use with this equipment. Those materials failing the first level assessment may continue with testing and be screened at second and then, if required, a final third level in order to determine the worst case materials. Dustscreen™ has been developed specifically for companies utilizing common process with a variety of powders.

At Chilworth Technology, we offer a complete process safety service. We understand the importance of customer satisfaction in retaining customers and growing our business. Our team of experienced process safety specialists is available to support and apply test data to our customers' unique situations. We can help in defining a Basis Of Safety for any process and developing test programs tailored to deliver the data you need in a highly cost effective manner.
Chilworth Technology can perform a wide variety of process safety tests examining explosion hazards of dusts, vapors and gases, thermal stability characteristics of powders, liquids and mixtures and reaction calorimetry to ensure safe operation of reactors.

- Many clients are unsure of the exact test, or data, required to solve a specific process problem. Chilworth Technology laboratories and consulting team are available to provide assistance in the selection of the most appropriate tests and, in the most cost-effective manner, to answer your query. This pre-testing consultation (as well as post-testing results discussion) is provided at no extra cost. Chilworth Technology prides itself on the high level of customer support it provides.

- Every test (except custom, one-off investigative tests) is conducted according to international standards and internal Standard Operating Procedures (SOPs).

- Test materials should, as far as is reasonably practical, be those from the process. This avoids the testing of materials with different specifications and impurity profiles than those actually used in process.

- For dust explosion testing, test standards dictate that materials should be "finest and driest" available on process. For consistency and conservatism, tests are performed on materials <5% moisture content and, where the standard methods dictate, less than a specific particle size (typically <75 micron). Blended powders are processed with caution to avoid separation of materials (e.g. by sieving) which could result in unrepresentative materials. When providing samples for testing, great care must be exercised to ensure that all samples are truly representative of the bulk material characteristics.

- For many tests, reduced versions of the full test are available. At Chilworth Technology, unless specifically stated, the test will be conducted fully in accordance with the Standard Method (e.g. EN, IEC, ASTM, etc.). In some cases, reduced form tests can be completed to generate specific data. The limitations of such a reduced method must be accepted prior to acceptance of the reduced form result. When comparing tests and results with external data, confirmation must be sought regarding the extent of compliance with the standard method.

- All test data generated by Chilworth is fully reported. This includes background information on the material (particle size analysis, moisture content and preliminary thermal screening), full test results and data interpretation to aid in application of the derived result.

- Chilworth Technology participates in many international “round-robin” calibration studies to verify the consistency of data with other international test laboratories. Results from such studies, where CTI participate, can be provided on request. Chilworth Technology recognizes the critical importance of providing its customers with data which answers specific concerns. Where a standard test does not exist to provide such data, tailoring of unique testing solutions is regularly undertaken.
A2. Group Go/NoGo Classification Test - Explosibility Test

Appendix A2. Group Go/NoGo Classification Test

<table>
<thead>
<tr>
<th>International Standard</th>
<th>ASTM E1226</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity of Sample</td>
<td>100 g</td>
</tr>
<tr>
<td>Alternative Tests</td>
<td>20 l Sphere Test, modified Hartman tube test</td>
</tr>
</tbody>
</table>

**Test Purpose**

To examine if a dispersed powder can present an explosion hazard under ambient conditions and at elevated temperatures.

**Test Method**

The Group Go/NoGo classification test is performed using equipment which is known as the vertical tube apparatus. It consists of a vertically mounted acrylic tube of approximately 1 ltr volume. The tube is fitted with a compressed air dust dispersion system and brass electrodes to which a high voltage transformer is connected. The transformer is used to create a high energy, continuous arc discharge between the two electrodes.

The test procedure reflects the objective of the classification test: specifically, all reasonable measures are taken to attempt to ignite the dispersed dust sample in air under ambient temperature and pressure conditions. The sequence of testing is as follows:

1. If “no ignition” is observed then the sample is sieved and the finest fraction is re-tested in (1) above.
2. If “no ignition” is still observed then the dust is dried in an oven at 105°C for 1 hour and both the original and fine fractions re-tested after the powder has cooled.
3. If “no ignition” is still observed then the constant arc electrodes are replaced with a hot wire resistance coil heated to approximately 1,000°C and the sieved and dried sample is tested.

In some circumstances material may be processed at elevated temperatures (above 110°C). In this case, if a material has not ignited after completing the 4 steps above then the sieved and dried sample is subjected to a further test whereby the product, in the form of a dust cloud, is dropped through a vertical furnace with a surface temperature of 1,000°C.

If any of the above tests produce flame propagation through the dispersed dust, away from the ignition source, the dust is classified as Group Go (explosible / flammable).

**Results and Interpretation**

If the material is found to be flammable as a dust cloud it is designated Group Go and further testing will be required to ensure safe operating conditions. If found to be Group NoGo (non-flammable) then no further dust cloud testing is required but please be aware that the flammable and non-flammable results may still need to be assessed.

**Reduced Versions of the Test**

No reduced version of this test exists.

**Test Limitations**

The test is unsuitable for classification of liquids / gases. The ignition sources used in the test are moderately energetic. It is uncommon (but not impossible) for materials found to be Group NoGo by the Group Go/NoGo test to ignite with higher ignition energies. If very high energy ignition sources can exist in a process (as identified by a suitable hazard and risk assessment), consideration should be given to performing the 20 l sphere test that uses a higher (10kJ) energy ignition source.

---

A3. Minimum Ignition Energy Test (MIE)

Appendix A3. Minimum Ignition Energy Test (MIE)

<table>
<thead>
<tr>
<th>International Standard</th>
<th>ASTM E2019</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity of Sample</td>
<td>250 g</td>
</tr>
<tr>
<td>Alternative Tests</td>
<td>-</td>
</tr>
</tbody>
</table>

**Test Purpose**

To determine the minimum energy of an electrostatic or mechanical spark capable of igniting a dispersed dust under ambient conditions.

**Test Method**

The Minimum Ignition Energy (MIE) is measured using equipment known as the vertical tube apparatus. It consists of a vertically mounted acrylic tube of approximately 1 ltr volume. The tube is fitted with a compressed air dust dispersion system and brass electrodes between which sparks of a known energy are passed.

MIE measurement involves repeatedly dispersing varied concentrations of dust through sparks of known energy. The powder concentrations for these tests are greater than the Minimum Explosive Concentration (MEC). If an ignition is observed then the spark energy is reduced until no further dust ignitions occur; at this point the Minimum Ignition Energy of the powder is determined. The sparks may either be purely capacitive in nature where the data is used to examine electrostatic hazards and / or via an additional inductor, fitted in the earth circuit where the data can be used in the assessment of mechanical sparks. It is usual for sparks, created with the addition of the inductor, to be more incendive (i.e. provide a lower MIE) compared with purely capacitive sparks. The use of the data must be known in selecting the correct test methodology.

**Results and Interpretation**

The Minimum Ignition Energy (M.J) is recorded as the lowest energy capable of igniting a dispersed dust (at its most sensitive concentration). It is always given as a range of values from the highest energy where no ignition was observed to the lowest energy where an ignition is observed (i.e. 10 - 25 mJ).

**Reduced Versions of the Test**

Several variants of this test are available:
1. Complete determination of MIE within limits of +/- 20% of the lowest ignition energy
2. Safety range study (examining the MIE at limits of 500, 100, 25 and 5 mJ only)

**Test Limitations**

The test is for solid samples only and cannot be used (directly) for gases and vapours (although a similar test is available for these materials). The result is determined at ambient temperature. The MIE can reduce dramatically at elevated temperature (correlations can be employed to approximate the MIE at elevated temperature). Solids containing volatile, flammable solvents may not give a reliable result in this test. In such cases, the MIE of the vapor (if flammable at ambient temperature) should be used as this is likely to be worst case and most realistic value.
A4. Minimum Ignition Temperature Test (Cloud)

**Test Purpose**
To determine the minimum temperature of a hot surface capable of igniting a dust cloud.

**Test Method**
The MIT test is conducted in the Godbert Greenwald furnace. The furnace consists of a vertically mounted vitreous tube which is open at the base. A glass observation chamber is mounted at the top of the vitreous tube and also connects the horizontally positioned sample holder. Dust is dispersed into the furnace using compressed air from a reservoir, into the sample holder and then the observation glass and vertical tube. The furnace is mounted on a stand, enabling the base or exit point of the furnace to be observed for any sign of ignition (flame) from the base of the furnace. A mirror is placed below the tube to enable the interior of the furnace to be viewed. The furnace is electrically heated to pre-determined temperatures from room temperature to 1,000°C.

Two thermocouples are placed centrally in the furnace wall to enable the test temperature to be suitably controlled and monitored. The thermocouples are capable of maintaining temperature measurements above 500°C to ±1% and below 300°C ±3%. The apparatus is set up in an enclosure from which dust and fumes can be extracted.

The sample may be prepared to a specified standard (normally <15% moisture and <75 microns) and dust concentrations are varied to ensure that the most sensitive concentration is covered. The test commences at 500°C and if an ignition is observed then the temperature is reduced in 20°C steps to 300°C. If ignition is still occurring then the test temperature is reduced in 10°C steps until a “no ignition” situation occurs.

The Minimum Ignition Temperature, of a dust cloud, is recorded as the lowest temperature of the furnace to result in ignition. The test continues until it has been ascertained that either the layer has ignited (visible flame) or has self-hvated without ignition (glow). If, after a period of thirty minutes, no ignition or self-heating is apparent then the test is terminated and repeated at a higher temperature. If ignition or self-heating occurs, the test is repeated at a lower temperature, if necessary prolonging the test beyond thirty minutes. The test is complete only when temperatures are established that result in a 10°C difference between an “ignition” and “no ignition”. If no ignition occurs at 600°C, tests are repeated at 500 and 300°C to confirm the no ignition result.

The resultant computer trace is analyzed to determine an estimated start point for the isothermal testing. Isothermal testing continues with the hot plate being set to the pre-determined start temperature. The test continues until it has been ascertained that either the layer has ignited (visible flame) or has self-hvated without ignition (glow). If, after a period of thirty minutes, no ignition or self-heating is apparent then the test is terminated and repeated at a higher temperature. If ignition or self-heating occurs, the test is repeated at a lower temperature, if necessary prolonging the test beyond thirty minutes. The test is complete only when temperatures are established that result in a 10°C difference between an “ignition” and “no ignition”. If no ignition occurs at 600°C, tests are repeated at 500 and 300°C to confirm the no ignition result.

The MIT test is principally used to ensure that process surface temperatures cannot cause auto-ignition of the dispersed dust. A safety margin is required when using MIT data to allow for the uncertainties of small scale testing (typically, 50°C of the MIT is used to define the maximum permissible surface temperature of enclosure for electrical equipment).

The MIT of a dust cloud, is one criteria used for the selection of suitable electrical equipment operating in dusty atmospheres. The second parameter also relevant to this application is the MIT Layer value of the powder. Ignition is signified by:
- visible glowing or flaming is observed, or
- a temperature rise of 50°C.

A safety margin of 75°C is applied to the experimental MIT Layer value when the data is used to define the maximum surface temperature of enclosures for electrical equipment.

Reduced Versions of the Test
Several reduced versions of this test exist:
1. Dust screen at an agreed isothermal temperature
2. For temperature rating of electrical equipment (equivalent equipment temperatures of 550, 300, 200, 135°C)

**Test Limitations**
The test is for solid samples only and is not applicable for gases and vapors (the auto-ignition temperature test is available for gases and vapors).

---

A5. Minimum Ignition Temperature Layer (MIT Layer)

**Test Purpose**
To determine the minimum temperature of a hot surface capable of igniting a powder layer (5 mm in depth).

**Test Method**
The density of the powder layer (packing density) is calculated by knowing the volume of the containment and by weighing the dust layer before and after it has been filled with a level layer of powder. The apparatus (a temperature controlled hot plate) is set up in a position free from draughts but capable of extracting smoke and fumes.

The ambient temperature is recorded and then the metal, containment ring is placed centrally on the hot plate and the sample measuring thermocouple placed in position. The 12.7 mm dust layer is formed by placing the sample into the ring with a spatula and distributing, mainly with a sideways movement of the spatula, until the ring is slightly overfilled. The layer is then levelled by drawing a straight edge across the top of the ring. Any excess material is then removed from the surface of the hot plate. A screening test is initially performed where the hot plate temperature is ramped at approximately 5 K/min up to a maximum temperature of 600°C. All information is collected using a computer controlled data logging system.

The result of this test is analyzed to determine a recommended start point for the isothermal testing. Isothermal testing continues with the hot plate being set to the pre-determined start temperature. The test continues until it has been ascertained that either the layer has ignited (visible flame) or has self-hvated without ignition (glow). If, after a period of thirty minutes, no ignition or self-heating is apparent then the test is terminated and repeated at a higher temperature. If ignition or self-heating occurs, the test is repeated at a lower temperature, if necessary prolonging the test beyond thirty minutes. The test is complete only when temperatures are established that result in a 10°C difference between an “ignition” and “no ignition”. If no ignition occurs at 600°C, tests are repeated at 500 and 300°C to confirm the no ignition result.

The MIT Layer value is defined as being the lowest temperature of a hot surface capable of igniting a 5 mm layer of powder. Ignition is signified by:
- visible glowing or flaming is observed, or
- a temperature rise of 50°C.

A safety margin of 75°C is applied to the experimental MIT Layer value when the data is used to define the maximum surface temperature of enclosures for electrical equipment.

Reduced Versions of the Test
Several reduced versions of this test exist:
1. Dust screen at an agreed isothermal temperature
2. For temperature rating of electrical equipment (equivalent equipment temperatures of 550, 300, 200, 135°C)

**Test Limitations**
The standard test examines 5 mm layers only (thicker layers may produce a lower result).
Appendix A6. Dust Explosion Severity Test (20 Litre Sphere Test)

Appendix A6. Dust Explosion Severity Test (20 Litre Sphere Test)

**Test Purpose** To measure the explosion severity of a powder sample as defined by the following:
- Pmax: The maximum explosion pressure (in bar(g))
- (dP/dt)max: The maximum rate of pressure rise (in bar.s⁻¹)
- Kst: The Kst value equates to the maximum rate of pressure rise in a 1 m³ vessel (in bar.m.s⁻¹)
- St Class: Explosion severity class (no unit)

**Test Method**

Pmax and (dP/dt)max are measured using a 20 litre spherical pressure test chamber (the lowest volume from which explosion results can be reliably scaled-up) and then the Kst value is calculated and the St class defined. The test commences by using a low dust concentration and continues over a range of powder concentrations. The source of ignition is provided by 2 x 5kJ chemical igniters linked to a computer controlled data logging system. The ignition source is activated automatically at 0.06 s after dispersion of the powder into the spherical test chamber in order to maintain a constant and reliable dust turbulence level. A first series of tests typically covers 7 powder concentrations and this is followed by two further series that concentrate on the optimum dust concentration that has been determined from the first series of tests.

**Results and Interpretation**

During triplicate testing of powders in the 20 litre sphere, a minimum of seven different powder concentrations are tested in the first series and the powder concentrations that produce the highest values for Pmax and (dP/dt)max are repeated twice more providing three tables of results. A fourth table is then produced to represent the mean values for each of the concentrations tested. The final quoted values for Pmax, (dP/dt)max and Kst are calculated from the mean of the maximum values during test series 1, 2 & 3. Maximum values are not quoted as any variation in turbulence or chemical igniter energy may affect the results marginally. Rather, the data is averaged over the three series of tests. The turbulence level is selected to correlate with data from large industrial scale vessels.

**Reduced Versions of the Test**

Several reduced versions of this test exist:
1. Dust Screen to identify a worst case material from a batch of products
2. Single series determination (7 dispersions) – not recommended for design
3. Screening test to identify a worst case material from a batch of products

**Test Limitations**

The test is for solid samples only and cannot be used (directly) for gases and vapors (although a similar test is available for these). The test considers ambient temperature and pressure conditions only. Hybrid mixture (dusts admixed with flammable gases) can be studied in the 20 litre apparatus with minor modification of the procedure.

Appendix A7. Limiting Oxygen Concentration Test (LOC)

Appendix A7. Limiting Oxygen Concentration Test (LOC)

**Test Purpose** To determine the highest oxygen concentration at which ignition of a dispersed dust at ambient temperature and pressure is not possible.

**Test Method**

The Limiting Oxygen Concentration (LOC), below which dispersed dust ignition is not possible, is measured in a 20 litre sphere explosion chamber. This apparatus is internationally accepted for use in determining maximum explosion pressure and Kst value of dusts. It provides maximum explosion pressure and ignition control and is well suited to limiting oxygen determination.

The test procedure consists of pre-mixing air and nitrogen in the correct ratios to establish known diminished oxygen atmospheres. The system must first be calibrated using an oxygen analyzer. Explosion trials are then performed at various (known) oxygen levels. It should be noted that the ignition source utilized in these tests are 1 x 2.5 kJ chemical igniters as against the 2 x 5 kJ ignition sources (10 kJ) employed for explosion severity measurements. The pressure/time data for each test is recorded using 2 x piezo electric pressure transducers linked to a computer controlled data logging system. The test starts, as per explosion severity determination, with a single series of tests at 21% oxygen (ambient conditions) to determine the most severe explosion (optimum dust concentration). The ratio of powder concentration to oxygen percentage is then retained as the oxygen level is reduced to determine the LOC. At the highest oxygen concentration where no ignitions are observed, the tests are repeated using a range of powder concentrations. If ignition is observed at this stage of the proceedings then the oxygen level is reduced and testing continues as before.

**Results and Interpretation**

An explosion pressure of 1.0 bar(g) is defined as an ignition. The LOC measurement is not normally used directly to provide inerting levels as a suitable safety factor should be applied to account for the sensitivity, accuracy and reliability of the process monitoring system.

**Reduced Versions of the Test**

Screening test [at pre-defined levels x 10, 12, 14 and 16% oxygen]

**Test Limitations**

The test is for solid samples only and cannot be used (directly) for gases and vapors (although a similar test is available for these). The test considers ambient temperature and pressure conditions only.
Appendix - Test Descriptions

A8. Minimum Explosible Concentration (MEC) in the 20 litre sphere

Appendix A8: Minimum Explosible Concentration (MEC) in the 20 litre sphere

<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity of Sample</td>
<td>150 g</td>
</tr>
<tr>
<td>Alternative Tests</td>
<td>1 m³ Sphere Test</td>
</tr>
<tr>
<td>Test Purpose</td>
<td>To determine the minimum concentration of a dust at which ignition is possible</td>
</tr>
<tr>
<td>Test Method</td>
<td>Minimum Explosive Concentration (MEC) is measured using the 20 litre sphere apparatus. The test commences with testing at a relatively high powder concentration (e.g. 250 g m⁻³). If this concentration of powder ignites with a 1 x 2.5 kJ ignition source, the dust concentration is reduced in steps until no ignition is evident. This condition must be repeated for three consecutive tests at the given concentration. If, during the repeat tests, an ignition is observed then the dust concentration is reduced further and the tests repeated until no ignition situation occurs for all 3 dispersions at a given powder concentration. The pressure time record of each explosion is recorded by piezoelectric pressure transducers, coupled to a computerized data logging system to provide the maximum pressure (P_max) and the maximum rate of pressure rise (dP/dt) max. The ignition source is activated automatically at 0.06 s after dispersion to define the dust turbulence level, according to the test standard. It is essential to the test that the apparatus is kept scrupulously clean to prevent false positive readings.</td>
</tr>
<tr>
<td>Results and Interpretation</td>
<td>An explosion pressure of ≥ 0.5 bar(g) is defined as an ignition. The Minimum Explosive Concentration (MEC) is sometimes used to design process with sufficient air throughput to keep dust levels below the flammable limit. However, some caution must be exercised with this approach since in practice dust clouds are rarely uniform, and settlement or unforeseen circumstances can easily arise to create localized flammable concentrations even though the mean concentration of the dust cloud may be below the MEC.</td>
</tr>
<tr>
<td>Reduced Versions of the Test</td>
<td>Screening test at pre-defined levels e.g. 125, 75, 50, 25 g m⁻³</td>
</tr>
<tr>
<td>Test Limitations</td>
<td>The test is for solid samples only and cannot be used (directly) for gases and vapors (although a similar test is available for these). The test considers ambient temperature and pressure conditions only.</td>
</tr>
</tbody>
</table>

A9. Differential Scanning Calorimetry (DSC)

Appendix A9: Differential Scanning Calorimetry

<table>
<thead>
<tr>
<th>International Standard</th>
<th>Several ASTM standard methods for applications of DSC exist</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity of Sample</td>
<td>50 mg</td>
</tr>
<tr>
<td>Alternative Tests</td>
<td>-</td>
</tr>
<tr>
<td>Test Purpose</td>
<td>To determine the energy associated with the decomposition of a material or mixture - potentially to screen for explosive properties. Semi-quantitative data relating to onset temperature can also be gained from this test technique. Kinetic analysis can be performed for decomposition reaction for extrapolation to larger scale. The results are not directly scaleable (i.e. need a margin of safety). For powders, lack of air availability may hinder detection of oxidation events. Pressure events (e.g. gas generation) are not detected by this method. Blends and mixtures are difficult to study owing to the inherent challenge of representative sampling (the Carius tube is a more appropriate technique in such cases).</td>
</tr>
<tr>
<td>Test Method</td>
<td>A small quantity of sample (typically 5 - 20 mg) is loaded to the test cell (either constructed of stainless steel, aluminium or gold). For safety studies sealed, high pressure cells are best suited to prevent evaporation losses. The sample is then ramped within the DSC instrument along with a reference pan of identical construction. The ramp rate is nominally between 1 and 20 K min⁻¹ although higher heat rates provide results of lower sensitivity with respect to onset temperature determination. Any exothermic or endothermic activity is measured through measurement of heat flow between the sample and reference pans. The amount of energy released or absorbed by the sample can be integrated as a measure of the overall energy of a reaction. Tests can be performed isothermally for the study of autocatalytic reactions or at different ramp rates for the extraction of formal kinetic data.</td>
</tr>
<tr>
<td>Results and Interpretation</td>
<td>Typically, one graph of power versus time is provided. Interpretation is conducted by the computer control system which will provide data (on the test graph) relating to the onset temperature and energy of the reaction (usually normalized to J g⁻¹). The onset temperature obtained is not absolute (due to the high phi factor and heat losses of the test technique) and a safety factor is required. Typically, for the high heating rates employed (≥5 K min⁻¹), a safety factor of up to 100 K may be employed. For this reason, more accurate onset temperature information is provided by the Carius tube used at lower heating rates and with a larger sample. The energy of a decomposition does not require such modification and is used directly. Tests performed under air and nitrogen can be compared to identify whether an event is attributable to oxidative processes or pure decomposition. Formal kinetic data can be extracted for decomposition reactions based on the analysis of results from multiple tests. Any decomposition energy &gt;800 J g⁻¹ indicates potential for explosive properties to exist in the material.</td>
</tr>
<tr>
<td>Reduced Versions of the Test</td>
<td>No reduced version of this test exists</td>
</tr>
<tr>
<td>Test Limitations</td>
<td>The results are not directly scaleable (i.e. need a margin of safety). For powders, lack of air availability may hinder detection of oxidation events. Pressure events (e.g. gas generation) are not detected by this method. Blends and mixtures are difficult to study owing to the inherent challenge of representative sampling a blend at such low masses.</td>
</tr>
</tbody>
</table>
Appendix A10. Carius (10 g) Tube Screening Test

**International Standard**
No formal standard applies although the method is described in the ABPI and IChemE publications on Chemical Reaction Hazard Assessment and is considered best practice.

**Quantity of Sample**
10 g per test (although 30 g preferred to allow duplication of test)

**Alternative Tests**
Many alternative DTA methods exist.

**Test Purpose**
The test is designed to provide a preliminary indication of the thermal behavior of a material. Exothermic, endothermic and gas generating events are determined in a semi-quantitative fashion. The test can be undertaken on a liquid, solid or mixture.

**Test Method**
A small quantity of material (10 - 15 cm³) is placed in the Carius tube which is positioned at the center of a furnace, connected to a pressure transducer and ramped (at a constant rate of typically 0.5 K min⁻¹ from ambient temperature to 400°C or a tube pressure of 55 bar(g) whichever comes first). Energetic events are indicated by positive (exothermic) or negative (endothermic) deviations from the baseline temperature differential between sample and oven. Pumped additions to initiate a reaction and agitation are possible with this test.

**Results and Interpretation**
The output contains three graphs. Graph (a) is a full temperature, pressure and time trace. Graph (b) is a plot of temperature versus the differential between the oven and sample. Graph (c) is a plot of ln (pressure) versus the reciprocal of absolute temperature. The onset of an event is recorded as the point at which a deviation in differential temperature is just observed (upwards = exotherm, downwards = endotherm). A safety factor of typically 50 K is used on onset temperature to account for the high heat losses of the test equipment. The peak height (and width) are a measure of the magnitude of the event although these are only qualitative and are not directly scalable. A wide peak is indicative of a mass transfer controlled reaction. An upward deviation from linearity in the Antoine plot indicates the onset of gas generation. The steepness of the rise is indicative of the rapidity of gas generation. The residual pressure in the tube after cooling gives a quantitative measure of the gases evolved (mass spectrometry can be employed to assess the nature of the gail).

**Test Limitations**
The results are not directly scaleable (i.e. need a margin of safety). For powders, lack of air availability may hinder detection of oxidation events.

**Reduced Versions of the Test**
No reduced version of this test exists.

Appendix A11. Diffusion Cell Screening Test

**International Standard**
This test meets the specification in the 1990 IChemE book “Prevention of Fire & Explosions in Dryers” pages 21 - 23 and is considered current best practice.

**Quantity of Sample**
150 g

**Alternative Tests**
Basket Tests

**Test Purpose**
The purpose of this test is to simulate conditions in silos or bags and at the bottom of dryers where material can collect in bulk with natural diffusion of air. If the heat developed by a reaction of substance with oxygen or by exothermic decomposition is not lost rapidly enough to the surroundings, self heating leading to self ignition can occur. Self ignition therefore occurs when the rate of heat production exceeds the rate of heat loss. The test procedure is useful as a preliminary screening test for powders.

**Test Method**
The bulk powder test is performed in a temperature programmed oven of 30 litres volume which is fitted with explosion vents and fan assisted air circulation. A glass cylinder of approximately 100 mm height and 50 mm diameter, closed at the base with a porous glass sinter and the top left open, to permit air diffusion is placed centrally in the oven. The oven temperature is continuously monitored by strategically placed thermocouples. Four sample thermocouples are used to detect any exothermic activity and provide an onset temperature. To (the temperature at which the sample temperature increases independently of the oven temperature). The temperature of the oven and sample are continuously recorded while the temperature of the oven is increased to 600°C or to the melting point of the solid, if lower, at a rate of 0.5 K min⁻¹. The test can also be conducted in isothermal mode where the oven temperature remains at a constant, set temperature.

**Results and Interpretation**
Exothermic decomposition and the ignition of powder layers most usually occurs by a process of self-heating due to atmospheric oxidation. When the temperature of a layer is raised sufficiently, then a condition can be reached where the heat generated by the exothermic reaction is sufficient to exceed heat losses, and a runaway increase in temperature develops. Powder in bulk, or thick layers, will ignite at a lower temperature than the same material in thin layers, because the surface area per unit mass, and hence the rate of heat loss per unit mass is decreased. Since ignition is initiated inside the bulk of powder there may be a considerable time delay before it becomes visible at the surface. The diffusion cell screening test simulates the conditions in hoppers, silos, bags or bulk powder dryers (i.e. any situation where natural diffusion of air through a powder can occur). The test can also be applied to conditions where product may build up in a drying process, resulting in bulk material or a thick layer forming for a long period of time. As the test has high heat losses, a safety margin of typically 50°C is applied to the exothermic onset temperature that has been determined from isothermal testing.

**Test Limitations**
The results are not directly scaleable and require a safety margin (typically 50°C). Due to the porous nature of the test cell base, any sample that melts cannot be studied beyond the melting point (owing to escape from the cell). Alternative tests for liquids should be considered in such cases.

**Reduced Versions of the Test**
No reduced version of this test exists.
### Appendix - Test Descriptions

**A12. Aerated Cell Screening Test**

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<thead>
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<th>Appendix A12. Aerated Cell Screening Test</th>
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<td><strong>Alternative Tests</strong></td>
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<tr>
<td><strong>Test Purpose</strong></td>
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<tr>
<td><strong>Test Method</strong></td>
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<tr>
<td><strong>Results and Interpretation</strong></td>
</tr>
<tr>
<td><strong>Reduced Versions of the Test</strong></td>
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<tr>
<td><strong>Test Limitations</strong></td>
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**A13. Air Over Layer Test**

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<td><strong>Alternative Tests</strong></td>
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<td><strong>Test Purpose</strong></td>
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<td><strong>Test Method</strong></td>
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<tr>
<td><strong>Results and Interpretation</strong></td>
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<tr>
<td><strong>Reduced Versions of the Test</strong></td>
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<tr>
<td><strong>Test Limitations</strong></td>
</tr>
</tbody>
</table>
Appendix A14. Determination of Safe Storage Temperatures for Bulk Materials (Basket Tests)

**International Standard**
BS EN 15188

**Quantity of Sample**
8 kg

**Alternative Tests**
None exists

**Test Purpose**
To determine the safe storage temperature for a bulk of powder in a specified container of any volume and geometry. The test permits natural diffusion of air through the sample and hence simulates conditions in large scale storage (e.g. in silos). The test results can be used to define transportation classification for potentially self-heating solids (e.g. UN Class 4, division 4.2).

**Test Method**
Cubic containers are made from stainless steel gauze. Normally three sizes of container are used of 25 mm, 50 mm and 100 mm side respectively. Sometimes materials do not behave linearly using the 25 mm basket and in these cases typical sizes are selected of 50, 75, 100 or even 150 mm side. Initially, the 50 mm basket is used as this provides a good indication of onset temperature with a fairly low usage of sample. The container is filled with material, the packing density measured and the basket is then suspended in the center of a fan assisted oven, held isothermally for a designated period of time (24 hours minimum). Thermocouples are placed in the center of the sample and between the sample container and the oven wall.

Observations are made as to whether an ignition has occurred, denoted by an increased rate of rise in temperature above that of the oven temperature or a no ignition situation, where the temperature may rise slightly (but at a decreasing rate) above the oven temperature before subsiding back to the oven temperature. The highest measured “no ignition” temperature (Tni) and the lowest measured “ignition” temperature (Ti) are determined to within a close margin (typically 3°C) as larger differences in temperature give larger errors when extrapolating the results.

**Results and Interpretation**
Using the results obtained from the testing of all three container sizes, a graph is plotted of log (V/SA), where V is the container volume and SA is the surface area of the container against (1000/T), where T is both Tni and Ti in K. This should produce 2 straight lines. A third line (average value) is then plotted between the Tni and Ti traces and this line is extrapolated on the graph to obtain safe storage temperatures for a vessel of any shape or size (providing the volume and surface area is known or can be calculated).

To calculate the induction time the Ti graphs for the three container sizes have to be examined to calculate the duration time before commencement of the exotherm (t). A graph is then plotted of ln(t) in seconds against log (V/SA). This will produce a straight line which can be extrapolated to provide an estimated induction time for a vessel of any shape or size.

**Reduced Versions of the Test**
Many options exist. These include the use of smaller baskets, fewer basket sizes (e.g. 2 instead of 3) and higher margins between ignition and no ignition events (e.g. 5 K instead of 3 K). All of these variables will decrease the accuracy of measurement and enhance errors in extrapolation.

**Test Limitations**
The test is not applicable to liquids or low melting solids. Samples which decompose (rather than oxidize) are more cost-effectively studied using alternative methods.

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