Safety Guide
Handling Dusts and Powders Safely

Contents

INTRODUCTION

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 plant 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 icing sugar, aluminium powder and even paracetamol. 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 potentially flammable powders and dusts across all industries, with the objective of helping operating companies minimise 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 plant 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 DEKRA 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 DEKRA UK 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 flammable dusts and powders.

Handling Dusts and Powders Safely

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
- Fines 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 materials present or a chemical reaction that may go out of control. These hazards are normally recognised when using common flammable liquids such as methanol and ethanol or flammable gases such as propane, butane or hydrogen but what about dust clouds, such as those formed from food ingredients (sugar, flour, etc.) or pharmaceutical products (aspirin, paracetamol, etc.)? Do these constitute a hazard? The answer is probably yes, as more than 70% of powders handled in industry are recognised as being flammable.

In order to address this issue and to ensure safe operating conditions for companies using or producing these materials, the European Union has implemented regulations that highlight the need to obtain process safety data in order to complete a compulsory risk assessment. These regulations include:

- The Chemical Agents Directive (CAD, 1998/24/EC).
- The Explosive Atmospheres Directives which include:
- ATEX 137 (1999/92/EC), implemented in the UK as the Dangerous Substances and Explosive Atmospheres Regulation (DSEAR) 2002, covering the minimum requirements to protect workers potentially at risk from flammable atmospheres.
- ATEX 95 (1994/9/EC), implemented in the UK as the Equipment and Protective Systems intended for use in potentially explosive atmospheres (EPS regulations covering equipment and protective systems intended for use in potentially explosive atmospheres.

The guide does not cover other topics such as occupational exposure or environmental issues (e.g. toxicity and eco-toxicity) nor does it detail process safety management practices or equipment selection.

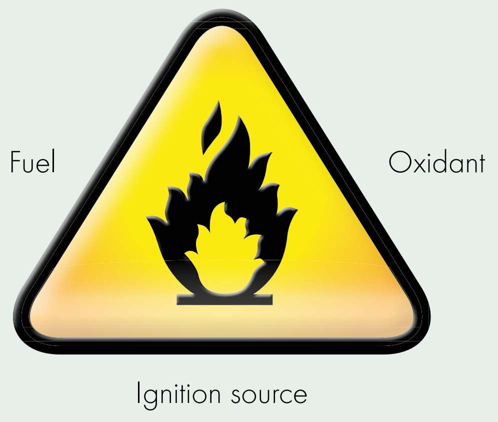
WHERE TO START?

What are the Conditions for a Fire or Explosion?

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 vapour (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").

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 oxidised). 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.

Conditions for a Fire



Conditions for an Explosion

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).

The safe processing of flammable materials (dusts, gases or vapours) is facilitated by either preventing the conditions required for an explosion to occur, or protecting the plant 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:

Avoidance of Ignition Sources

> Knowing the ignition sensitivity of the powder and ensuring that no potential source of ignition of sufficient energy to ignite the dust cloud exists in the process.

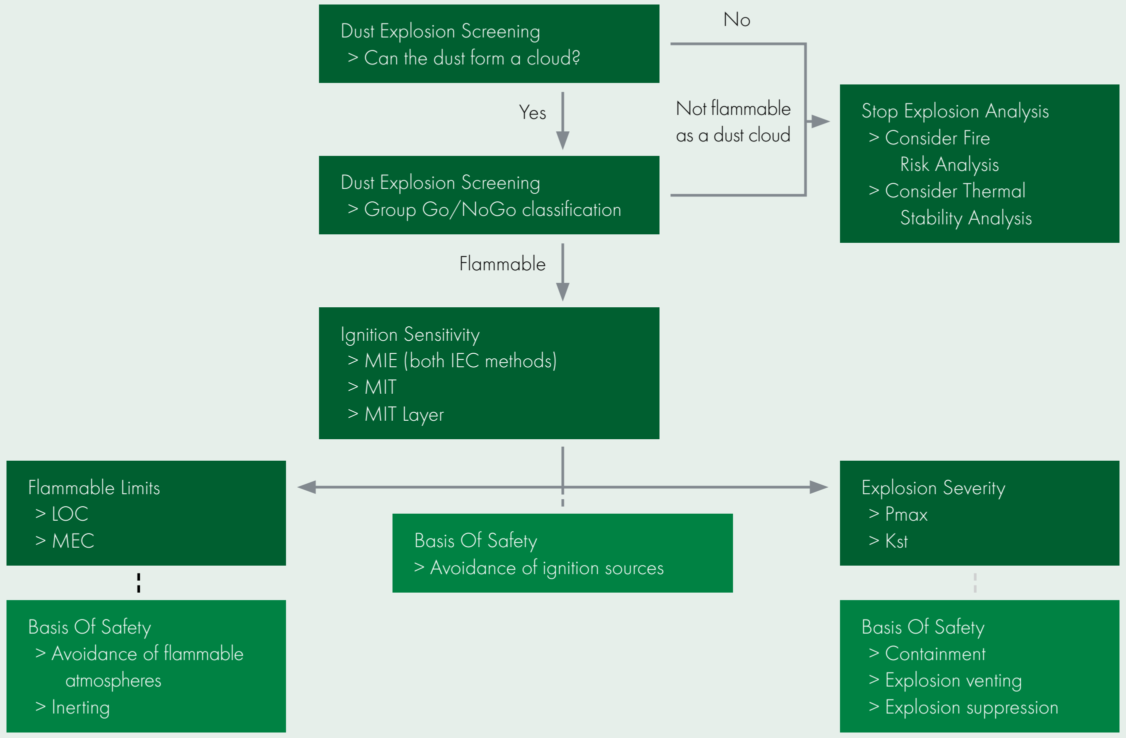
Prevention

> 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”)

Protection

> Explosion venting
> Explosion suppression
> Explosion containment

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



A complete explanation of the figure is given on page 13

Figure 1. Strategy for Dust Explosion Testing

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 behaviour, vapour pressure, oxidising capability, explosive properties and flammability. However whilst most of the gas and vapour flammability hazards are routinely addressed by the contents of an MSDS, the properties of dusts and powders are not so well covered. Occasionally, a powder may be noted as being “Not flammable but capable of forming an explosive atmosphere when dispersed as a cloud”. This statement is often confusing as powders can either be flammable as a powder layer or in the form of a dust cloud.

> **Evaluation of the Chemical Structure and Formula**
Explosive behaviour (in terms of detonation or rapid deflagration potential) is generally limited to certain energetic functional groups (such as azides, nitro-compounds, peroxides, etc.). The presence of any such group is a strong indication 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 powders[1] 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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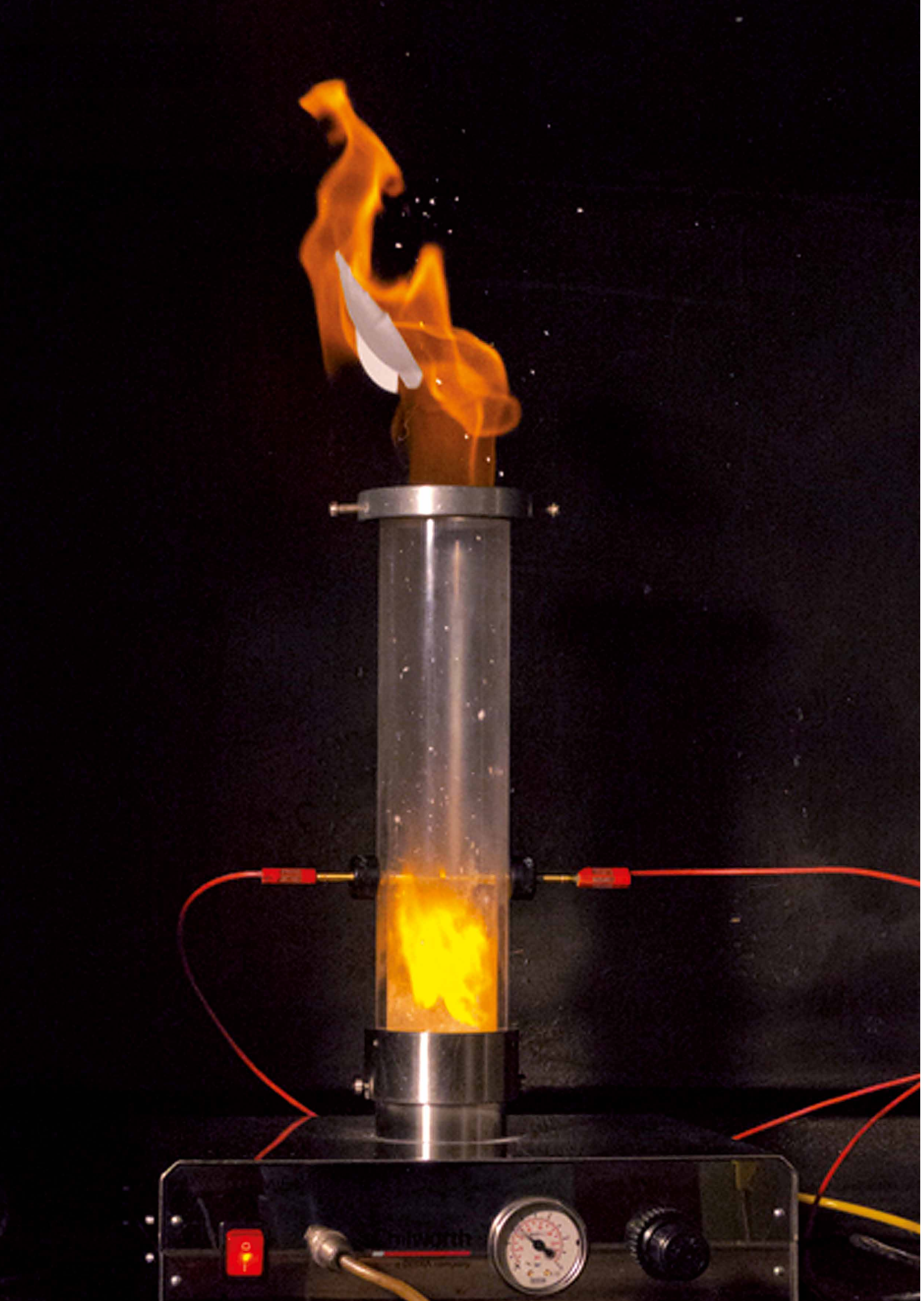
[1] For this document the words: “powder” and “dust” are synonymous



EXPLOSIVE PROPERTIES EVALUATION

The preliminary evaluation of explosive properties can (and should) be undertaken well before large scale manufacture commences.

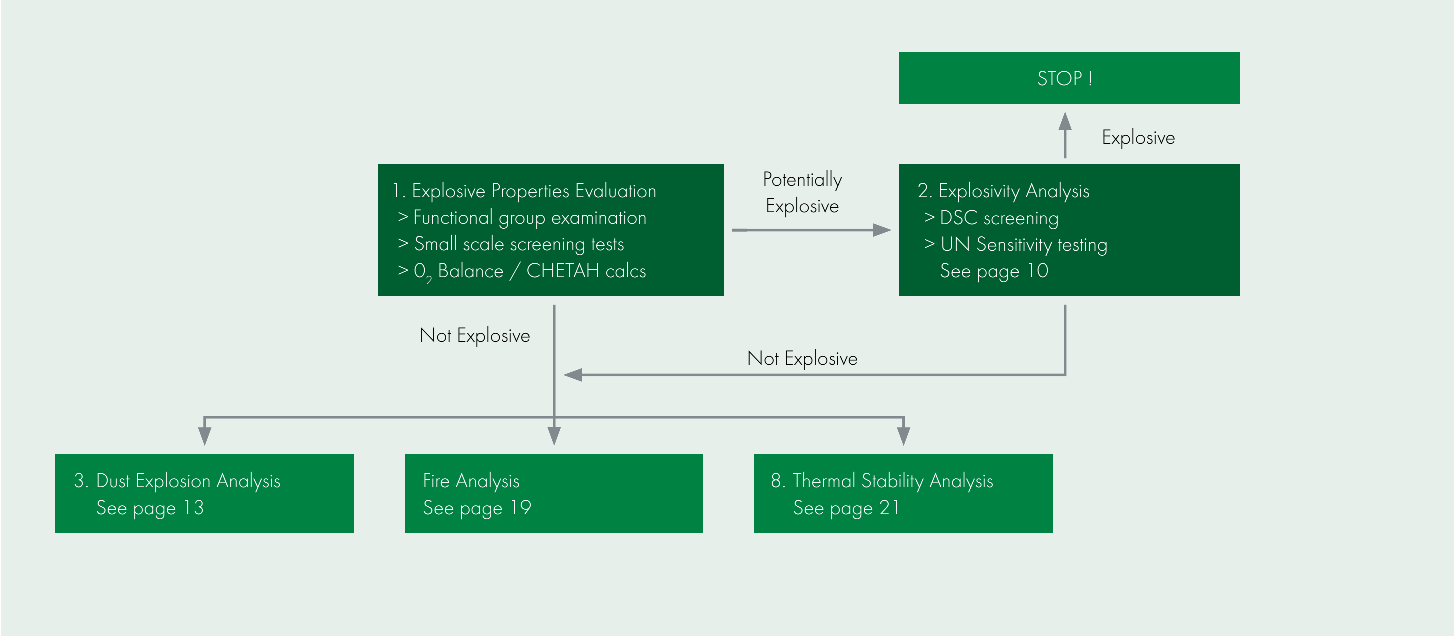


Figure 2. A Process Safety Strategy for Powders

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 behaviour (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

Explosivity Analysis

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-1, 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.

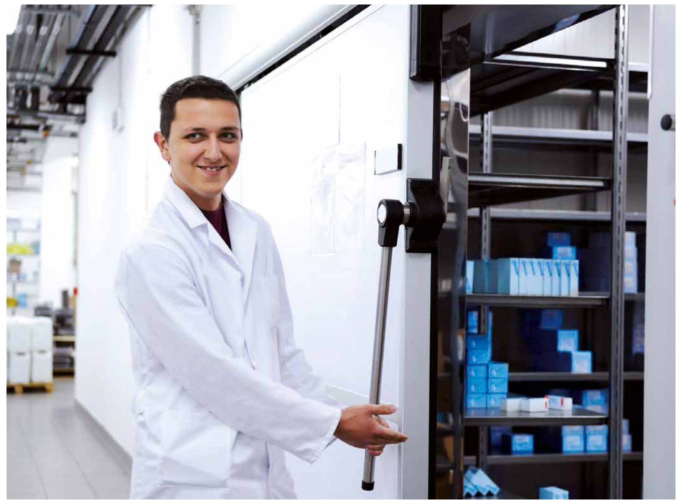
For transportation and classification purposes, tests to evaluate the severity of a detonation or rapid deflagration may need to be completed. These tests are again, normally undertaken using the UN Transport of Dangerous Goods Recommended test methods.

Differential Scanning Calorimeter

STOP! – Where Do We Go Next?

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.

DUST EXPLOSION ANALYSIS

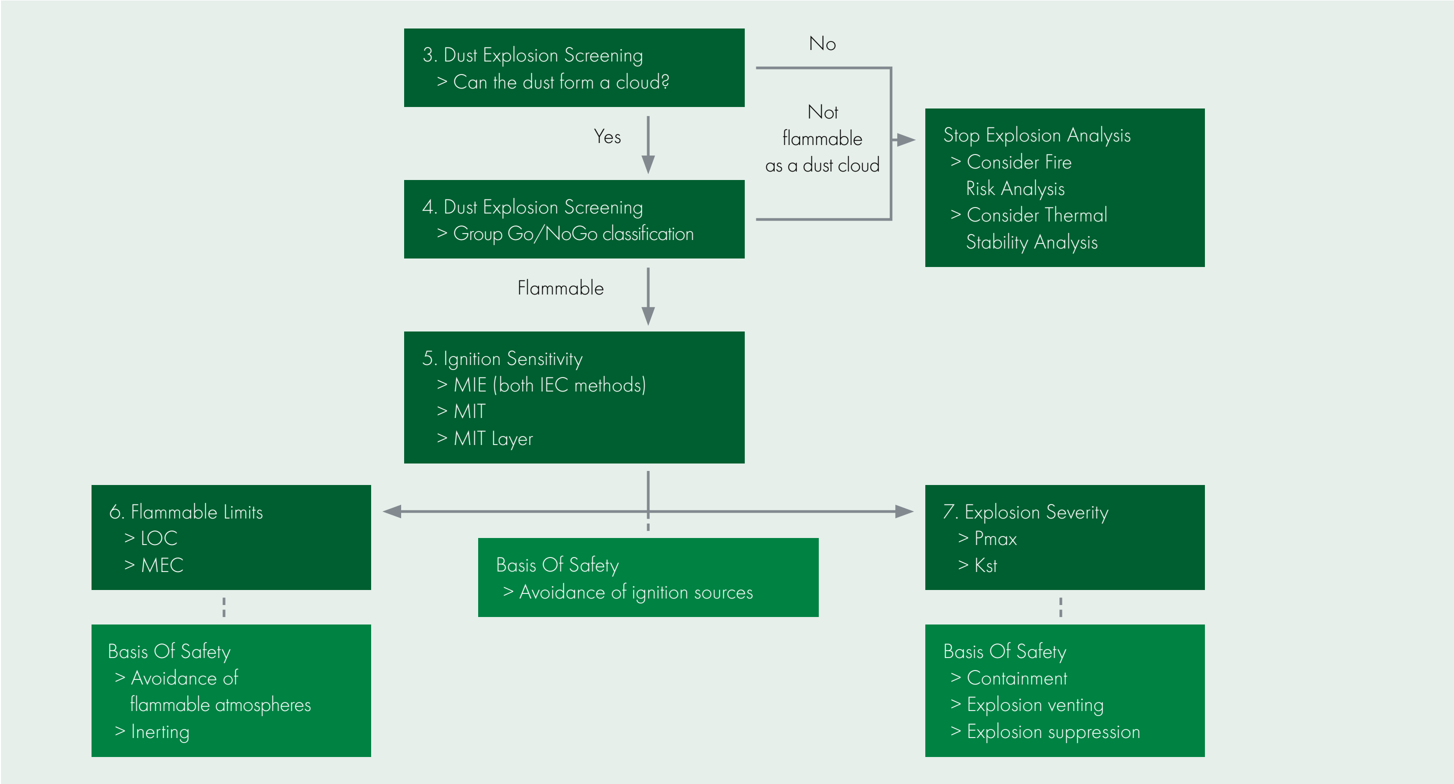


Figure 3. Strategy for Dust Explosion Testing

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 plant situation. Testing of unrepresentative samples will undoubtedly compromise the validity of the data.

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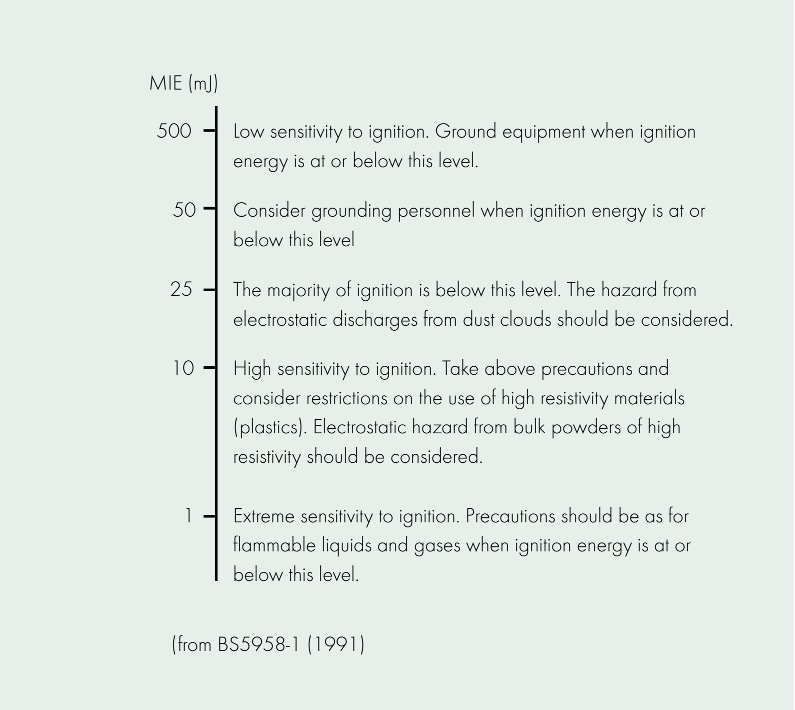
[2] For solvent damp powders, the flammability properties of the solvent must be considered (even if the dust cannot form a cloud). A basis of safety for the flammable atmosphere should still be specified. Data for the dust will be required for processes where the dust is dried and is capable of forming a cloud.

Dust Explosion Screening

If a powder is capable of forming a flammable dust cloud then there are numerous ways of protecting plant 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 UK, to perform a combustible dusts and combustible flyings determination test (see appendix A.2i), where a material may be classified as combustible or non-combustible. 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.2ii).

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 a flammability 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 combustible dusts and combustible flyings determination test. If after exhaustive testing, the material proves to be non-combustible (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.



MIE (mJ)	
500	Low sensitivity to ignition. Ground equipment when ignition energy is at or below this level.
50	Consider grounding personnel when ignition energy is at or below this level
25	The majority of ignition is below this level. The hazard from electrostatic discharges from dust clouds should be considered.
10	High sensitivity to ignition. Take above precautions and consider restrictions on the use of high resistivity materials (plastics). Electrostatic hazard from bulk powders of high resistivity should be considered.
1	Extreme sensitivity to ignition. Precautions should be as for flammable liquids and gases when ignition energy is at or below this level.

(from BS5958-1 (1991))

Figure 4. Guide to Electrostatic Precautions for Powders

Minimum Ignition Temperature Test (MIT)

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3 Group NoGo classification is made if the material cannot be ignited by a high energy spark discharge, glowing hot coil or hot surfaces at up to 1000°C. However, it should be remembered that thermal stability and fire risks still need to be assessed even if a material is classified as Group B in the form of a dust cloud.

Ignition Sensitivity

In the case of a positive result in the combustible dusts and combustible flyings determination, the ease with which a powder can be ignited is determined (see Box 5 of Figure 3). Potential ignition sources can include electrostatic discharges, mechanical (grinding / frictional) sparks or hot surfaces.

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 inductor into the circuit (the inductive method). The inductive method uses a 1 mH inductor in the ground loop which results in the extended duration of the spark discharge. The IEC 61241-2-3 / EN 13821 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 precautions 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 Layer Ignition Temperature (LIT, 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 30 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 ignition from hot surfaces (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 Layer Ignition Temperature (LIT) of a layer of powder (nominally 5 mm). Under the latest European 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 LIT), then avoidance of ignition sources may not be acceptable as the ultimate Basis of Safety. In such cases, ignition sources are identified and minimised 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)

If the avoidance of ignition sources does not reduce the risk of an explosion to a sufficiently low or acceptable level, then an alternative Basis of Safety is required. In the first instance, a means of explosion prevention should be sought (see Box 6 of Figure 3). Typically, this could involve working outside of the flammable limits of the dust or working under an inert or partially inerted atmosphere.

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 nonuniformity 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 nonflammable. Without this necessary data, very low levels of oxygen must be established, controlled and monitored. These data are usually generated using the 20L Sphere apparatus.

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 plant operatives.

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 pressurisation 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.

Whichever 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 of 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 characterised 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 LIT of the dry powder should be determined and the results compared to the Auto-Ignition Temperature (AIT) of the solvent - the limiting value 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 LOC 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. It only takes a few 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 fine powder cannot accumulate “under all foreseen 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 vapour then the dust cloud ignition sensitivity values (MIE, MIT & LIT) 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 LOC 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 dual 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 DEKRA document “A Guide to Process Safety”.

FIRE ANALYSIS

Table 1.
Combustibility Class Table - German "Burning Number" (BZ)

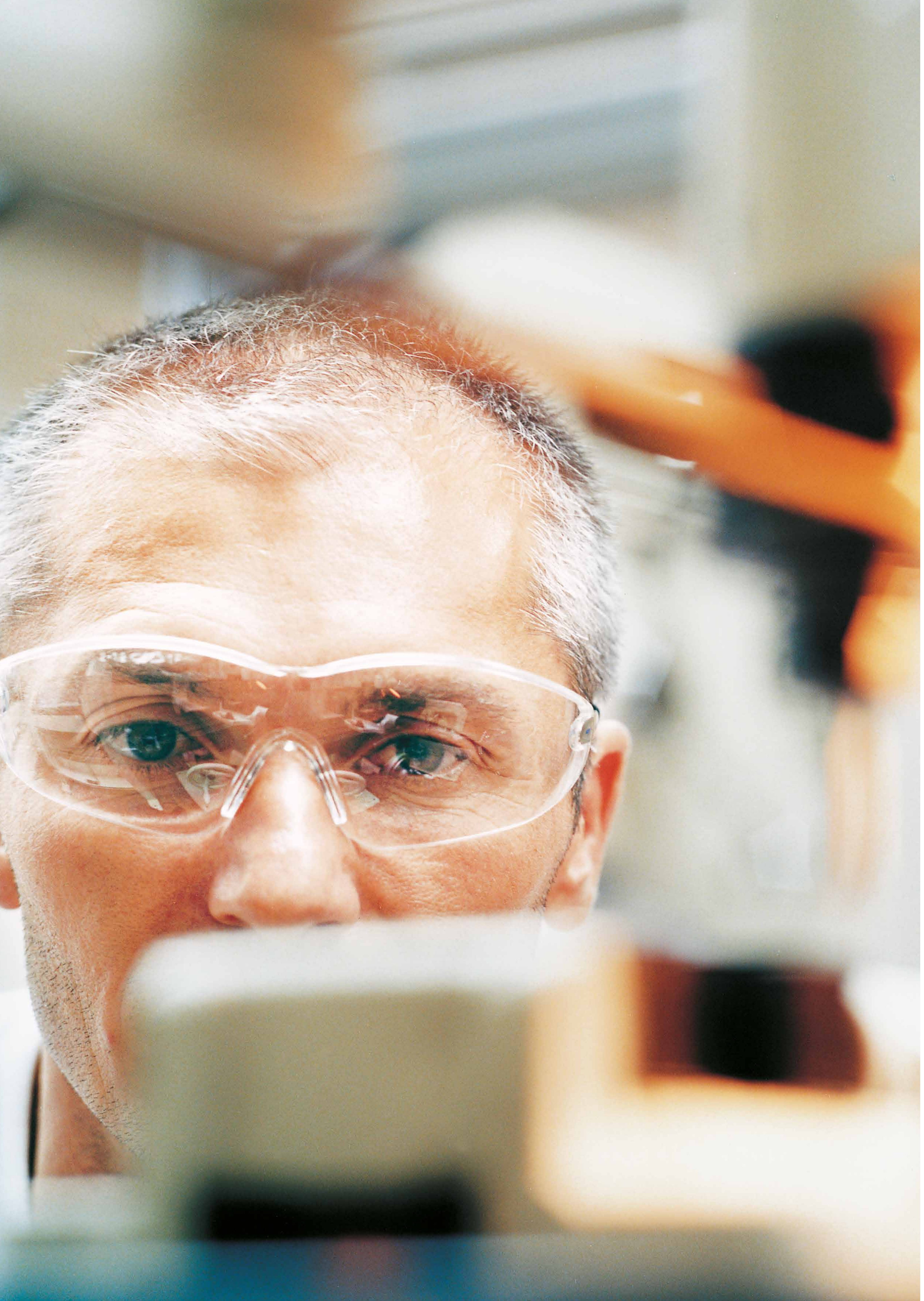
1 - No ignition
2 - Ignition and rapid extinction
3 - Local smouldering or burning
4 - Propagation of smouldering
5 - Propagation of open fire
6 - Flash fire

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 localised 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 Behaviour 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 localised 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 Behaviour 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.

VDI Burning Behavior Test Set-up



THERMAL STABILITY ANALYSIS

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 in 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 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 (typically <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 characterised 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.

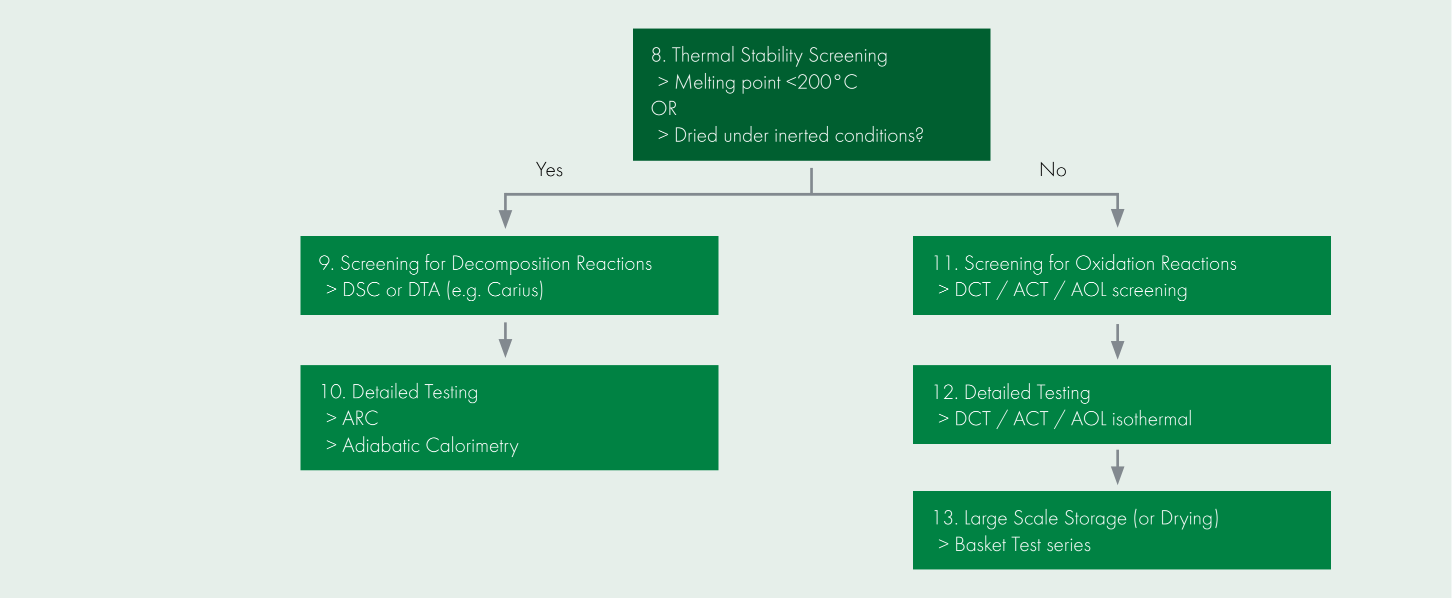


Figure 5. A Strategy for Thermal Stability Assessment of Powders

Testing for Potential Molecular Decomposition Reactions

Screening

Conventional Differential Scanning Calorimetry (DSC) or Differential Thermal Analysis (DTA) methods should be used to identify decomposition events for liquids, solids or mixtures (see Box 9 of Figure 5). These tests use very small scale, contained methods with limited air availability and are suitable for the examination of pure decomposition or self-reaction events only. The preliminary tests require a substantial safety margin to be applied (e.g. 50°C for Carius Tube (DTA) tests and up to 100°C for DSC tests).

> The DSC, whilst providing onset temperature (T0) and heat of reaction (3Hdec) data does not yield any information on gas generating events.

> The DTA methods generally provide information on gas generating events, as well as onset temperature, but do not fully quantify the magnitude of an exothermic event, other than differentiating between major and minor activity.

If the onset temperature determined in either of these tests for an exothermic or gas generating event (corrected using the appropriate safety margin) is below the maximum possible processing or exposure temperature of the material (Tmax), then more detailed testing is required. Testing can cease at this stage if the onset temperature is an adequate margin above Tmax.

Detailed Testing

If more sensitive analysis is required, adiabatic calorimetry is typically employed (see Box 10 of Figure 5). A range of commercially available calorimeters can be used for detailed thermal stability analysis. The most common test methods include Accelerating Rate Calorimetry (ARC) and adiabatic Dewar methods. The safety margins required for such methods range from 10°C (for the most sensitive methods) to 30°C (for ARC tests). The UN test methods to determine the Self Accelerating Decomposition Temperature (SADT) of a material can also be used. This approach requires no safety margin as the tests are intended to directly simulate the heat loss conditions of commercial scale packages or vessels.

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 $1m^3$. 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. Fluidised 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 Test (AOL)**
This test involves examining the thermal stability of thin layers of powder where air is readily available. The data can be used when assessing tray drying situations, the tops of spray driers or powder layer build-up in ducts and other process plant.

With all of the test methods, screening tests are initially recommended, where the sample temperature is ramped 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 selfheating 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 $1m^3$ 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 volume. 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.

EQUIPMENT SPECIFIC TEST RECOMMENDATIONS

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 plant 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

Process Description	Test Data Required					
	MIE	MIT	MIT Layer	20 litre	LOC	Thermal
Charging of vessel	Yes	Yes	Yes			
Discharging of vessel	Yes	Yes	Yes			
Blending	Yes	Yes	Yes	Yes	Yes	
De-agglomeration	Yes	Yes		Yes	Yes	
Milling	Yes	Yes	Yes	Yes	Yes	
Compaction	Yes					
Compression	Yes					
Capsule filling	Yes					
Granulation	Yes			Yes	Yes	
Tray drying	Yes			Yes	Yes	Yes
Microwave dryers	Yes			Yes	Yes	Yes
Fluid Bed drying	Yes			Yes	Yes	Yes
Flash or Ring dryers	Yes				Yes	Yes
Spray Drying	Yes	Yes		Yes	Yes	Yes
Spray coating	Yes			Yes	Yes	
Pneumatic conveying	Yes					
Filters	Yes	Yes		Yes	Yes	Yes
Storage (silo etc.)	Yes			Yes	Yes	Yes

It should be recognised 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 type of thermal stability tests that may be required for different types of dryer:

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

Type of Dryer	Air Over Layer	Aerated Cell	Diffusion cell	DSC
Spray	Yes		Yes	Yes
Fluid Bed		Yes	Yes	Yes
Vacuum			Yes	Yes
Tray	Yes		Yes	Yes
Flash or Ring	Yes		Yes	Yes

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.

SUMMARY

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.

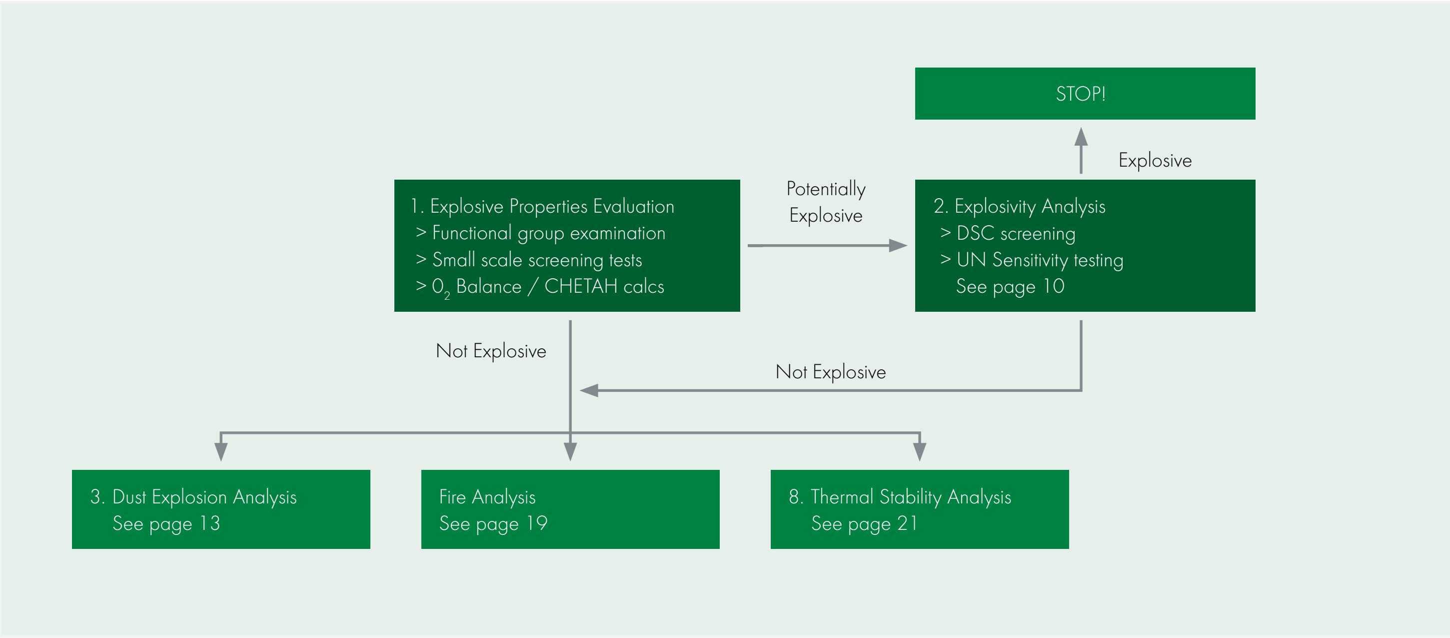


Figure 6. A Process Safety Strategy for Powders

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 signedoff 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 large scale plant as closely as possible. While many parameters can be obtained from standard tests, others may require innovative, bespoke testing solutions. DEKRA 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 plant design criteria for a range of flammability/thermal stability properties without having to perform a detailed analysis for each property.

- **Combustible Dust Risk Overview**
  This package is a highly cost effective way of screening for seven hazardous properties (including MIE, MIT, LIT, explosion severity, burning behaviour, thermal stability and flammable gas generation potential). The results are displayed in tabular and graphical form to enable easy understanding of the data. The package initially provides an overview of the properties but if a material is shown to be of high risk in any one of the properties then extended testing can be performed to provide detailed characterisation.

- **DustScreenTM**
  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. DustscreenTM has been developed specifically for companies utilising common process with a variety of powders.

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At DEKRA, 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.

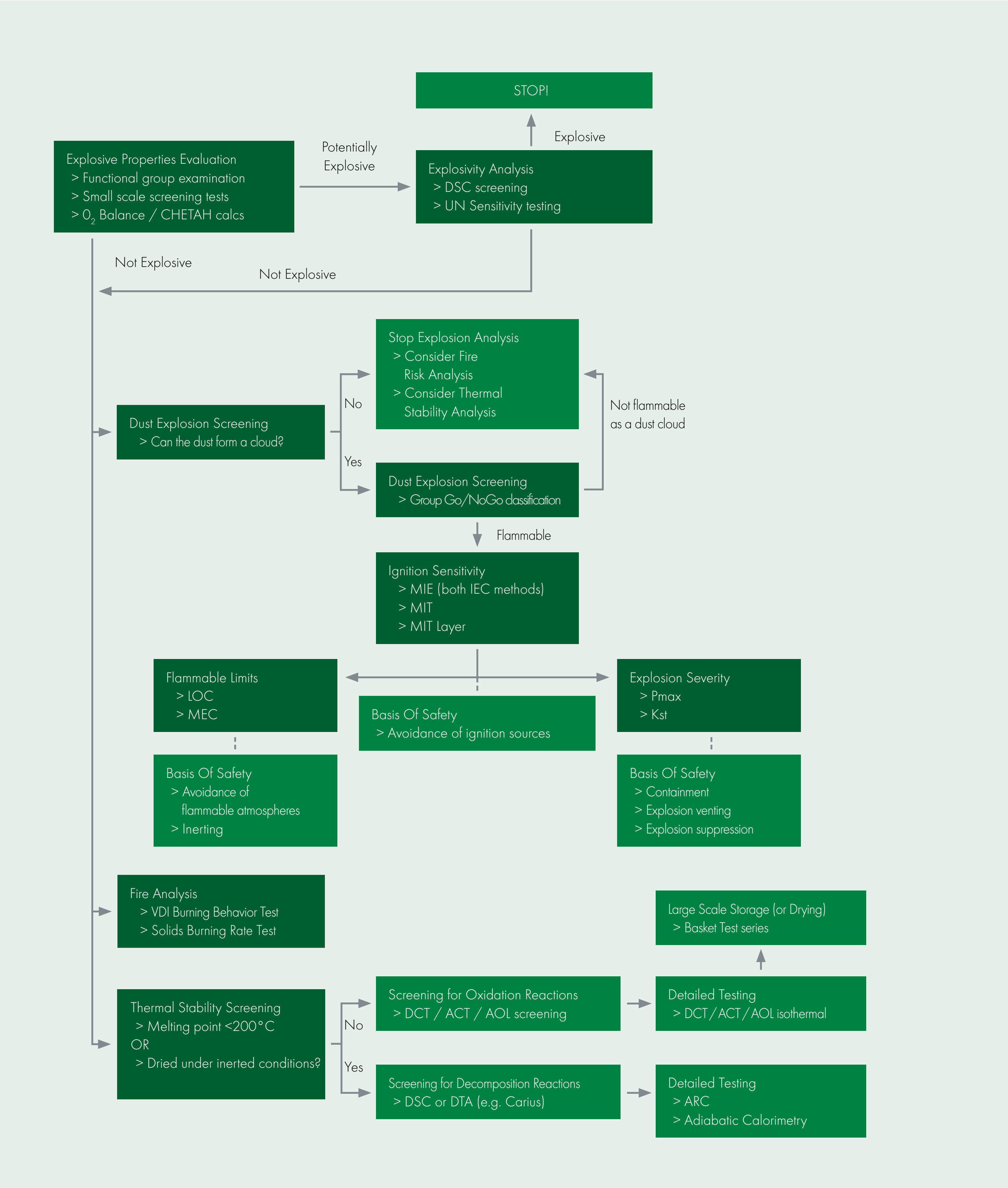

Figure 7. Strategy for Dust Explosion Testing

APPENDIX - TEST DESCRIPTIONS

A1. The DEKRA Process Safety Approach To Testing

DEKRA Process Saftey can perform a wide variety of process safety tests examining explosion hazards of dusts, vapours 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. DEKRA Process Saftey 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. DEKRA Process Saftey 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 (SOP's).
- 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 on 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 <10% moisture content and, where the standard methods dictate, less than a specific particle size (typically <63 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 DEKRA Process Saftey, 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 DEKRA Process Saftey 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.
- DEKRA Process Saftey 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. DEKRA Process Saftey recognises 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 A2i. Group Go/NoGo Classification Test

International Standard	ASTM E1226
Quantity of Sample	100 g
Alternative Tests	20l Sphere Test, modified Hartman tube test
Test Purpose	To examine if a dispersed powder can present an explosion hazard under ambient conditions and at elevated temperature
Test Method	The 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 litre 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. The dust is initially tested in the "as received" condition at a range of concentrations. 2. If "no ignition" is observed then the sample is sieved and the finest fractions are re-tested as in (1) above. 3. 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. 4. 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 fire risk and thermal stability characteristics may still need to be assessed.
Reduced Versions of the Test	No reduced version of this test exists
Test Limitations	The test is not suitable 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 20l sphere test that uses a higher (10kJ) energy ignition source.

Appendix A2i. Group Go/NoGo Classification Test

International Standard	Kuhner operating manual
Quantity of Sample	200 g
Alternative Tests	Group Go/NoGo Classification
Test Purpose	To examine if a dispersed powder can present an explosion hazard under ambient conditions
Test Method	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 1 kJ chemical igniters. The pressure/time record for each explosion is recorded using 2 x piezo electric pressure transducers 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.
Results and Interpretation	A dust which cannot be induced to explode over a wide range of concentrations with an ignition energy of IE = 1 x 2.5 kJ and/or 1 x 5.0 kJ and/or 2 x 5.0 kJ (chemical igniters) is classified as not explosible. This means that most probably the dust cannot be exploded at all, except by application of even stronger ignition sources
Reduced Versions of the Test	No reduced version of this test exists
Test Limitations	The test is for solid samples only and is not applicable for gases and vapours.

A3. Minimum Ignition Energy Test (MIE)

Appendix A3. Minimum Ignition Energy Test (MIE)

International Standard:	ASTM E2019, BS EN ISO/IEC 80079-20-2
Quantity of Sample:	250 g
Alternative Tests:	-
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 litre 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 can 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 (mJ) 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 vapour (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)

Appendix A4. Minimum Ignition Temperature Test (Cloud)	
International Standard:	ASTM E1491, BS EN ISO/IEC 80079-20-2
Quantity of Sample:	200 g
Alternative Tests:	BAM Furnace
Test Purpose:	To determine the minimum temperature of a hot surface capable of causing ignition of a dust cloud
Test Method:	The MIT test is conducted in the Godbert Greenwald furnace. The furnace consists of a vertically mounted vitreosil tube which is open at the base. A glass observation chamber is mounted at the top of the vitreosil 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 micron) 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.
Results and Interpretation:	The Minimum Ignition Temperature, of a dust cloud, is recorded as the lowest temperature of the furnace at which ignition is obtained, minus 10°C for furnace temperatures. Information from 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, 2/3rds 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 layer (typically assumed to be 5 mm maximum).
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 450, 300, 200, 135°C)
Test Limitations:	The test is for solid samples only and is not applicable for gases and vapours (the auto-ignition temperature test is available for gases and vapours)

A5. Minimum Ignition Temperature Layer (MIT Layer)

Appendix A5. Minimum Ignition Temperature Layer (MIT Layer)	
International Standard:	ASTM E2021, BS EN ISO/IEC 80079-20-2
Quantity of Sample:	500 g
Alternative Tests:	N/A
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 ring and by weighing the dust ring 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-1 up to a maximum temperature of 400°C. All information is collected using a computer controlled data logging system. The resultant computer trace is analysed 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-heated without igniting (glowing). 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 400°C, tests are repeated at 200 and 300°C to confirm the no ignition result.
Results and Interpretation:	The MIT Layer 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 enclosure 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 450, 300, 200, 135°C)
Test Limitations:	The standard test examines 5 mm layers only (thicker layers may produce a lower result)

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

Appendix A6. Dust Explosion Severity Test (20 Litre Sphere Test)	
International Standard:	ASTM E1226, EN 14034 and Kuhner operating manual
Quantity of Sample:	750 g
Alternative Tests:	1 m3 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. The pressure / time record for each explosion is recorded using 2 X piezo electric pressure transducers 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/dtmax. 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
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). The test considers ambient temperature and pressure conditions only. Hybrid mixture (dusts admixed with flammable gases) can be studied in the 20 l sphere apparatus with minor modification of the procedure.

A7. Limiting Oxygen Concentration Test (LOC)

Appendix A7. Limiting Oxygen Concentration Test (LOC)	
International Standard:	ASTM WK1680, EN 14034-4 and Kuhner Operating Manual
Quantity of Sample:	500 g (or 1000 g if also measuring explosion severity)
Alternative Tests:	1 m3 Sphere Test
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 analyser. Explosion trials are then performed at various (known) oxygen levels. It should be noted that the ignition source utilised in these tests are 1 x 2.5 kJ chemical ignitors 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 e.g. 10, 12, 14 and 16% oxygen)
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). The test considers ambient temperature and pressure conditions only.

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

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

International Standard:	ASTM E1515, EN 14034-3 (2005) and Kuhner Operating Manual
Quantity of Sample:	150 g
Alternative Tests:	1 m3 Sphere Test
Test Purpose:	To determine the minimum concentration of a dust at which ignition is possible
Test Method:	Minimum Explosive Concentration (MEC) is measured using the 20 litre sphere apparatus.
Results and Interpretation:	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 localised flammable concentrations even though the mean concentration of the dust cloud may be below the MEC.
Reduced Versions of the Test:	Screening test (at pre-defined levels e.g. 125, 75, 50, 25 g.m-3)
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). The test considers ambient temperature and pressure conditions only.

A9. Differential Scanning Calorimetry (DSC)

Appendix A9. Differential Scanning Calorimetry (DSC)

International Standard:	Several ASTM standard methods for applications of DSC exist
Quantity of Sample:	50 mg
Alternative Tests:	-
Test Purpose:	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. Due to the very small sample size, the use of this technique in the study of reaction mixtures or heterogeneous samples is not recommended due to the difficulty involved in representative sampling (the Carius tube is a more appropriate technique in such cases).
Test Method:	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 evapourative 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
Results and Interpretation:	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 normalised 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 >800 J.g-1 indicates potential for explosive properties to exist in the material.
Reduced Versions of the Test:	No reduced version of this test exists
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. 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 representatively sampling a blend at such low masses.

A10. Carius (10 g) Tube Screening Test

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 behaviour 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 centre 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 of assess the nature of the gas).
Reduced Versions of the Test:	No reduced version of this test exists
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.

A11. Diffusion Cell Screening Test

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 400°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 exotherm onset temperature that has been determined from isothermal testing.
Reduced Versions of the Test:	No reduced version of this test exists
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.

A12. Aerated Cell Screening Test

Appendix A12. Aerated Cell Screening Test

International Standard:	This test meets the specification in the 1990 IChemEbook "Prevention of Fire & Explosions in Dryers" pages 16 - 18 and is considered current best practice
Quantity of Sample:	150 g
Alternative Tests:	None exists
Test Purpose:	The purpose of this test is to simulate the conditions in dryers (or similar situation) in which an air stream passes through the a bulk of material (e.g. fluid bed or rotating drum dryers). 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 Aerated powder test is performed in a temperature programmed oven of 30 litres volume which is fitted with explosion vents, the oven has fan assisted air circulation. A glass cylinder of approximately 100mm height and 50mm diameter, closed at the base and fitted with a sintered glass lid which allows air to be passed downwards through the test cell. Pre-heated air is passed downwards through the cell at 0.6 l.min-1. 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, air flow and sample are continuously recorded while the temperature of the oven is increased to 400°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:	As in the Bulk Powder (diffusion cell) test, 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. Increased air availability through the material may also affect the onset temperature (up or down) and the severity of the exothermic decomposition. The aerated cell screening test simulates process conditions where air passes through powder and can provide a more representative onset temperature and indication of severity of exothermic decomposition in these situations. A safety margin of typically 30°C is applied to the onset temperature of reaction based on isothermal ignition tests (for aerated environments). For static powder environments, the Diffusion cell test is more appropriate. It is commonly recommended that both tests should be considered to cover both normal and abnormal operating conditions in these types of driers. A difference of up to 50°C for To has been observed with some materials for the 2 tests.
Reduced Versions of the Test:	No reduced version of this test exists
Test Limitations:	The results are not directly scaleable and require a safety margin (typically 30°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.

A13. Air Over Layer Test

Appendix A13. Air Over Layer Test

International Standard:	This test meets the specification in the 1990 IChemEbook "Prevention of Fire & Explosions in Dryers" pages 18 - 20 and is considered current best practice
Quantity of Sample:	50 g
Alternative Tests:	None exists
Test Purpose:	The purpose of this test is to simulate the conditions in dryers such as spray, cross flow, band and tray driers in which hot air passes over a thin layer of material. The test can also be used to simulate the conditions of deposits on the internal surfaces of any type of equipment operating at elevated temperature. If heat developed by a reaction of the substance with oxygen or by exothermic decomposition is not lost rapidly enough to the surroundings, self heating leading to self ignition can occur.
Test Method:	The powder layer test is performed in a temperature programmed horizontal furnace of 70 mm diameter and 300 mm in length. Sample is placed in a metal tray (75 mm x 40 mm x 15 mm deep) and the filled tray is inserted into the oven. Heated air passes over and around the sample with a velocity of 4.5 m.min-1. The layer depth can be matched to the industrial application up to a maximum depth of 25 mm. Thermocouples are used to monitor and record the temperature within the sample, oven and air flow as the temperature of the oven is increased to 400°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:	The minimum ambient temperature for self-ignition of a layer depends on a number of parameters including the nature of the layer, its depth and time of exposure to heat. If these are predictable, then a reliable assessment of the hazard can be made by small scale laboratory testing. The Air Over Layer screening test alone is never used to provide maximum safe operating temperatures for driers. However, using the information obtained from the screening test, a series of isothermal tests can be performed where the sample is maintained at a constant temperature for a period exceeding the required drying temperature and time. If necessary, the isothermal tests are performed at progressively lower temperatures until no self-heating takes place. A safety margin of typically 20°C is applied to the onset temperature of reaction based on isothermal ignition tests (for powder layer environments). For static bulk powder environments, the Diffusion cell test is more appropriate whereas for air through powder purging, the Aerated Cell Test is more appropriate.
Reduced Versions of the Test:	No reduced version of this test exists
Test Limitations:	The results are not directly scaleable and require a safety margin (typically 20°C). The sample tray is not completely leak proof and therefore if any sample melts, it cannot be studied beyond the melting point (owing to escape from the cell). Alternative tests for liquids should be considered in such cases. Layer thickness is a critical parameter in this test and the layers studied must be representative of the industrial situation.

A14. Determination of Safe Storage Temperatures for Bulk Materials (Basket Tests)

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 150mm 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 centre of a fan assisted oven, held isothermally for a designated period of time (24 hours minimum). Thermocouples are placed in the centre 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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