SHAPA TECHNICAL PAPER 16

Dust Testing
for
DSEAR and ATEX Compliance

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This document provides an introduction to dust explosion testing and its importance in making process plant safe from fire and explosion. It describes the standard laboratory tests and the parameters they measure, enabling plant engineers and system designers to make informed judgement on safety matters such as: what is safety critical information and what is simply "interesting to know"; whether explosion and ignition information can be reliably taken from published data; is data required for every dust and powder being handled, or can "worst case" materials be identified?

Dust explosions are fortunately rare occurrences, but they have the potential to kill and legislation under the Health & Safety at Work Act 1974 and the Dangerous Substances and Explosion Atmospheres Regulations 2002 is in place to ensure safety in the workplace.

UK Statistics – fire and explosion

Analysis of dust fires and explosions reported to the Health & Safety Executive over a ten year period show 295 events (being a combination of fire, explosion and fire & explosion incidents) and the results are summarised in Figure 1 below, with 41 explosions resulting in injury or death.

Figure 1 – no. of reported UK incidents

Figure 2 – breakdown by industry

Figure 2 shows a breakdown by industry of the explosion incidents. The most common ignition sources
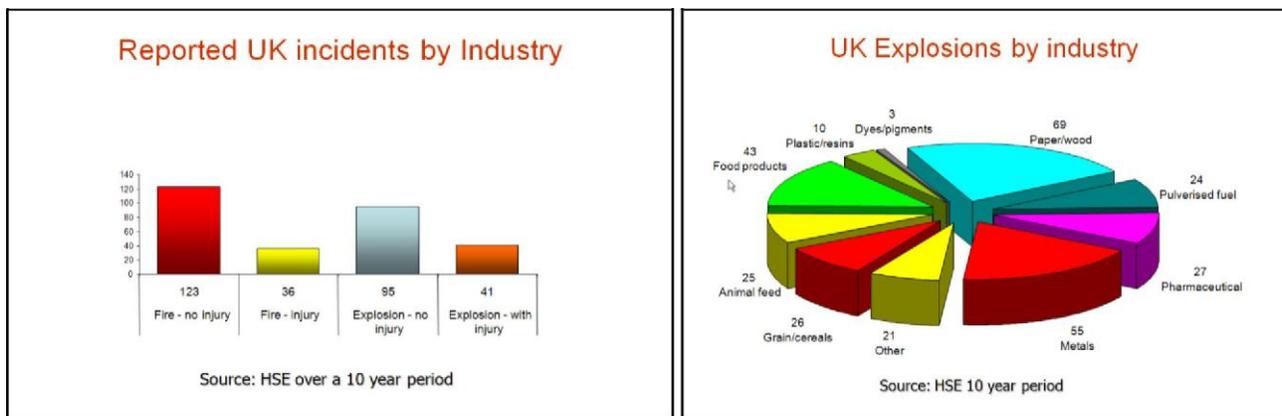
identified were mechanical sparking, smouldering dust deposits and frictional heating.

What is an explosion?

An explosion is a freely travelling combustion front and can be thought of as a fast fire. When confined, the combustion front moves at a relatively slow speed (typically 10 metres per second, but strongly material dependent), yet generates a rise in pressure that will travel at the speed of sound (330 metres per second).

Explosions have a characteristic pressure/time relationship with a substantial portion being uniform i.e. a constant rate of pressure rise. When the combustion front reached the vessel walls, it has consumed all the available fuel or oxygen and self-extinguishes, resulting in a maximum pressure attained. Laboratory experiments demonstrate that this pressure, known as the maximum explosion pressure or Pmax, is independent of the vessel volume.

Rates of pressure rise are dependant on volume; the larger the volume, the slower the pressure rise. The cube law accurately describes the relations between volume and rate of pressure rise.

K_{ST} = explosion constant for dust K_G = explosion constant for gas or vapour

A number of conditions must be satisfied simultaneously for a dust explosion to occur:

1. the dust must be combustible
2. the dust must have a particle size distribution that will propagate flame
3. the dust must be in suspension in the atmosphere with sufficient oxygen to sustain combustion
4. the dust concentration must be within the explosible range
5. there must be present an ignition source of sufficient energy to initiate the explosion

Terminology

Dust explosions may be classified as being either primary or secondary in nature. Primary dust explosions occur inside process plant or similar enclosures and are generally controlled by pressure relief through purpose-built ducting to atmosphere. Secondary dust explosions are the result of dust accumulation inside the factory being disturbed and ignited by the primary explosion, resulting in a much more dangerous uncontrolled explosion inside the workplace.

Historically, fatalities from dust explosions have largely been the result of secondary dust explosions. The two photographs below show the aftermath of a large secondary dust explosion that occurred at Imperial Sugar, Georgia on February 8th, 2008, killing 14 and injuring 36.

The official investigation into the incident by the US Chemical Safety Board can download from the CSB website www.csb.gov.

Secondary explosions may be avoided by good housekeeping and appropriate process control, stopping dust from leaking, implementing good cleaning regimes and keeping the factory clear of dust.

There is debate about how thick a layer of dust is needed to support a secondary explosion, but a simple test is to walk through it – leaving footprints is an indication of secondary explosion hazard. Accumulations of dust on ledges, tops of bins etc. should not be tolerated.

Secondary dust accumulation
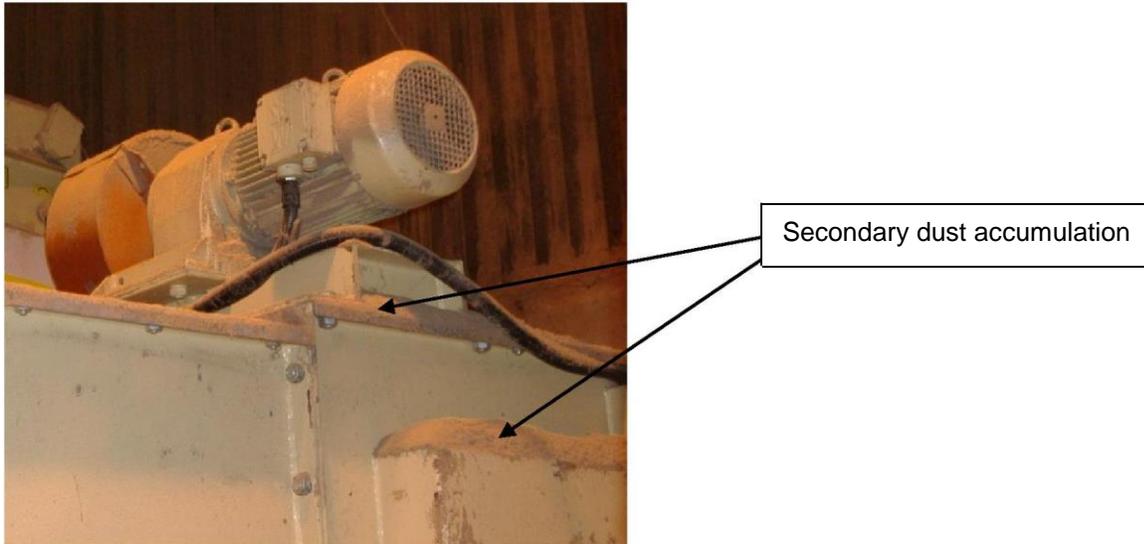
Laboratory Tests

A number of laboratory tests have been developed and refined over many years to characterize the explosion and ignition potential of dusts. The tests described here are designed to simulate plant operation under "worst case" conditions that will provide a single value for the test material that can be applied across a wide range of plant processes and equipment. For example, the 20 litre sphere test apparatus induces a dust turbulence that is similar to that found inside a hammermill. Conditions inside (say) a hopper will be considerably less onerous, but the lower explosibility is simply absorbed as additional safety margin to simplify the testing procedure. Occasionally, plant conditions may exceed that induced in the standard test and a more strenuous test may be required, as in the case of a jet mill micronizer or a contained explosion forcing jet flame ignition into an adjoining vessel. Under these circumstances, seeking specialist advice is recommended.

Test standards are continually under development and it is recommended that testing is undertaken to the latest applicable EN standard wherever possible.

The Classification Test

The classification test provides a qualitative assessment of whether or not a suspended dust is capable of initiating and sustaining an explosion in the presence of small sources of ignition. The test may be carried out in either the Hartmann Tube or 20L sphere apparatus.

An explosible dust - termed "Group A" - will propagate flame away from the ignition source.

A non-explosible dust - termed "Group B" - will not propagate flame away from the ignition source.

There is currently no European standard for classification, but one is in preparation.

NOTE: Experience has shown that a small number of materials classified as Group B in the Hartmann Tube may indeed be capable of supporting combustion at higher energies than those used in the standard test (hot coil & 10kV spark). Should higher energy ignition sources, or temperatures, be present or suspected in the industrial application, classification must be continued in the 20L sphere and MIT furnace – see pages 4 & 8.

Sample preparation
The dust is tested 'as received", unless the particle size and moisture content are not known on plant, in which case the sample is dried at 105°C for 1 hour and sieved below approximately 63μm to investigate the flammability of a fine dust fraction.

Test method
Initially a small amount of dust, typically 0.5 g, is placed in the dispersion cup around the mushroom shaped deflector and it is then suspended around the ignition source either by an instantaneous or continuous blast of air.

If flame propagation is observed the dust is regarded as explosible and the test ended. If flame propagation does not occur, the amount of dust and the dispersion conditions are varied followed by drying and sieving of the material sample to encourage flammability.

Ignition sources
Tests are conducted in two series of trials with different sources of ignition, being electric spark and hot coil. The electric spark is produced by a high voltage transformer having a 10kV, 0.024A output, the spark bridging a 0.5cm gap between a pair of ignition electrodes. This provides a spark having an energy in the region 8-10J. The hot coil is made from 20SWG Kanthal 'A" wire and consists of approximately 8 turns having an external diameter of 1cm. It is heated to a bright red heat (approximately 800 °C) by a transformer having an output of 10V and 20A.

Test criterion
The criterion for explosion is the observation of flames, no matter how small, moving away from the ignition source. Sparks or smoke from particles landing on the hot coil are ignored.

A sequence of 10 failures to ignite to hot coil and 10 failures to ignite to electric spark, both over a range of dust concentration and dispersion conditions, are required for Group B classification.

Minimum Ignition Temperature Test
The minimum ignition temperature (MIT) is the lowest temperature of a hot surface that will cause a dust cloud to ignite and propagate flame. The test follows BS EN 50281-2-1:1999 (Methods of determining minimum ignition temperatures).

Sample preparation
The sample is prepared to be homogeneous and should generally be fine enough to pass a 71 μm sieve. However, if it is necessary to test a coarser dust, the sample will be sieved to 500 μm and the fact will be stated in the test report.

Test method
Approximately 0.1 g of dust is placed in a dust holder at the top of a temperature controlled furnace with an open bottom. The dust is dispersed by compressed air downwards past the hot surface of the furnace to see if ignition occurs and flames are produced below. If the dust does not ignite, the furnace

temperature is increased and the test repeated until ignition of the dust occurs or a maximum temperature of 800 ºC is reached.

Once ignition has been established, the mass of the dust sample and injection pressure are varied to find the most vigorous flame discharge. The temperature of the furnace is then reduced incrementally until flame propagation is no longer observed. At this temperature, the dust mass and injection pressure are varied to confirm that no ignition is found over ten consecutive tests.

Test criterion

The minimum ignition temperature (MIT) is the lowest temperature of the furnace at which flame is observed minus 20 ºC for furnace temperatures over 300 ºC or minus 10 ºC for furnace temperatures under 300 ºC.

For items of plant such as driers, the internal temperature is generally limited to two thirds of the minimum ignition temperature when measured in degrees Centigrade. For example, a dust with a minimum ignition temperature of 450 ºc would require a maximum operating temperature in the drying process of 300 ºC.

Example MIT test results	
Sample	Minimum ignition temperature
Grain dust	490 ºC
Coal dust	no ignition up to 850 ºC
Flour	400 ºC
Sugar	480 ºC
Aluminium dust	650 ºC
Sewage sludge	430 ºC

Layer Ignition Temperature Test

The layer ignition temperature (LIT) test determines the lowest temperature at which a layer of dust of specific thickness, usually 5mm, ignites on a heated surface. Layer ignition temperatures are generally much lower than dust cloud ignition temperatures (MIT). It is an important test for industrial premises where heat generating equipment is present - such as electric motors or light fittings - as it evaluates the maximum permissible surface temperature to prevent fires and smouldering material.

The test is carries out according to BS EN 50281-2-1:1999 (Methods of determining minimum ignition temperature).

Sample preparation

The sample is prepared so as to be homogeneous and representative of the material handled on plant. The dust sample will generally be tested "as received", although sieving to sub 200 µm may be carried out to remove large particles. If it is necessary to test a coarser dust, passing a test sieve with a nominal size of aperture up to 500 µm, the fact will be stated in the test report.

Test method

A layer of known thickness is created by filling a 100mm diameter ring placed on the hotplate apparatus, levelling with a straight edge and removing any excess. A thermocouple positioned on top of the layer monitors the dust temperature during the test and the temperature is continuously recorded using data logging equipment.

The ambient temperature is also measured locally to the apparatus and must be within the range 15 ºC to 35 ºC.

Test criterion

Tests are repeated with fresh layers of dust until a minimum ignition temperature has been found. This is the lowest temperature, rounded down to the nearest integral multiple of 10 ºC, at which ignition occurs in a layer of given thickness. The highest value at which ignition does not occur is also recorded. The latter

temperature must be no more than 10 °C lower than the minimum ignition temperature recorded and is confirmed by at least three tests.

Ignition is considered to have occurred if:

1. visible glowing or flaming is observed; or
2. a temperature of 450°C is measured; or
3. a temperature rise of 250 K above the temperature of the heated plate, is measured.

Any thickness of dust layer may be investigated, but 5 mm is generally regarded as representing the thickest layer of dust likely in a factory having fully enclosed plant and process operations allied to a good standard of housekeeping. Where there is the possibility that dust layers in excess of 5 mm may be formed, the maximum permissible surface temperature must be reduced.

By limiting the temperature of any surface to below the layer ignition temperature (LIT) - usually with a 75 °C safety margin - fires caused by hot surfaces may be avoided.

NOTE: Some materials exhibit more than one stage of self-heating, and it may sometimes be necessary to prolong the test in order to fully explore this possibility. With organic dusts, combustion will usually take the form of charring followed by the appearance of smouldering with glowing which will progress through the layer and leave a residue of ash. With dust layers composed of certain divided metals, ignition may be characterised by the relatively sudden appearance of highly incandescent smouldering combustion progressing rapidly through the layer.
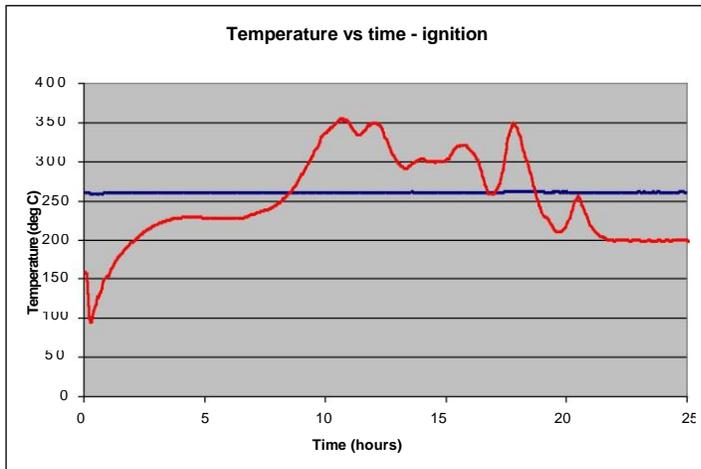

Typical 5 mm layer ignition temperature test trace for sewage sludge, showing ignition on a hot surface at 260°C

Lower Explosion Limit Test

The lower explosion limit (LEL) - also known as the minimum explosion concentration - is the lowest concentration of dust cloud that will allow combustion. The lower explosion limit decreases with decreasing particle size and decreasing moisture content. Therefore the particle size distribution and moisture content are determined for the sample as tested and stated in the test report.

The test following BS EN 14034-3:2006 (determination of the lower explosion limit LEL of dust clouds) is carried out in the 20 litre sphere apparatus.

20 litre sphere apparatus
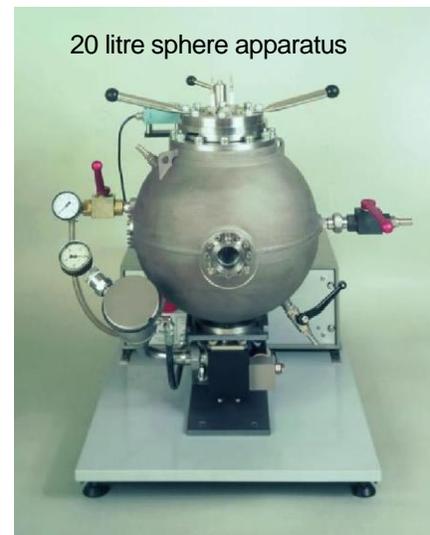
Test method
10 g of dust is placed in the dust container and the explosion chamber is evacuated to 0.4 bar. An automatic test sequence is initiated to pressurise the dust container to 20 bar and activate the two chemical igniters, each having an energy of 1 kJ, 60 msec after the dust has been dispersed.

A test series is undertaken with a systematic decrease of the dust concentration until no ignition of the dust / air mixture is observed. After each test, the explosion vessel is cleaned to remove any dust remaining from the previous test.

Test criterion
An ignition is deemed to have occurred if the maximum explosion pressure is at or above 0.5 bar.

The final test is repeated to ensure no ignition is found in three consecutive tests and confirm the lower explosion limit has been reached.

Knowledge of the lower explosion limit may be used as evidence of explosion prevention in areas where the concentration of dust can be reliably controlled. The test is also helpful in determining where hazardous area zones exist for dust (zone 20, zone 21 & zone 22) under ATEX or DSEAR legislation.

Minimum Ignition Energy Test

The minimum ignition energy (MIE) test measures the ease of ignition of a dust cloud by electrical and electrostatic discharges. The test is carried out according to BS EN 13821:2002 (Determination of minimum ignition energy of dust / air mixtures).

Ignition energy is influenced by a considerable number of factors, the most important being; median particle size, dust moisture content, dust cloud turbulence and dust concentration. Inductance in the spark generation circuit may also have an effect as protracted sparks (i.e. with inductance) are generally more incendive than purely capacitive discharges. Investigation of MIE for electrostatic discharges is carried out with no inductance in the spark generating circuit.

Sample preparation
Where the particle sizes of the material in the plant are not known, tests are carried out on dust samples of particle sizes e.g. less than 63 μm. Where the moisture content in the plant is not known, the is carefully dried e.g. at 50 °C under vacuum, or at 75 °C and atmospheric pressure until the sample weight has reached a constant value.

Test method
The MIKE3 apparatus consists of a borosilicate tube placed over a dispersion cup and fitted with two electrodes. The electrodes are connected to a circuit that produces an electrical spark of known energy.

A weighed sample of dust is placed in the dispersion cup, which is blown up through the tube with compressed air past the ignition source. If flame propagation is observed, the energy of the spark is reduced until no flame propagation is seen for 10 consecutive tests. At that spark energy, a range of dust sample weights and ignition time delays are tried to thoroughly investigate potential dust explosion conditions.

Test criterion
The test result is stated as an energy range, indicating that ignition took place at the higher value and no ignition took place at the lower value. The MIE is a measure of how sensitive an explosible dust is to electrical spark ignition. It gives guidance on whether ignition by electrostatic discharge from plant personnel or process conditions is likely to occur in practice.

Limiting Oxygen Concentration Test

The limiting oxygen concentration (LOC) is the highest concentration of oxygen that prevents a dust explosion taking place. The test is mandatory if inerting is to be used as a method of dust explosion prevention under ATEX or DSEAR.

The test is carried out according to BS EN 14034-4:2004 (Determination of the limiting oxygen concentration LOC of dust clouds).

Test method
The test is carried out in the 20 litre sphere apparatus. 5 g of dust is placed in the dust container and the explosion chamber is filled with a known concentration of oxygen in nitrogen. An automatic test sequence is initiated to pressurise the dust container to 20 bar and activate the ignition source 60 msec after the dust has been dispersed. Once ignition is achieved at a known oxygen concentration, the level of oxygen is reduced until a point is reached at which no ignition occurs. At an oxygen level 1 % above the minimum found, the quantity of dust injected is adjusted to find the most explosible concentration in a reduced oxygen atmosphere (which is generally at lower dust concentrations than at normal atmospheric conditions). Subsequent tests are carried out with this mass of dust, and the oxygen concentration is gradually reduced until no ignition is found in three consecutive tests.

Test criterion
An ignition is deemed to have occurred if the maximum explosion pressure is at or above 0.5 bar. Test work is normally undertaken with nitrogen, but it is important to test with the appropriate inert gas in use on plant. Substantially different results are obtained with different inert gases e.g. steam, nitrogen, carbon dioxide.

In practical applications, it is usual to include a safety margin when applying the test results to plant design. The safety margin should reflect the size of the plant and its control system for rectifying oxygen increases, plus the level of oxygen monitoring installed, the sensor accuracy and response time.

Example test results	
Sample	LOC (%) in nitrogen
Coal dust	14
Sewage sludge	11
Aluminium dust	5
Maize starch	9

Explosion Indices Test (K_{st} & P_{max})

K_{st} and P_{max} are explosive properties measured in the laboratory to quantify the severity of a dust explosion. The explosion indices test follows BS EN 14034-1:2004 (determination of the maximum explosion pressure P_{max} of dust clouds) and BS EN 14034-2:2006 (determination of the maximum rate of explosion pressure rise of dust clouds K_{st}).

Test method

The tests are carried out in a 20 litre sphere apparatus which reproduces a high state of turbulence to simulate worst case process plant conditions.

A weighed quantity of dust is placed into the dust container. The main explosion chamber is then evacuated to 0.4 bar absolute. An automatic test sequence is initiated to pressurise the dust container to 20 bar gauge, and then the fast acting valve on the dust container outlet is opened to allow material into the explosion chamber.

The rebound nozzle ensures an even distribution of dust within the explosion chamber and the control system activates two 5 KJ chemical igniters at the centre of the sphere 60 ms after the dust has been dispersed.

Explosion pressures are measured for a range of dust concentrations using piezo-electric pressure transducers. The tests are carried out over three series to ensure a thorough investigation of the explosion properties. From the tests, the arithmetic mean of the maximum values (both maximum pressure and maximum rate of pressure rise) is obtained.

The K_{st} value is calculated as the equivalent pressure in a 1 m^3 sphere from the cube law (K_{st} = cube root of volume x explosion pressure rise), as previously described.

ST class

The ST class is based on the K_{st} value as follows:

ST class	K_{st} (bar m/sec)
ST 0	0
ST 1	< 200
ST 2	Between 200 & 300
ST 3	>300

P_{max} & K_{st} data is used to design explosion protection systems, typically explosion venting & explosion suppression.

| Example test results			
Sample	K_{st} (bar m/sec)	P_{max} (bar)	ST class
Grain dust	89	9.3	1
Coal dust	85	6.4	1
Flour	63	9.7	1
Sugar	138	8.5	1
Wood dust	224	10.3	2
Aluminium dust	515	11.2	3
Sewage sludge	102	8.1	1

Self-heating Materials

Many materials exhibit self-heating where biological and/or chemical reactions (usually oxidation) take place at an imperceptibly slow rate at room temperature. However, the rate of reaction increases rapidly with temperature and given the right storage conditions may lead to spontaneous combustion. The prediction of situations where spontaneous fires may occur and the identification of simple precautions to prevent such fires are essential to manage the risk effectively.

A simple screening test is used to identify materials with self-heating or exothermic decomposition properties.

Test method

The test is carried out in a mechanically ventilated oven with an inner chamber of sufficient size to accommodate the test piece and allow free air circulation around it. A cubical wire mesh basket measuring 100 mm on each edge if filled with the material under test and a 0.5 mm stainless steel sheathed chromel/alumel thermocouple is inserted into the centre of the sample to monitor the central temperature. The filled cube is then suspended in the oven together with a second thermocouple to register oven temperature and both are connected to a data logger so that any tendency for self-heating can be observed and recorded.

For the screening test, the temperature of the oven is set to increase at a rate of 0.5 $^\circ$C per minute from room temperature up to a maximum of 400 $^\circ$C for approximately 24 hours. The central thermocouple will detect any exothermic reaction where the temperature increases independently of the oven temperature. The onset of exotherm may be measured by determining the temperature at which the central temperature exceeds the oven temperature (crossing point) and by this method comparisons can be made between different materials under test.

Ramped 100mm basket test
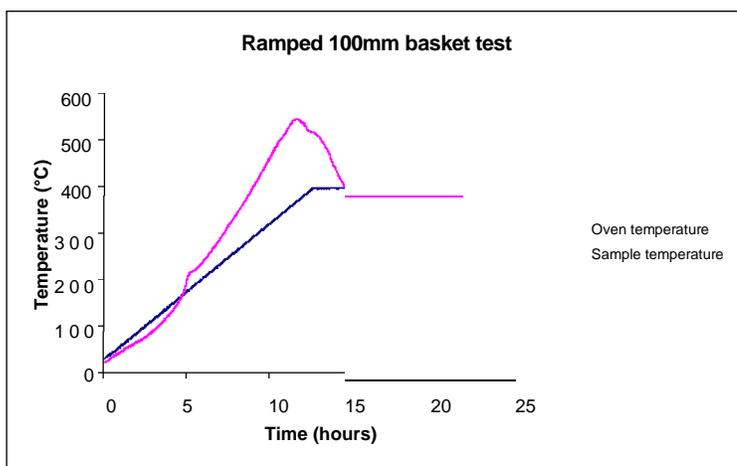
Test criterion

If the central temperature of the sample tracks the oven temperature for the whole of the test duration (24 hours), the material may be said to exhibit no propensity for self-heating over the time and temperature ranges investigated.

Generic versus Actual Test Data

Of course, many common materials have been tested before and there considerable body of data available from previous laboratory work. The problem facing plant engineers and system designers is whether or not such data is truly representative of the materials to be handled by the plant under consideration. Published test results usually have little by way of description or detailed specification, and the question remains "is this like my material?"

To take an example, the explosion indices of sugar (sucrose) are commonly quoted as P_{max} = 8.5 bar and K_{st} = 138 bar m/sec. Yet sugar has a much wider range of explosibility, depending upon the manufacturing source, particle size and moisture content, as illustrated in the table below. It cannot even be characterised as an ST1 material, although in most applications this is probably the case.

Material	Mean particle size (micron)	K_{st} (bar m/sec)	P_{max} (bar)	ST class
Sugar	290	11	3.9	ST 1
Sugar	29	59	8.2	ST 1
Sugar	30	138	8.5	ST 1
Sugar	<20	205	9.1	ST 2

With some materials, it is possible to carry out an assessment of explosion risk through the oxygen balance thus:

$$C_xH_yO_z + (x + y/4 - z/2)O_2 \rightarrow XCO_2 + (y/2)H_2O$$
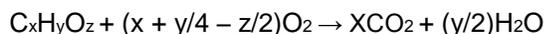
where x,y,z are the number of moles of carbon, hydrogen and oxygen respectively

The oxygen balance, which is a measure of the lack of oxygen in the molecule necessary for stoichiometric combustion, is derived from the following formula:

-16 x (2x +y/2-z) x 100/(molecular weight)

This approach is useful for chemical compounds, but is of little value for foodstuffs, for example. When dealing with a multitude of different materials, a combination of form, quantity and chemical group can be appropriate guidelines. Taking food ingredients as an example, a number of product categories could be considered; flours, sugars, starches, cocoas, milk powders (including whey), egg powders, glutens, enzymes, emulsifiers, powdered fats, gelling agents and miscellaneous. Examination of existing explosibility and ignition data then allow us to make a number of reasonable assumptions:

1. Flours will tend to have high MIE, whereas sugars will generally be much lower.
2. MIT of flour is likely to be lower than sugar.
3. High fat flours, such as soya, may have lower LIT temperatures.
4. Dusty materials, such as cocoa powder, are likely to be drawn into dust extraction filters. If these are protected by explosion venting, knowledge of K_{st} & P_{max} is required to validate the protection design.

In this way, a structured examination of the plant and materials handled can be most useful in shaping the test requirements for the project.

Data Required for DSEAR & ATEX Compliance

Explosion prevention and explosion protection must both be considered for DSEAR & ATEX compliance:

How could dust be ignited in the process?		
	Test	Action
Hot surface	LIT for dust layers MIT for dust clouds	Specify equipment surface temperature limits, including safety margin
Electrostatic spark	MIE	Influences the choice of plant materials (conductors/non-conductors), earth bonding and personnel antistatic precautions
Self-heating	Screening test	Identify materials with the potential for self-heating. Isothermal basket tests will be required to specify safe storage conditions

What is the best way to avoid an explosive atmosphere?

	Test	Action
Dust control	LEL	Plant should be operated with low level of dust present e.g. use dust-suppressed materials
Oxygen control	LOC	Plant should be operated under an inert gas e.g. nitrogen. An operational safety margin is required, based upon the inert gas control system and plant layout

How can the plant be made safe?

	Test	Action
Explosion venting	Explosion indices K_{st} and P_{max}	Verify vent design is adequate for the materials being handled
Explosion suppression	Explosion indices K_{st} and P_{max}	Verify suppression design is adequate for the materials being handled
Explosion containment	Explosion indices K_{st} and P_{max}	Verify explosion containment and explosion isolation design is adequate for the materials being handled

The following data should be regarded as essential, safety critical information:

LOC – where inerting is the basis of safety

Explosion indices (P_{max} & K_{st}) where explosion protection is the basis of safety – i.e. explosion venting, explosion suppression or explosion containment

MIT – for all plant where a dust cloud may come into contact with a high temperature surface, such as a dryer

LIT – for all plant where dust may accumulate on any surface that may get hot through electrical or mechanical energy such as an electrical motor or gearbox

MIE – for all plant, particularly where electrostatic ignition is possible

Screening test for any material that is suspected of being capable of self-heating

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