

ISSA Prevention Series No. 2018 (E)



International Section
of the ISSA
for Machine Safety

Determination of the Combustion and Explosion Characteristics of Dusts

suva

**Schweizerische
Unfallversicherungsanstalt
Postfach 4358
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1998
ISBN 92-843-1092-X
ISSN 1015-8022



THE INTERNATIONAL SOCIAL SECURITY ORGANIZATION (ISSA)

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To improve occupational safety and health protection in plants, the



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was formed in 1975. It deals with questions concerning the safety of machines, installations and systems. The Chair and the Secretariat are located at the headquarters of the Berufsgenossenschaft Nahrungsmittel und Gaststätten (professional association of the food and catering industry), D-68165 Mannheim.

To emphasize occupational safety in plants in the chemical industry, including the plastics, explosives, oil and rubber industries, the



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was formed in 1970. The Chair and the Secretariat are located at the headquarters of the Berufsgenossenschaft der chemischen Industrie (professional association of the chemical industry), D-69115 Heidelberg.

Determination of the Combustion and Explosion Characteristics of Dusts

Publisher

International Section of the ISSA for Machine Safety
Dynamostrasse 7-11
D-68165 Mannheim

Preface

The "Machine Safety" section in the "Permanent Committee of the International Social Security Association (ISSA) for the prevention of occupational risks" has established several working parties to handle the question of safety of machines, installations and systems. Working party members are international experts from universities and research institutions as well as industrial enterprises and the prevention departments of accident insurers. This ensures that practice-oriented proposals for solutions are developed for not only straightforward but also complex questions relevant to safety. A specific task of the working party "Dust explosions" of the section involves explosion protection. This booklet - which has been compiled in close co-operation with the "Chemistry" section of the ISSA - describes methods for determination of the combustion and explosion characteristics of dusts. Combustion and explosion characteristics are an important basis for the development of protection concepts in actual practice. The "Machine Safety" section thus contributes to the maintenance and further development of a high and - through the active cooperation of the members of its working parties in commissions of the EU - comparable technological development among industrial countries. Thanks to its appreciation of the comprehensive, worldwide importance of effective prevention, the section can also offer support and guidance in developing countries.

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Introduction

This booklet is part of a comprehensive account of the dust explosion protection of machines and apparatus published by the Machine Safety section of the ISSA. The basis of this series of publications is the ISSA booklet "Basic principles for the prevention of dust explosions of machines and apparatus [1]", which describes the important relationships and includes definitions.

Assessment of the fire and explosion hazard in the handling of combustible dusts as well as the planning and dimensioning of protective measures requires knowledge of the combustion and explosion characteristics of dusts [2, 3]. For correct use of the characteristics, it is important to know how they are determined. This publication thus briefly describes the test methods and test equipment currently in use. On no account should it be regarded as an operating manual or a set of test instructions. Further details can be found in the literature if required.

Unless stated otherwise, the tests are always carried out under atmospheric conditions.

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1. Sample preparation

Combustion and explosion characteristics of dusts depend on the nature of the dust sample used. Several important parameters which influence the combustion and explosion behavior are the particle size, particle shape, moisture content, purity and, if applicable, the content of flammable solvents. Representative samples are thus indispensable for the tests. Safety documentation should include as many combustion and explosion characteristics as possible; moreover, at the minimum the particle size distribution and the mean particle size of the test sample must be known.

If the dust sample is subjected to preparatory steps before the test, e.g. by screening, drying or grinding, this must be communicated with the test results.

2. Safety characteristics of dust layers

Procedures to determine the following characteristics are treated:

- **Flammability**
- **Burning behavior**
- **Minimum ignition temperature of a dust layer (smolder temperature)**
- **Self-ignition**
- **Exothermic decomposition**
- **Spontaneous decomposition (“deflagration”)**
- **Impact sensitivity**

2.1 Flammability

The flammability (ignitability) of a dust deposit or layer specifies the ease with which the dust can be ignited by one or more ignition sources. If the dust deposit can be ignited in the tests, the dust is considered combustible.

Test apparatus

The test uses a ceramic plate and different ignition sources such as a gas flame, burning cigarette, match or flint sparks (Figure 1).

Test procedure

A product train of width 2 cm and length 4 cm is deposited on a ceramic plate. Different ignition sources are used to establish whether the dust deposit can be ignited.

Evaluation

If the dust deposit can be ignited, the dust is considered to be combustible. Details of the ignition source used must be given.

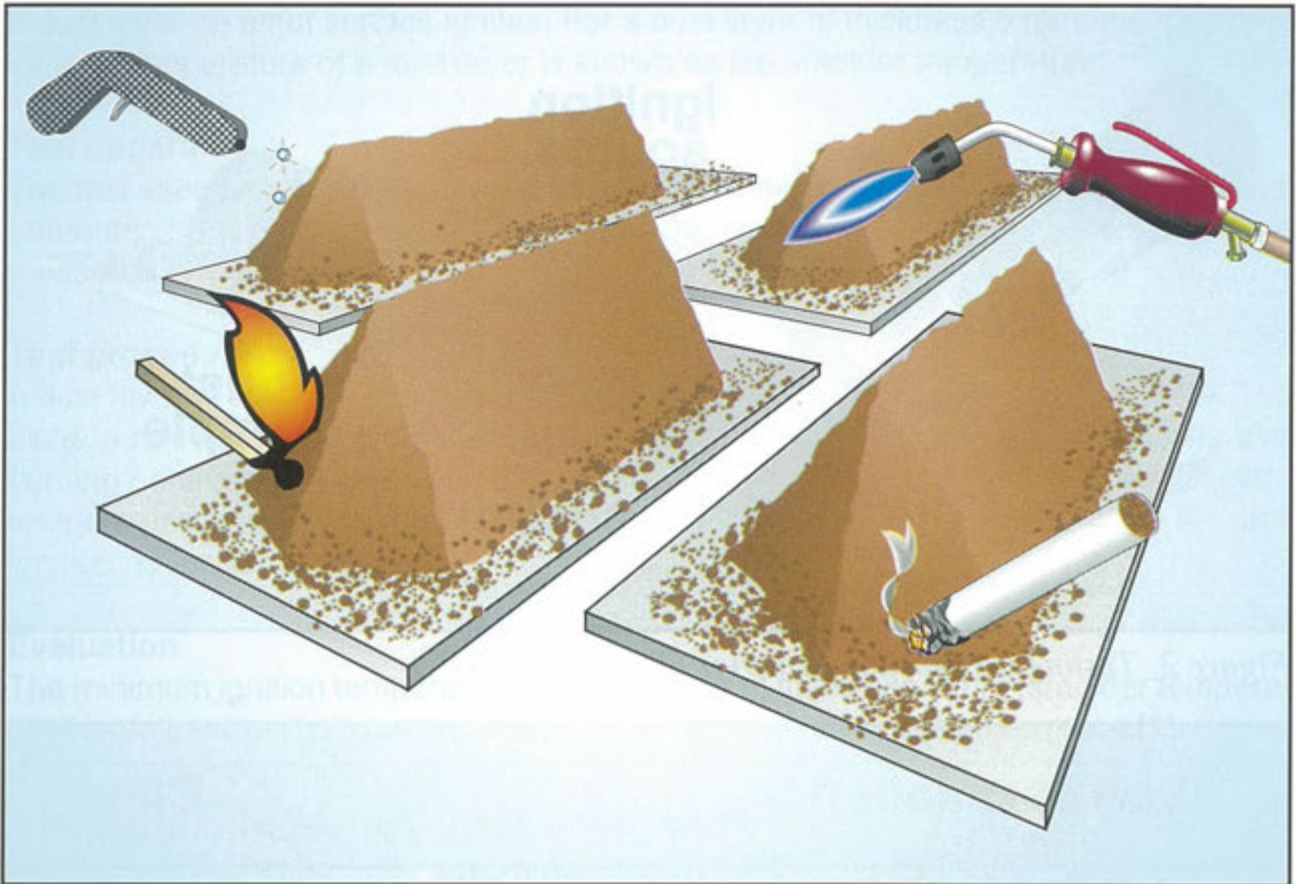


Figure 1: Testing the flammability of dust deposits with different ignition sources

2.2 Burning behavior

The burning behavior is used to describe the nature of the fire in a dust deposit.

Test apparatus

The test uses a ceramic plate and usually a glowing platinum wire. In isolated cases, a gas flame may also be used as the ignition source (Figure 2).

Test procedure

A product train of width 2 cm and length 4 cm is deposited on a ceramic plate. The glowing platinum wire is inserted in the product train in an attempt to ignite the dust. After successful ignition, the severity of the subsequent burning is observed. The test can be performed at room temperature or at elevated temperature (usually 100° C).

Evaluation

The burning behavior of the dust sample is assessed and the dust rated by a class number according to the classification in Table 1.

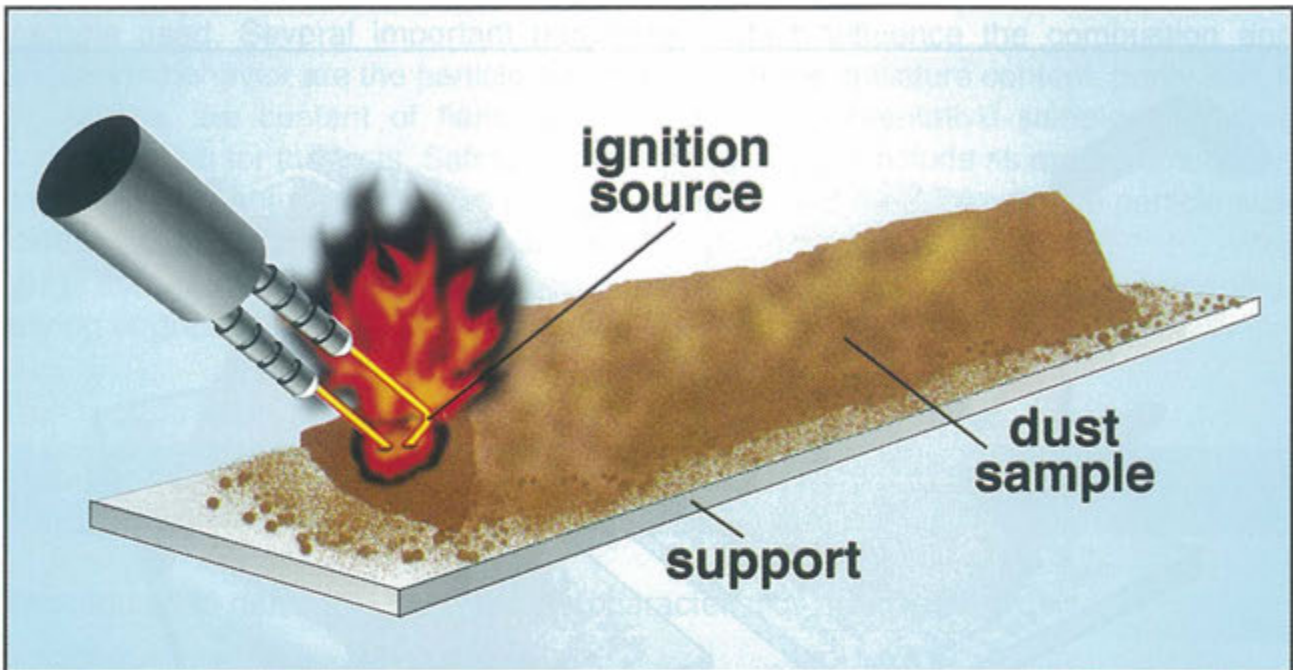


Figure 2: Testing the burning behavior of dust deposits

Type of reaction	Class
No burning, no ignition	1
Brief burning, rapid extinction	2
Localized combustion or smoldering (no or only very minor propagation)	3
Spread of a smoldering fire or slow, flameless decomposition	4
Spread of an open fire (burning with flame development)	5
Very rapid burning through with flame development or rapid, flameless decomposition	6

Table 1: Burning behavior of dust layers

2.3 Minimum ignition temperature of a dust layer (smolder temperature)

The minimum ignition temperature [4] of a dust layer is the lowest temperature at which a dust layer on a hot surface ignites. For a dust layer of thickness 5 mm, the minimum ignition temperature of a dust layer is known as the smolder temperature.

Test apparatus

The test uses an electrically heated, circular plate made of aluminum or stainless steel (diameter 200 mm, thickness 20 mm). The temperature of the plate and the sample are measured (Figure 3).

Test procedure

A dust layer of diameter 100 mm and thickness 5 mm is deposited on the hot plate. The plate is maintained at a constant temperature for 2 hours and the flammability and burning behavior (e.g. open flame or smoldering fire) is described. Initially, a high plate temperature is used which is successively lowered - generally in steps of 10 K - until ignition no longer takes place.

Evaluation

The minimum ignition temperature of a dust layer of thickness 5 mm (smolder temperature) is defined as the lowest temperature at which ignition just takes place [5].

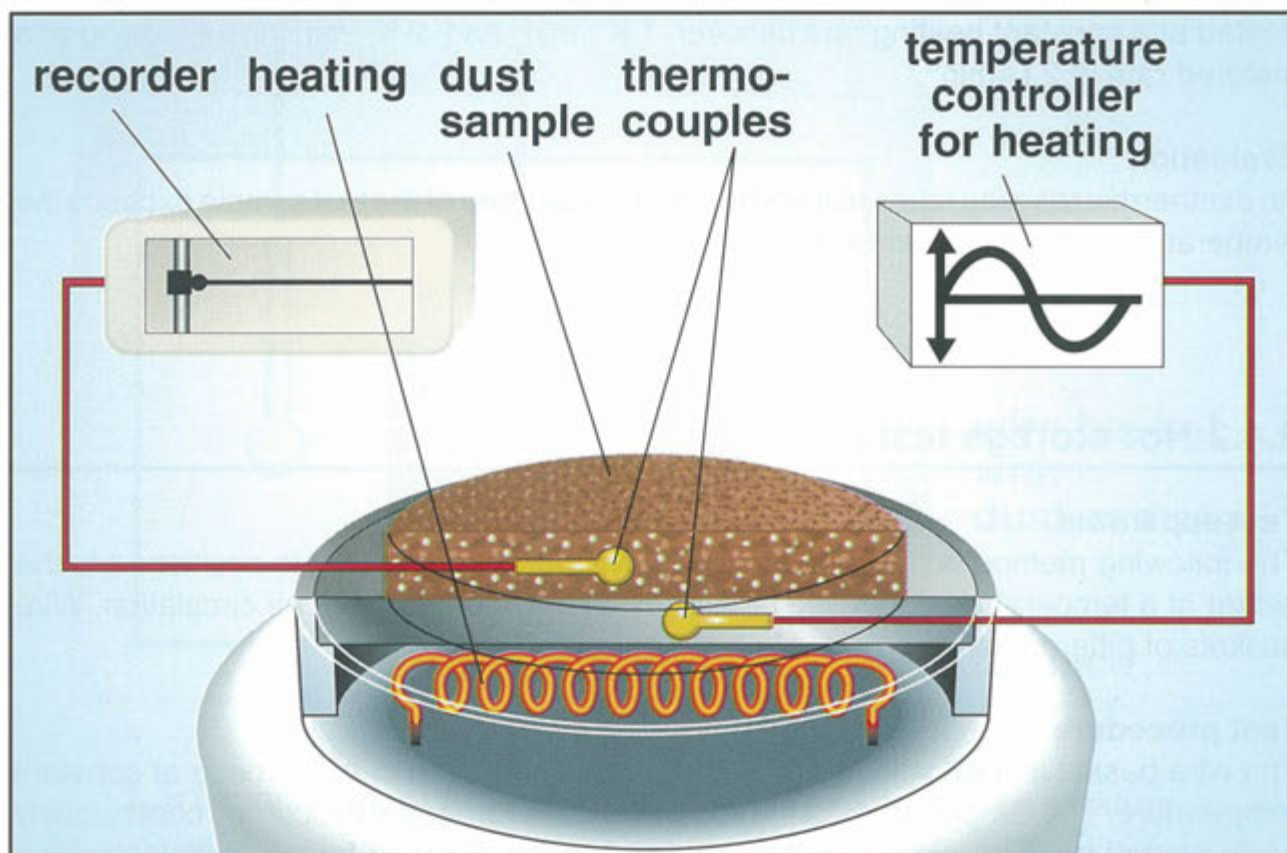


Figure 3: Test apparatus for determining the minimum ignition temperature of a dust layer

2.4 Self-ignition

The self-ignition behavior shows how well a bulk dust sample can be oxidized with atmospheric oxygen with heat development up to ignition. Self-heating and, if applicable, self-ignition take place only when the heat development in the dust sample is greater than the heat dissipation. For experimental determination of the self-ignition behavior, the following procedures can be used:

- Quick test following Grewer
- Hot storage tests

2.4.1 Quick test following Grewer (screening test)

Test apparatus

An electrically heated oven or furnace is used for the test. The upper part of the furnace contains two wire baskets each with a volume of 8 cm³ to accommodate the test sample and the reference substance. Air is circulated through the furnace from below.

Test procedure

The test sample and the reference substance (graphite) are placed in the furnace and heated at a constant heating rate between 1 K · min⁻¹ and 2 K · min⁻¹ in air flowing at a metered rate of 2 l · min⁻¹.

Evaluation

An exothermic reaction takes place when the temperature of the test sample exceeds the temperature of the reference substance in the test.

2.4.2 Hot storage tests

Test apparatus

The following method is described in [6]. A cylindrical wire basket is positioned in the center of a temperature-controlled laboratory oven or furnace with air circulation. Wire baskets of different shapes and volumes are used (Figure 4).

Test procedure

The wire basket is filled with the dust under test and stored in the furnace at constant temperature. The temperatures of the sample and the furnace are recorded continuously while heated fresh air circulates through the furnace. Starting from a sufficiently high value (e.g. result of the test in 2.4.1), the furnace temperature is lowered, usually in steps of 10 K (with fresh test substance in each case), until ignition is no longer observed.

Evaluation

Self-heating occurs when the sample temperature exceeds the furnace temperature. If the temperature in the bulk dust sample exceeds 400°C , this is assessed as self-ignition. The lowest furnace temperature at which self-ignition occurs in the test is the volume-specific self-ignition temperature. Specification of a self-ignition temperature is meaningful only when accompanied by details of the sample geometry, the sample volume and the storage time. If the experiments are performed with different volumes, extrapolation to larger volumes is also possible.

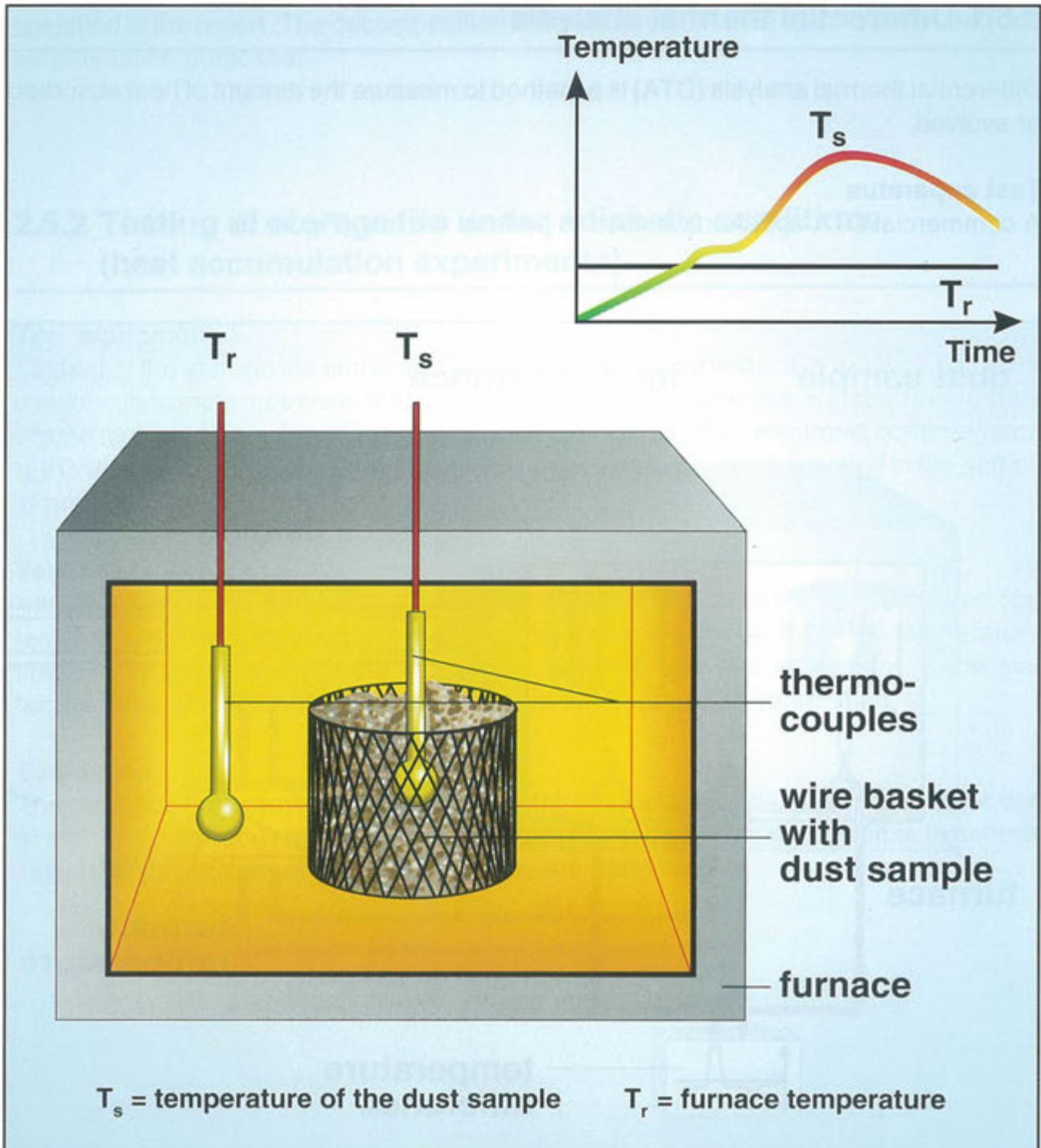


Figure 4: Test apparatus for determining the self-ignition temperature of bulk dust samples

2.5 Exothermic decomposition

An exothermic decomposition is a reaction which occurs in the absence of air which can lead to a temperature and possibly also a pressure increase. The following methods can be used to test the exothermic decomposition of a dust.

2.5.1 Differential thermal analysis

Differential thermal analysis (DTA) is a method to measure the amount of heat absorbed or evolved.

Test apparatus

A commercial DTA apparatus is used to perform the test (Figure 5).

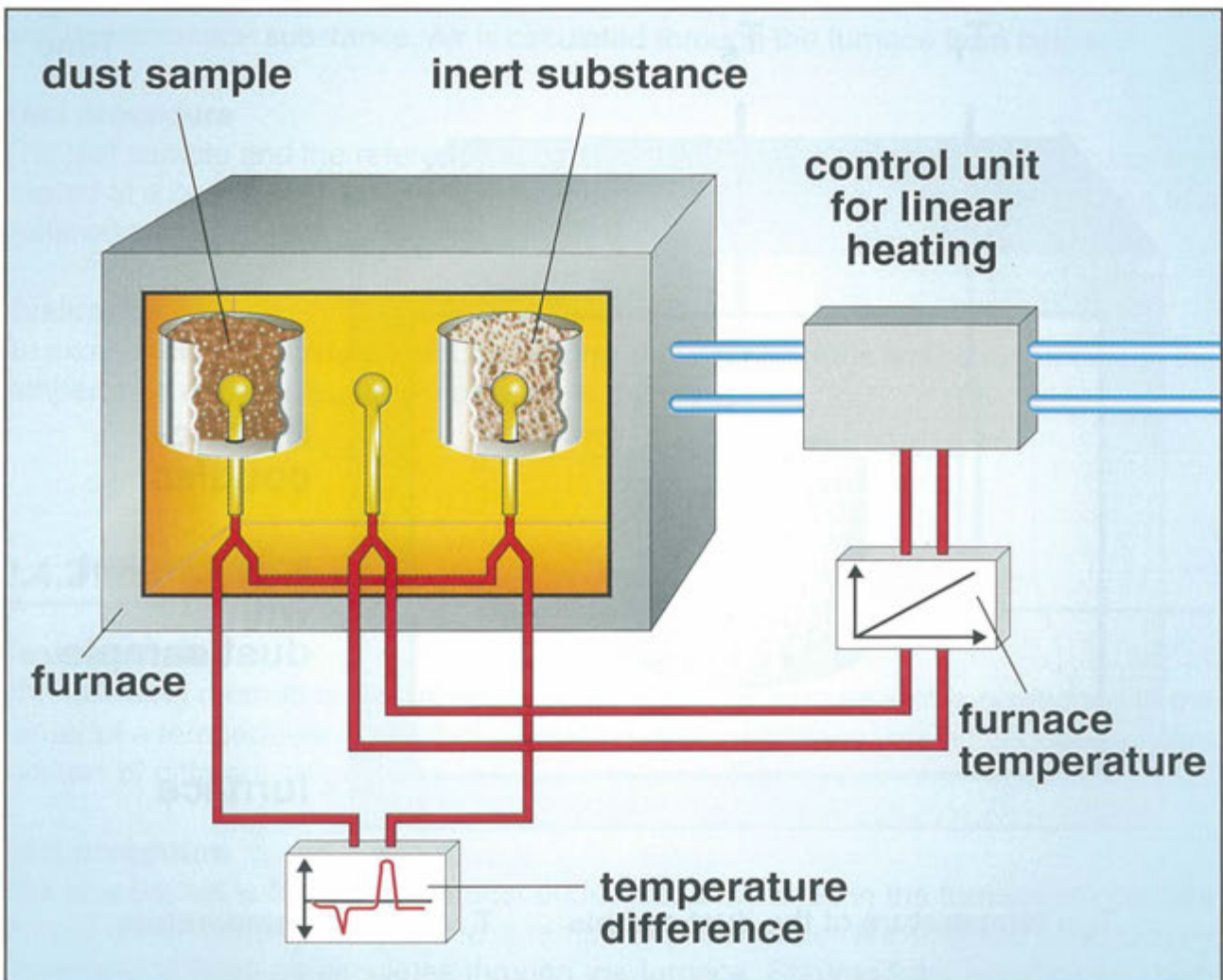


Figure 5: Test apparatus for determining the amount of heat absorbed or evolved - differential thermal analysis (DTA apparatus)

Test procedure

Two sample crucibles are inserted in the furnace. One crucible contains the test sample, the other an inert reference substance. Both substances are heated under the same conditions and their temperatures continuously recorded. The heating rate ranges from $1 \text{ K} \cdot \text{min}^{-1}$ to $10 \text{ K} \cdot \text{min}^{-1}$.

Evaluation

The decomposition temperature is defined as the temperature at which the temperature of the sample exceeds that of the inert reference material. The heating rate must be specified in the report. The decomposition temperature from a DTA analysis is used only for estimation purposes.

2.5.2 Testing of storage life under adiabatic conditions (heat accumulation experiments)

Test equipment

Testing of the storage life under adiabatic conditions is carried out in a 0.2 - 1.5 l Dewar vessel with sample quantities of at least 100 cm^3 . The Dewar vessel is placed in a furnace whose temperature is corrected continuously with the aid of an electronic control system to the temperature measured in the sample. A heating coil can be inserted in the sample to preheat it (Figure 6).

Test procedure

After the sample has been added to the Dewar vessel, it is preheated to the desired start temperature. The temperature is measured as a function of time. From the temperature-time profile thus obtained, the induction time (time between attainment of the start temperature and attainment of the maximum temperature) is determined.

Evaluation

The induction time provides information on the highest storage temperature of the dust at which no hazardous reactions are expected within a specified storage time. In general, several tests at different start temperatures are performed.

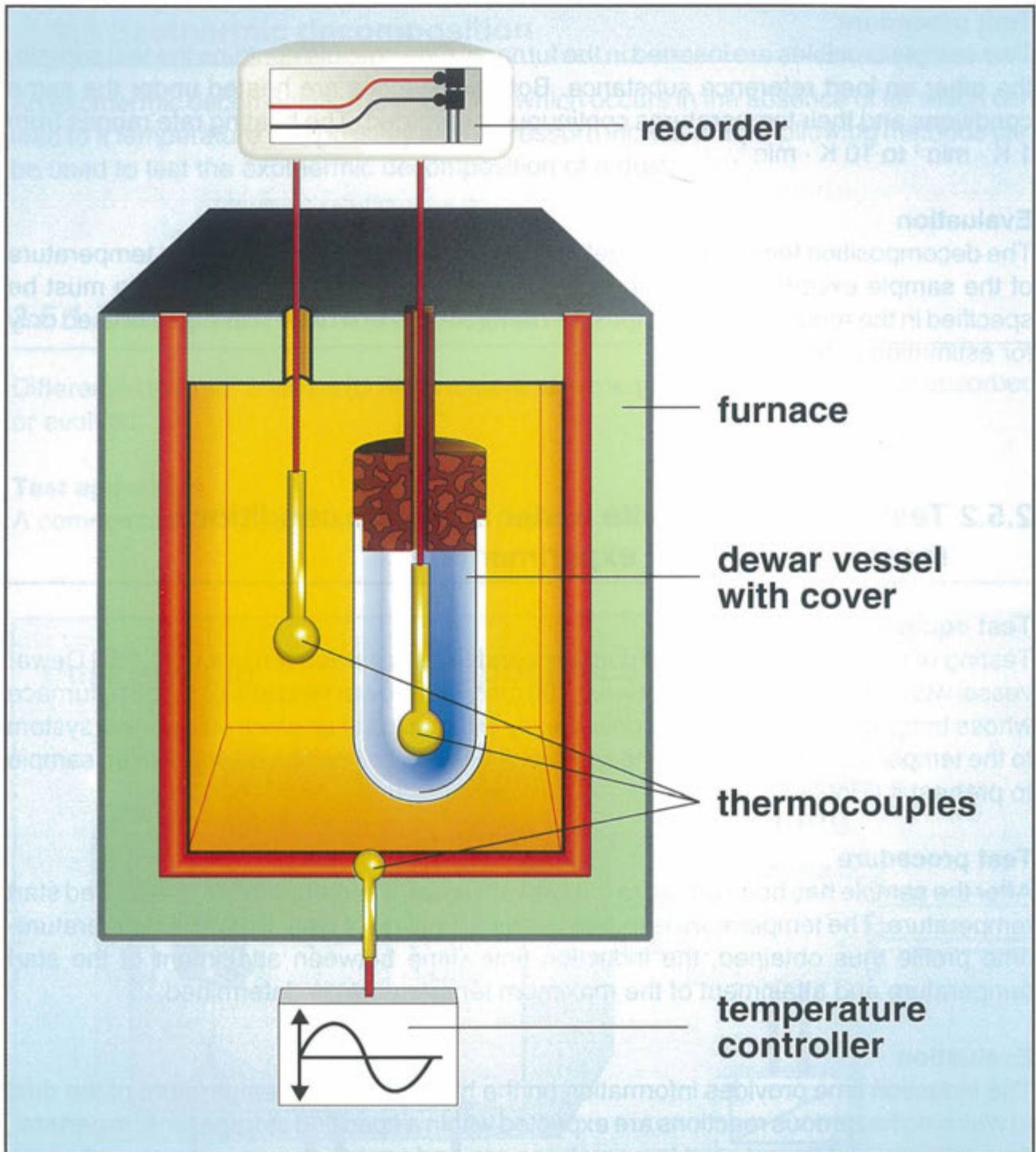


Figure 6: Test apparatus for determining the storage life under adiabatic conditions

2.5.3 Testing for spontaneous decomposition (“deflagration”)

As the expression “deflagration” is interpreted in different ways [7], within the scope of this booklet the term “spontaneous decomposition” will be used. This is understood to mean a local decomposition reaction which is initiated by an external ignition source and which, in contrast to combustion, can also propagate automatically in the absence of atmospheric oxygen.

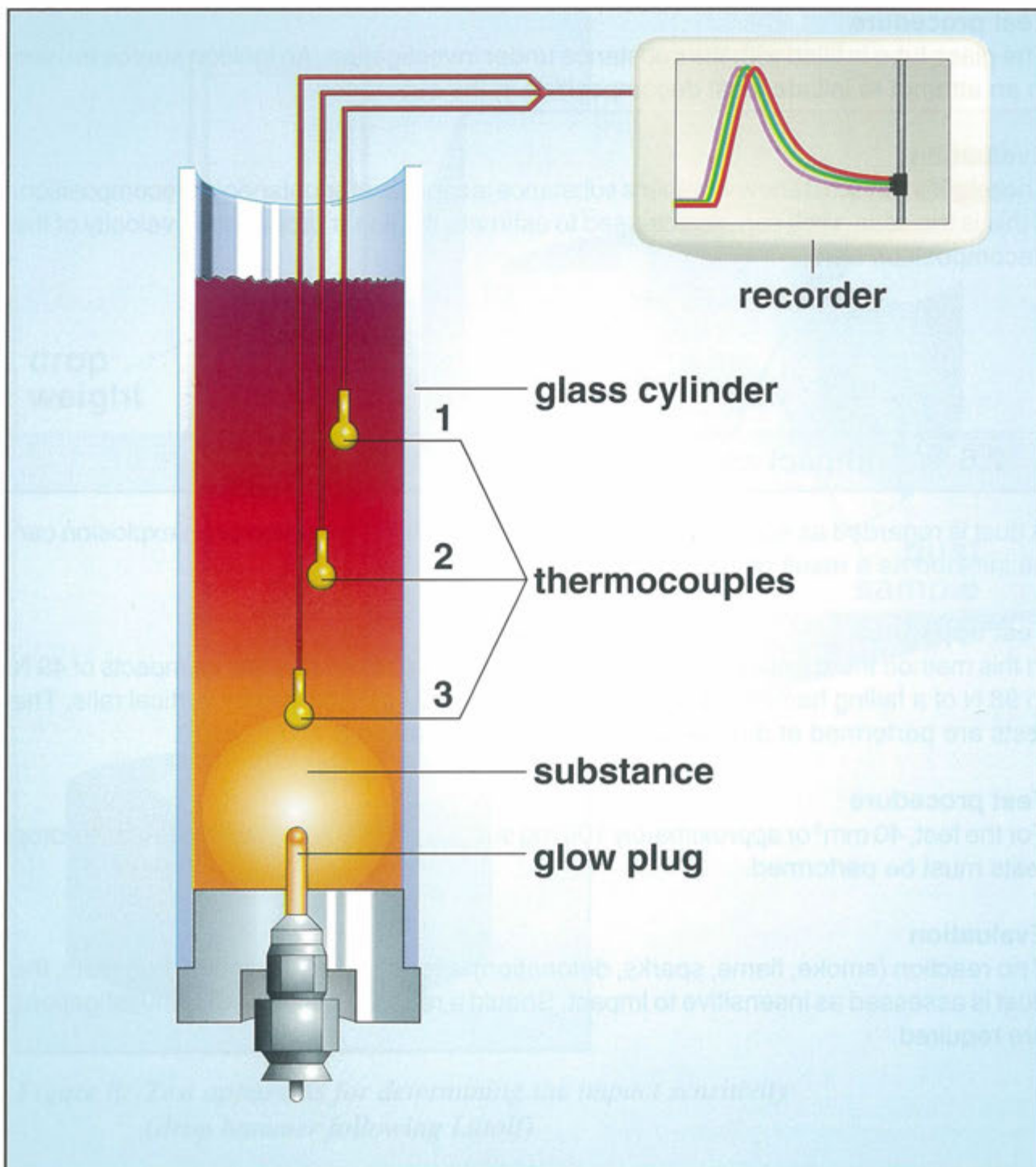


Figure 7: Test apparatus for determining the spontaneous decomposition capability

Test apparatus

A simple test apparatus to investigate the capability of a substance to undergo spontaneous decomposition comprises a vertical glass tube closed at the bottom. The tube is equipped with thermocouples arranged along its length at regular intervals as axially as possible. Various ignition sources such as a glow plug or heating coil can be used to initiate the decomposition reaction (Figure 7).

Test procedure

The glass tube is filled with the substance under investigation. An ignition source is used in an attempt to initiate local decomposition in the substance.

Evaluation

The results of this test show whether a substance is capable of spontaneous decomposition. If this is the case, they can also be used to estimate the linear propagation velocity of the decomposition front.

2.6 Impact sensitivity

A dust is regarded as sensitive to impact if a decomposition reaction or an explosion can be initiated as a result of impact stress.

Test apparatus

In this method the substance is placed on a steel anvil and is subjected to impacts of 49 N to 98 N of a falling hammer. The drop hammer (5 - 10 kg) is guided by vertical rails. The tests are performed at drop heights between 0.4 m and 0.8 m (Figure 8).

Test procedure

For the test, 40 mm³ or approximately 100 mg substance should be used. At least six drop tests must be performed.

Evaluation

If no reaction (smoke, flame, sparks, detonation) is found in any of the six drop tests, the dust is assessed as insensitive to impact. Should a reaction occur, further investigations are required.

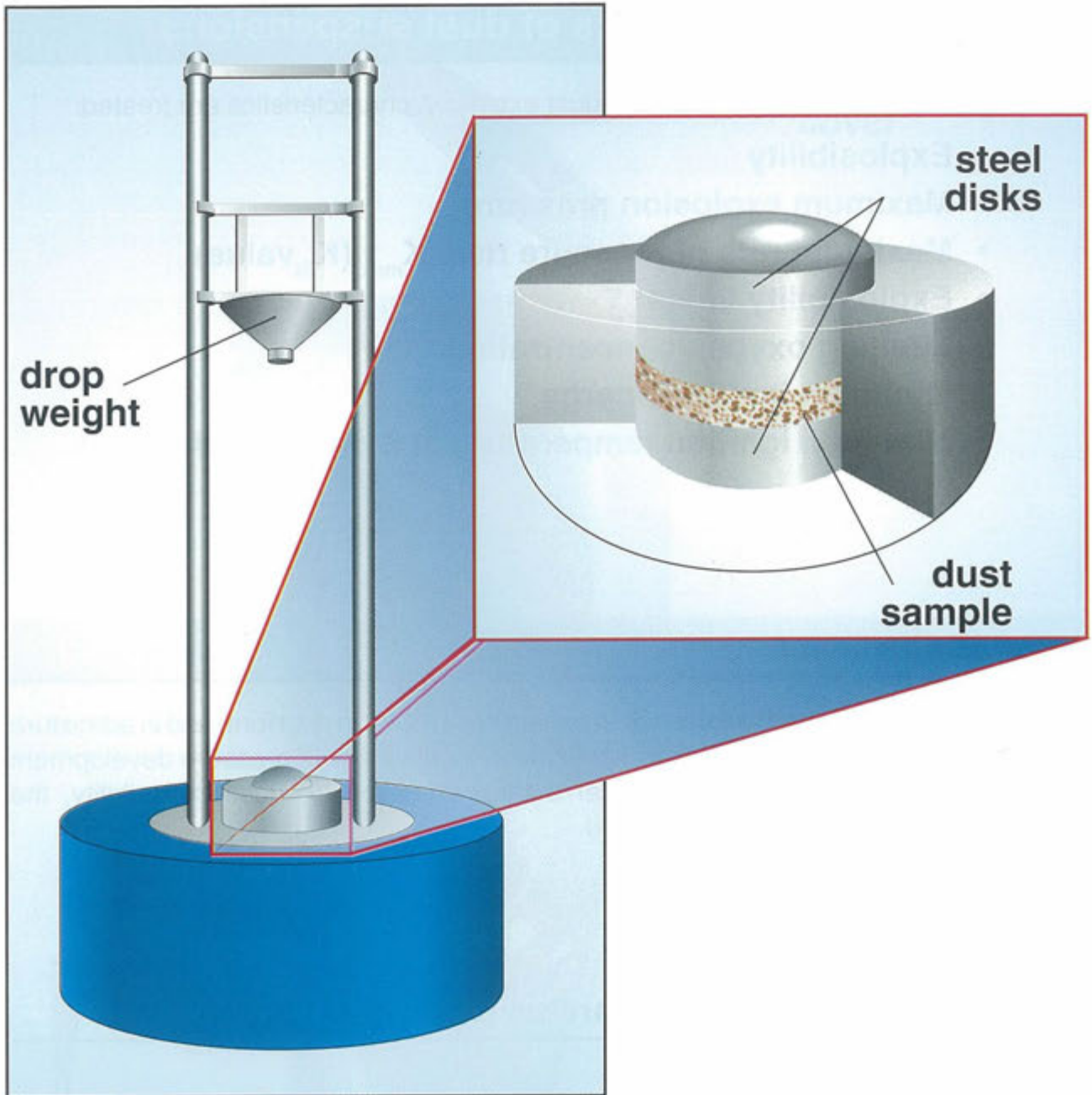


Figure 8: Test apparatus for determining the impact sensitivity (drop hammer following Lütolf)

3. Safety characteristics of dust suspensions

Procedures to determine the following dust explosion characteristics are treated:

- **Explosibility**
- **Maximum explosion pressure**
- **Maximum rate of pressure rise, K_{\max} (K_{St} value)**
- **Explosibility limits**
- **Limiting oxygen concentration**
- **Minimum ignition energy**
- **Minimum ignition temperature of a dust cloud**

3.1 Explosibility

A substance is considered to exhibit dust explosibility when, in dust form and in admixture with air, it reacts to produce progressive flame propagation with pressure development following ignition. For the experimental determination of the dust explosibility, the apparatus described below can be used.

3.1.1 Modified Hartmann apparatus (open glass tube)

Test apparatus

The modified Hartmann apparatus comprises a vertical glass tube of volume 1.2 l. The tube is closed at the bottom and has a pressure relief opening at the top. Either a high voltage continuous spark or a heating coil (incandescent coil) is used as the ignition source (Figure 9).

Test procedure

The dust sample is added to the Hartmann apparatus. After activation of the ignition source, the dust is dispersed by a metered blast of compressed air from an external pressure vessel. The tests are performed over a wide range of dust concentrations.

Evaluation

If flame propagation is observed, it may be assumed that the substance is explosible in dust form. If no ignition occurs, the dust must be tested in a closed vessel as described in section 3.1.2 before a final evaluation can be made.

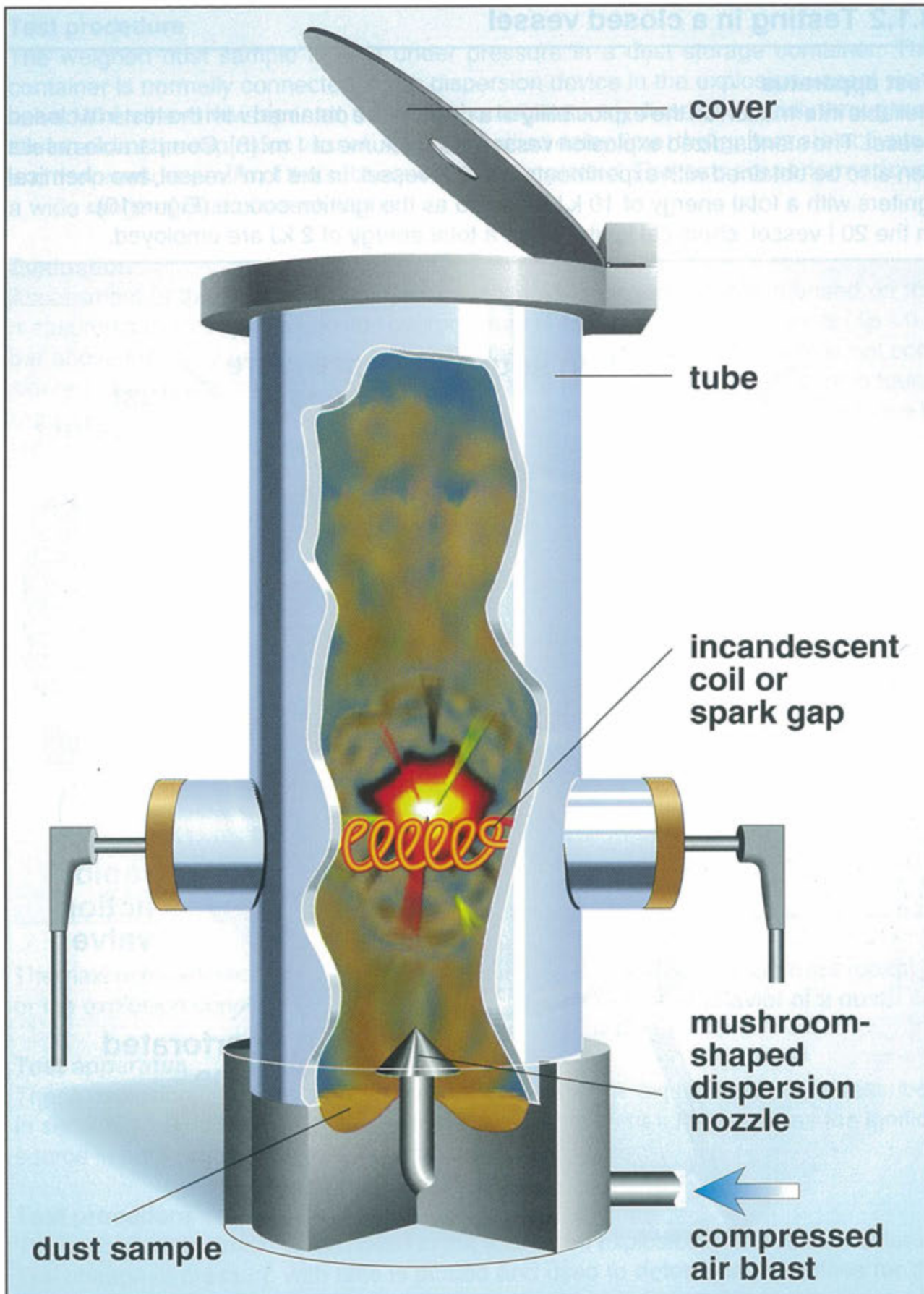


Figure 9: Test apparatus for determining the dust explosibility (modified Hartmann apparatus)

3.1.2 Testing in a closed vessel

Test apparatus

Reliable information on the explosibility of a dust can be obtained with the test in a closed vessel. The standardized explosion vessel has a volume of 1 m^3 [8]. Comparable results can also be obtained with experiments in a 20 l vessel. In the 1 m^3 vessel, two chemical igniters with a total energy of 10 kJ are used as the ignition source (Figure 10). In the 20 l vessel, chemical igniters with a total energy of 2 kJ are employed.

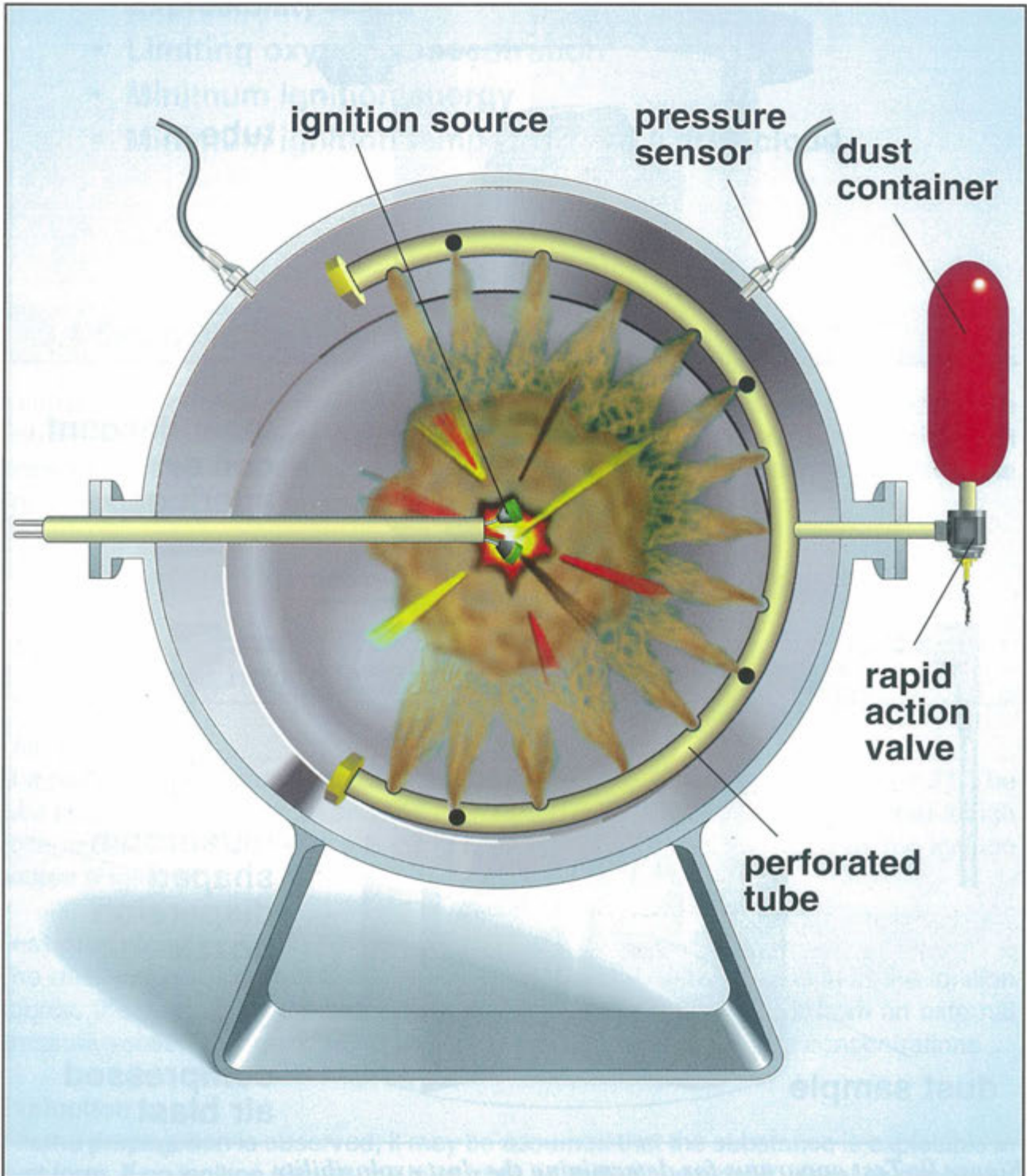


Figure 10: Test apparatus for determining the dust explosibility (1 m^3 vessel)

Test procedure

The weighed dust sample is kept under pressure in a dust storage container. The container is normally connected to the dispersion device in the explosion vessel via a valve. When the valve is opened, the dust is homogeneously dispersed rapidly throughout the interior of the explosion vessel. After a specified delay time, the igniters are activated and the pressure profile of the subsequent reaction plotted. The tests are performed over a wide range of dust concentrations.

Evaluation

Assessment of the explosibility of dust/air mixtures in closed vessels is based on the measured pressure. If no explosion overpressure is detected in any of the tests ($\Delta p \leq 0.3$ bar above the initial pressure specific to the test procedure), the substance is not considered to be dust explosible. On the other hand, if an explosion overpressure is found ($\Delta p > 0.3$ bar above the initial pressure specific to the test procedure), the substance is regarded as explosible when in dust form.

3.2 Maximum explosion overpressure, maximum rate of pressure rise, K_{\max} (K_{St} value)

The maximum explosion overpressure p_{\max} and the maximum rate of pressure rise $(dp/dt)_{\max}$ or the explosion constant K_{\max} (K_{St} value) describe the reaction behavior of a dust.

Test apparatus

These explosion characteristics can be determined in the explosion vessels described in section 3.1.2. Chemical igniters with a total energy of 10 kJ are used as the ignition source in both cases.

Test procedure

The procedure is similar to that used in the test of the explosibility of dust/air mixtures. The change in pressure with time is plotted and used to determine the values for the explosion overpressure p_m as well as the rate of pressure rise $(dp/dt)_m$ (see Figure 11). The tests are performed over a wide range of dust concentrations until the maximum values for the explosion overpressure and the rate of pressure rise are found (see Figure 12).

Evaluation

The determination of the maximum explosion overpressure p_{\max} and the maximum rate of pressure rise $(dp/dt)_{\max}$ is shown in Figure 12.

The maximum rate of pressure rise $(dp/dt)_{\max}$ depends on the volume of the explosion vessel and is converted with the aid of the so-called *cube-root law* into the volume-independent explosion constant K_{\max} :

$$(dp/dt)_{\max} \cdot V^{1/3} = \text{constant} = K_{\max}$$

The maximum rate of pressure rise is specified in $\text{bar} \cdot \text{s}^{-1}$, the volume in m^3 and the explosion constant in $\text{bar} \cdot \text{m} \cdot \text{s}^{-1}$. An explosible dust is classified into one of three dust explosion classes on the basis of its K_{\max} value:

K_{\max} [$\text{bar} \cdot \text{m} \cdot \text{s}^{-1}$]	Dust explosion class
> 0 to 200	St 1
> 200 to 300	St 2
> 300	St 3

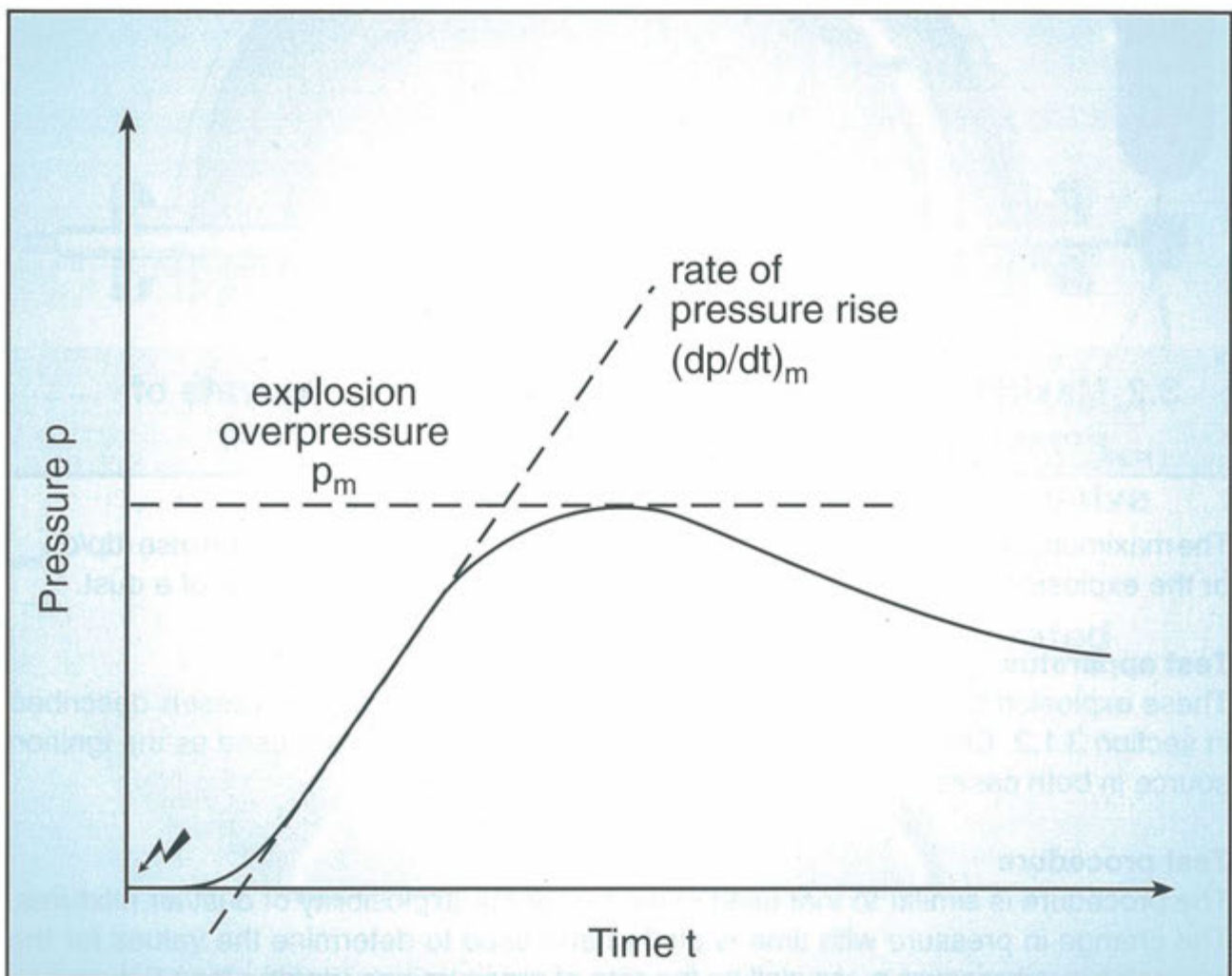


Figure 11: Pressure profile of a dust explosion in a closed vessel at any particular dust concentration in the explosible range

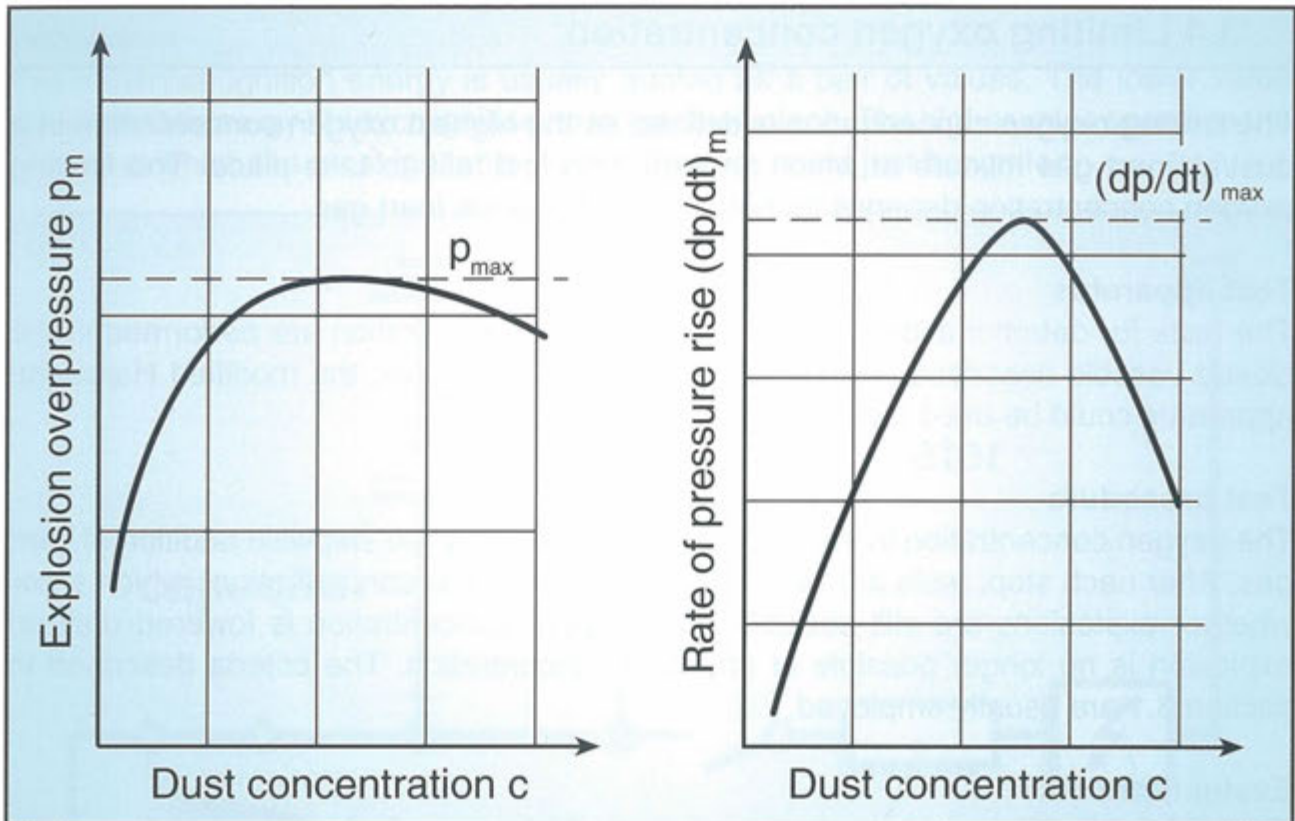


Figure 12: Explosion overpressure and rate of pressure rise as a function of the dust concentration

3.3 Explosibility limits

The explosibility limits describe the concentration range of a dust in admixture with air in which explosions are possible. Usually, only the lower explosibility limit is determined.

Test apparatus

For determination of the lower explosibility limit, the procedures used are the same as those for determination of the explosibility in closed vessels (see section 3.1.2).

Test procedure

An explosible dust concentration is first determined which is then lowered in subsequent tests until the dust suspension can no longer be ignited. The test is normally performed as described in section 3.1.2.

Evaluation

The highest dust concentration at which the dust/air mixture could no longer be ignited is specified as the lower explosibility limit. Evaluation is usually made as described in section 3.1.2.

3.4 Limiting oxygen concentration

The limiting oxygen concentration is defined as the highest oxygen concentration in a dust/air/inert gas mixture at which an explosion just fails to take place. The limiting oxygen concentration depends on both the dust and the inert gas.

Test apparatus

The tests for determination of the limiting oxygen concentration are performed in the closed vessels described in section 3.1.2. As an alternative, the modified Hartmann apparatus could be used (see section 3.1.1).

Test procedure

The oxygen concentration in the test vessel is lowered by the stepwise addition of inert gas. After each step, tests are performed at different dust concentrations which show whether explosions are still possible. The oxygen concentration is lowered until an explosion is no longer possible at any dust concentration. The criteria described in section 3.1 are usually employed.

Evaluation

The highest limiting oxygen concentration at which an explosion could no longer be observed even at the optimum dust concentration is designated the limiting oxygen concentration.

3.5 Minimum ignition energy

The minimum ignition energy of a dust/air mixture is defined as the lowest capacitively stored electrical energy which just ignites the most ignitable dust/air mixture following discharge across a spark gap.

Test apparatus

The minimum ignition energy can be determined in the apparatus described in section 3.1 (see also Figure 13). To generate an electrical spark of known energy, a capacitor of defined capacitance is charged up to a specified high voltage. The spark is discharged via an electrode arrangement in the dust/air mixture and its energy can be changed by variation of the capacitance and/or the charging voltage [9]. Usually, an inductance is included in the discharge circuit for protraction of the spark discharge.

Test procedure

The energy of the spark discharge is lowered in steps. Tests are performed after every step in the explosible range at different dust concentrations. The energy of the spark discharge is lowered until no ignition of the dust/air mixture occurs at any dust concentration.

Evaluation

The minimum ignition energy is usually quoted as a pair of values. The lower value specifies the energy at which ignition no longer took place. The higher value specifies the energy at which the most ignitable dust/air mixture could just be ignited.

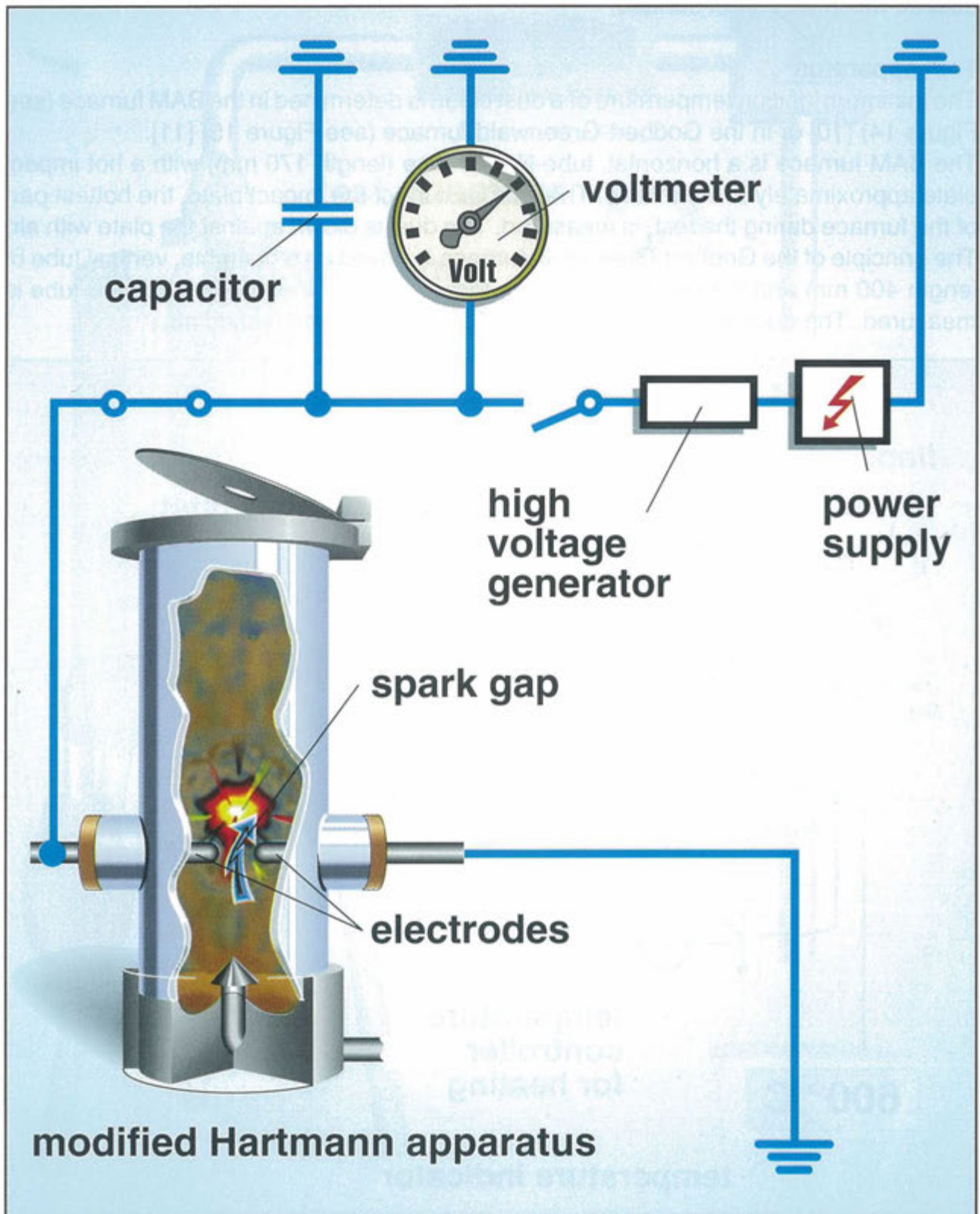


Figure 13: Test apparatus for determining the minimum ignition energy of dust/air mixtures

3.6 Minimum ignition temperature of a dust cloud

The minimum ignition temperature of a dust cloud describes the ignition behavior of a dust/air mixture at a hot surface.

Test apparatus

The minimum ignition temperature of a dust cloud is determined in the BAM furnace (see Figure 14) [10] or in the Godbert-Greenwald furnace (see Figure 15) [11].

The BAM furnace is a horizontal, tube-like furnace (length 170 mm) with a hot impact plate approximately in the middle. The temperature of the impact plate, the hottest part of the furnace during the test, is measured. The dust is blown against the plate with air. The principle of the Godbert-Greenwald furnace is based on a heatable, vertical tube of length 400 mm and diameter 36 mm. The temperature of the inner wall of the tube is measured. The dust is blown into the tube by a blast of compressed air.

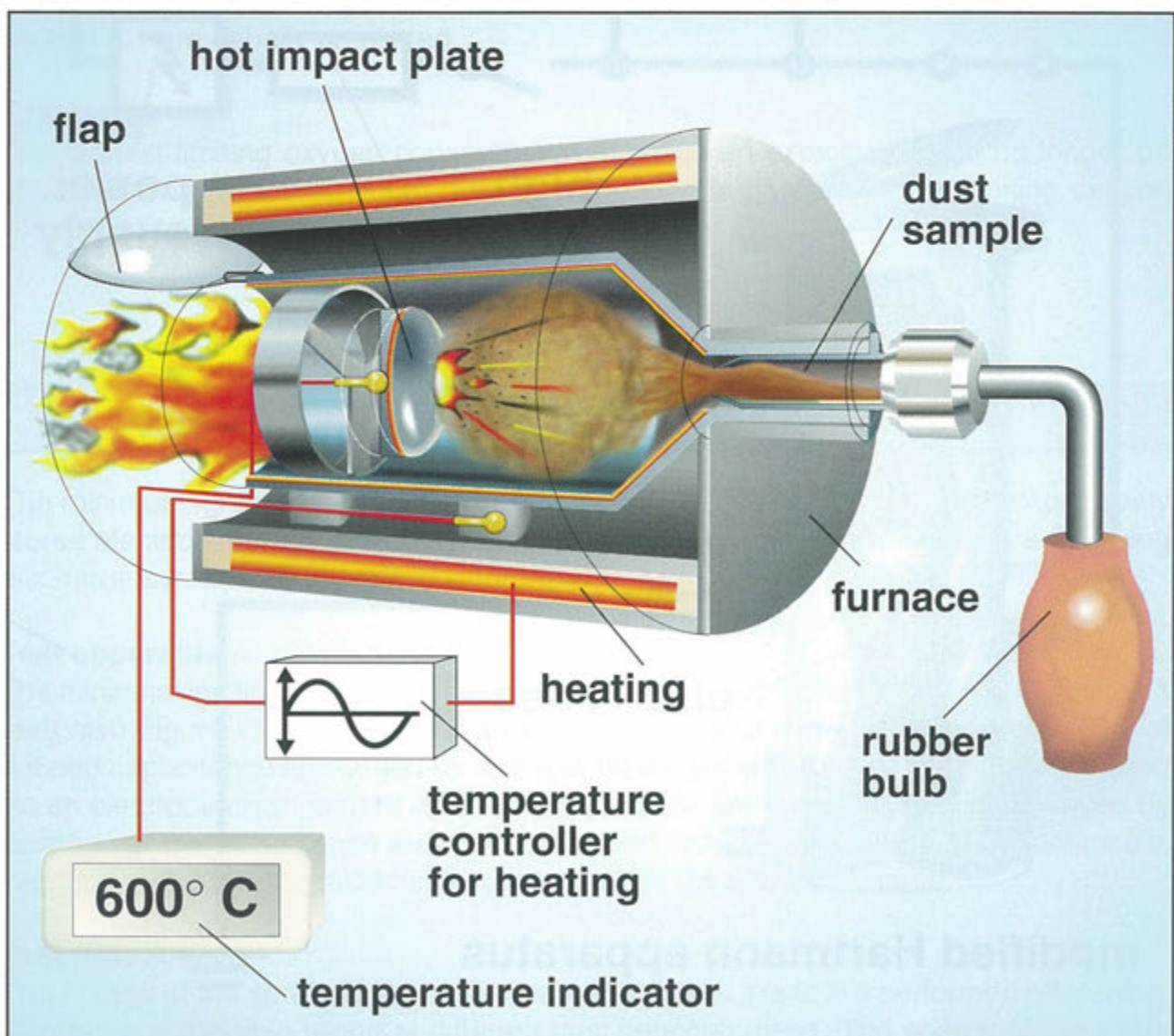


Figure 14: Test apparatus for determining the minimum ignition temperature of dust clouds (BAM furnace)

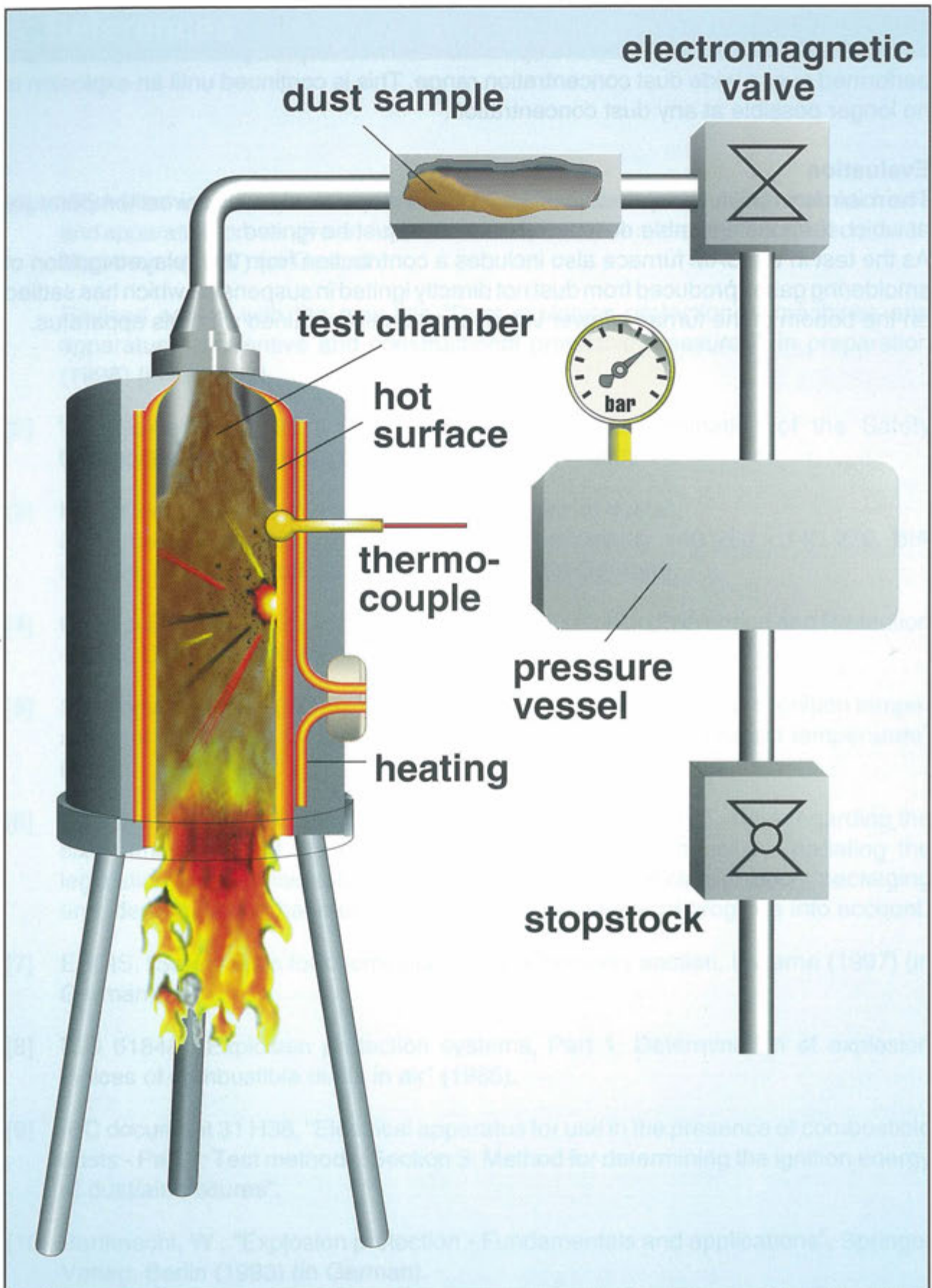


Figure 15: Test apparatus for determining the minimum ignition temperature of dust clouds (Godbert-Greenwald furnace)

Test procedure

The test temperature is lowered in steps of 10 K. After every temperature step, tests are performed over a wide dust concentration range. This is continued until an explosion is no longer possible at any dust concentration.

Evaluation

The minimum ignition temperature of a dust cloud is specified as the lowest temperature at which the most ignitable dust/air mixture could just be ignited.

As the test in the BAM furnace also includes a contribution from the delayed ignition of smoldering gases produced from dust not directly ignited in suspension which has settled on the bottom of the furnace, lower values are usually obtained with this apparatus.

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- [11] IEC document 31 H(CO)4: "Methods for determining the minimum ignition temperature of dusts - Part 2: Dust cloud in a furnace at a constant temperature".

ISSA publications (explosion protection)

ISSA

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Working party "Dust explosions"

Dust explosion protection of machines and apparatus

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Documentation of liquid gas (G)
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Static electricity - Ignition hazards and protection measures (G/E/F/It)
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The activities of the ISSA International Sections comprise:

- the exchange of information between interested bodies involved in the prevention of occupational risks;
- the organization of meetings of expert committees and working groups, round table discussions and colloquia on an international level;
- surveys and studies;
- the promotion of research work;
- the publication of information pertinent to its aims.

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