



ESCIS

Expert Commission  
for Safety in the Swiss Chemical Industry

**Safety**

**Series**

# **Drying of Solids**

**Booklet 6 2003**

**Translation of 2nd, revised German edition**



# PREFACE

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*Wherever chemicals are processed on an industrial scale, frequent use is made of drying processes. Solid substances in particular often have to undergo drying. When drying takes place, the substance being dried is subjected to physical stresses which can lead to hazardous situations and possibly accidents.*

*As long ago as 1985, ESCIS published "Trocknen von Feststoffen" ("Drying of Solids") as Booklet 6 of its "Safety" series of publications. The booklet contained measures for safe drying of solids in vacuum drying ovens and paddle dryers, as well as tests for safety assessment of the substance to be dried. Developments in relation to drying processes have made great progress and brought many changes. This has made a new edition of ESCIS Booklet 6 necessary.*

*Relative to the first edition of 1985, the new booklet deals with the drying process more broadly and less in relation to specific apparatus. This process-specific treatment of the basic operation of drying demonstrates the fundamental hazards and risks, as well as means of protection and safety measures. This provides a basis for risk evaluation which enables numerous drying installations to be assessed. The method used in the first edition of segregating products into safety classes for drying (SCD), and the corresponding assignment of protective measures, is no longer given prominence in the new edition, although it can still be used for a preliminary risk assessment in many cases. For this reason, extracts from the first edition are included here in Annex B for reference.*

*This second edition of ESCIS Booklet 6 "Drying", has been completely revised, and substantially expanded and deepened, relative to the first edition. Particular attention has been given to examples of applications which have come into practice since the first edition.*

*Basel, January 2003*

**Expert Commission for Safety in the Swiss Chemical Industry  
(ESCIS)**



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# DRYING OF SOLIDS

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## 1. Introduction

### 1.1 Changes and additions relative to the first edition

This booklet describes the hazards associated with the drying of solids. **Wherever possible, the limits at which the hazards occur are stated according to the most recent state of knowledge.** Suitable tests are explained for identifying and evaluating the safety-relevant properties of the substances to be dried.

The booklet describes protective measures corresponding to the state of the art, and thereby provides a basis for rational decisions regarding both the selection of appropriate and suitable measures and their practical application. The recommended safety measures meet **minimum requirements** which have proved themselves in industrial practice and under conditions of "good housekeeping". Exceptions and deviations from these safety measures should only be undertaken by specialists and after careful assessment of the specific case. If necessary, the opinion of external experts must also be obtained.

In contrast to the first edition, which was concerned exclusively with drying in paddle dryers and drying ovens, the new booklet has been expanded to include the most common thermal drying methods. As a consequence of this process-oriented (and no longer apparatus-specific) approach, the former segregation into safety classes<sup>1</sup> is no longer used, and specific consideration of minimum safety equipment for specific dryers has been omitted. **Only those safety problems which are relevant to the basic operation of drying, and their possible solutions, are considered.**

This means that this booklet cannot be used to select a suitable drying method for a particular situation, because such a selection depends primarily on the overall processing circumstances such as, for example, batch or continuous operation, the nature and consistency of the product to be dried (slurry or filter cake), as well as specifications for the dried product (flowability, particle size distribution) and its further processing (see, for example, [15]).

### 1.2 Scope of application

This booklet relates exclusively to drying processes in which the transfer of heat takes place by convection or contact; it does not apply to freeze, radiation, or microwave drying. Drying from the molten state is also

not included in the scope of this booklet; it must be assessed separately. The booklet is also restricted to the drying process itself, and to the apparatuses and their components intended for this purpose.

Other aspects of handling solids (charging, discharging, filling, and emptying of apparatus and containers for transportation, as well as separation, mixing, sieving, transportation, and storage) are not the subject of this ESCIS booklet.

The following dryers are considered:

- a) Based on the principle of contact drying
  - Paddle dryer
  - Drying oven
  - Nutsch dryer
  - Tray dryer
  - Thin-film dryer
- b) Based on the principle of convection drying
  - Spray dryer
  - Fluid-bed dryer/granulator
  - Belt (conveyor) dryer
- c) With integrated drying processes
  - Fluidized spray dryer<sup>2</sup>
- d) Integrated with other basic operations
  - Dryer-pulverizer<sup>3</sup>
  - Spin-flash dryer<sup>4</sup>

<sup>1</sup> Cf. Annex B, extracts from ESCIS Booklet 6, "Trocknen von Feststoffen" ("Drying Solids"), 1985 edition: safety classes for drying (SCD).

<sup>2</sup> Fluidized spray dryer = combination of a short spray dryer with an integrated fluid-bed dryer.

<sup>3</sup> Dryer-pulverizer = dryer in which the product is spread by means of a pulverizer and air current.

<sup>4</sup> Spin-flash dryer = transition-layer dryer in which the product is spread by means of an air current and agitators.

For combinations of these dryers, and for dryer types not listed here, or for similar drying processes, the contents of this booklet can be applied analogously. Examples and how they can be handled are:

- Rotary dryers – as paddle dryers
- Conical batch dryer with screw conveyor – as paddle dryers
- Pneumatic-conveyor dryer – as spray dryers
- High-speed paddle dryers or paddle dryers with high-speed agitators<sup>5</sup> – as thin-film dryers

In the case of reaction stages which take place in the drying apparatus before the actual drying itself (e.g. in paddle dryers), only the drying of the reaction product is considered. If the solvent to be removed is also, for example, the reactand, the relevant data for thermal reaction safety must also be taken into consideration when determining the drying temperature.

Further measures for ensuring safety and occupational hygiene in the general handling of dusts and solvents are given only partial consideration. The same also applies to measures for controlling the problem of heat accumulation when filling thermally unstable bulk products.

For further safety measures of specific relevance to drying operations, reference should be made to the ESCIS booklets 1 "Sicherheitstests für Chemikalien" [1], 2 "Static Electricity" [2], 3 "Inerting" [3], 5 "Milling of Combustible Solids" [4], and 8 "Thermal Process Safety" [5], as well as the other references listed in Chapter 6.

### 1.3 The process step "drying"

Drying is understood to mean the separation of liquid from a wet solid by vaporizing the liquid and removing the vapor. Since this process requires energy (mainly in the form of heat), it is referred to as **thermal drying**. Every thermal drying process is therefore characterized by a simultaneous transfer of heat and substance. Depending on the temperature, the drying process takes place either below the boiling point of the liquid being removed, or at its boiling point. The latter type of drying generally results in shorter drying times.

The liquid to be separated can be contained in the product to be dried in three different ways (or combinations thereof), viz.

- as free, unbonded liquid on the surface of the particles;
- in the case of hygroscopic substances, as liquid held in the pores of the particles by capillary and adsorption forces;
- as crystalline water bonded in the crystal structure of the solid substance.

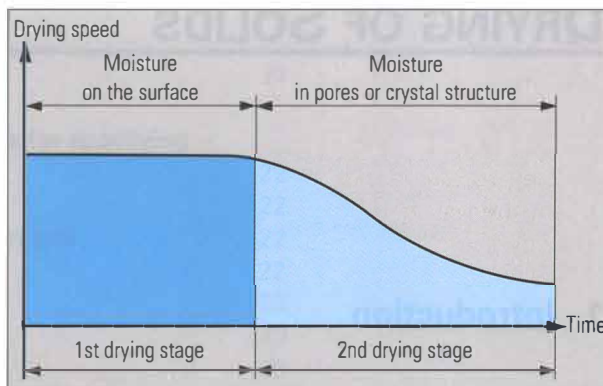


Fig. 1: Drying process over time (schematic) [6]

In view of this, the time and energy required to remove the liquid also differs correspondingly. The drying process can therefore generally be divided into at least two phases (see Fig. 1):

- In the first drying phase – with constant drying speed – the liquid evaporates or vaporizes rapidly and evenly on the particle surfaces. This results in formation of a liquid gradient inside the particle with subsequent transportation of liquid from within the particle to its surface by diffusion.
- The second drying phase – with steadily diminishing drying speed – begins when the amount of liquid arriving from within the particle is no longer sufficient to moisten the surface. The evaporation or vaporization process then takes place within the particle. The liquid inside the particle has to diffuse to the surface as vapor through layers of substance which are already dry. The resistance to be overcome increases and the speed of transportation decreases. The increase in resistance and decrease in speed are greater for larger particle diameters (or layer thickness) and for finer capillaries in the particles of solid substance. Toward the end of the drying process there is therefore usually a small amount of residual moisture which remains in the substance being dried.

Particularly in continuous processes it is therefore often appropriate to perform the two drying phases in two different dryers (pre- and post-dryer). In the first apparatus there is a short period of drying at a high temperature to remove the surface moisture, and in the second apparatus there is longer period of drying, possibly at lower temperatures, to remove the capillary moisture.

However, in practice the drying process is rarely continued until the theoretically possible residual moisture is attained, but terminated for economic reasons as soon as the requirements regarding the final moisture content in the dried product are fulfilled. It may be necessary for the dried product to be cooled before being discharged.

Besides wet solids, suspensions can also be thermally dried. (However, for economic reasons, it is then advan-

<sup>5</sup> Speed at circumference greater than 1 m s<sup>-1</sup>

tageous to remove as much liquid as possible in a preceding mechanical separation process.) Thermal drying does not include the drying of gases and organic liquids, such as the removal of water with the aid of sorption agents.

The heat required for drying consists mainly of:

- heat to raise the temperature of the product being dried to the drying temperature
- heat for the actual vaporization
- heat lost to the surroundings.

This heat must be supplied to the product being dried by conduction, convection, or radiation. Depending on how the heat is supplied, drying is separated into contact, convection, and radiation drying.

### 1.3.1 Contact drying

In contact drying, the heat required is supplied to the substance being dried indirectly by conduction. The substance being dried either rests on heated surfaces (e.g. in a vacuum drying oven), or is transported over them, or is continuously mixed (e.g. in tray dryers, paddle dryers).

Contact drying is usually performed under vacuum. This lowers the boiling point of the liquid to be vaporized so that temperature-sensitive substances can also be dried. The recovery of organic liquids by condensation in indirect heat exchangers (surface condensers) is therefore more readily possible than with convection drying.

Vacuum drying is usually performed in batch, because for continuous operation vacuum-tight entry and exit locks would be necessary. The vacuum is usually created by means of liquid-ring vacuum pumps or injector pumps, because these are less sensitive to uncondensed vapors. Rotary vane pumps are also increasingly used: the question of flame barriers then arises.

### 1.3.2 Convection drying

In convection drying, heat is transferred directly to the product being dried by means of a hot gas, usually air. There are several different ways in which the product being dried can be brought into contact with the hot air, for example:

- Air flowing over (e.g. circulating-air drying oven)
- Air flowing through (e.g. belt dryer)
- Fluidization (e.g. fluid-bed dryer)
- Entrainment (e.g. pneumatic-conveyor dryer)
- Atomization (e.g. spray dryer)

Without special measures, convection drying with air is generally only possible for substances which are chemically insensitive, and particularly not easily flammable. The use of inert gases instead of air is considerably more expensive, and the recovery of organic liquids by condensation or adsorption of the vapors is complicated.

### 1.3.3 Freeze drying

Freeze drying is a type of contact drying in which the product to be dried takes the form of an aqueous solution which is first frozen. The ice is then removed by sublimation under vacuum at temperatures below 0 °C. The remaining solid is flowable, porous, and easily re-soluble. Freeze drying is especially suitable for gentle drying of foodstuffs (e.g. coffee) and pharmaceutical products which are either highly temperature-sensitive or decompose after lengthy storage in dissolved form.

### 1.3.4 Radiation drying

In radiation drying, the necessary heat is transferred directly onto the product being dried in the form of infrared radiation from radiant heaters. By this means, very short drying times can be obtained, but the radiation temperature must be very high (400–2000 °C). Since infrared radiation hardly penetrates into the product being dried, this method of drying can only be used for drying thin films (e.g. paints or ceramic products).

## 2. Hazardous situations

### 2.1 General considerations

The various methods of drying (contact or convection) with their specific types of dryer lead to entirely different hazardous situations. For example, in contact dryers (except thin-film dryers) the occurrence of explosive dust clouds does not normally have to be considered a possibility, because movement of the product is generally only slow (see Table 1). On the other hand, in convection dryers operated with air, (except belt dryers), explosive mixtures of dust and air must almost always be considered a possibility. **The fact that at higher temperatures the mixtures are more readily ignitable, as well as the ignition sources present in each case, must always be borne in mind.** The ignition sources range from electrically or mechanically generated sparks and electrostatic discharges, through local overheating (caused, for example, by friction, foreign material, or smoldering agglomerates) and hot surfaces, to excessively high working temperatures which trigger self-ignition or decomposition reactions. The thermal stress on the product being dried also varies from one type of dryer to another, even though the same drying process may be used. For this reason, the extent to which a description of the hazardous situations associated with drying can be general and all-embracing is limited.

What follows is a description of the typical types of stress and the hazardous situations which result from them, both in general and in relation to specific dryers.

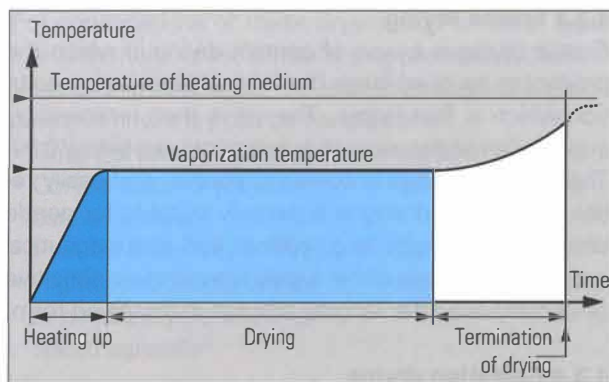


Fig. 2: Temperature over time (schematic) in product being dried [6]

## 2.2 General hazards of drying

### 2.2.1 Exothermic decomposition

For efficient drying, the product to be dried must have heat supplied to it. The accompanying increase in temperature inevitably causes a thermal stress on the product being dried. The magnitude and duration of this thermal stress depend on the selected drying process and the type of dryer. The purpose of the tests and measures recommended in this booklet is to ensure that if the dryer is operated as intended, the products are not subjected to an unacceptable thermal stress.

While the magnitude of the thermal stress is generally determined by the maximum temperature attained by the product being dried, its duration is defined by the mean period which the product being dried spends in the dryer (see Fig. 2).

However, if the dryer is operated other than intended, much longer thermal stressing of the product being dried may occur at the highest temperature possible in the dryer. Depending on the specific circumstances, this may initiate thermal decomposition of the product.

### 2.2.2 Fire/explosion hazards

**Explosive dust-air mixtures** may be present in convection dryers – at least locally – when they are in operation (see 2.3.2). Such mixtures can also occur in contact dryers when dust deposits are fluidized as a result of faults or certain operations, e.g. discharging. If effective ignition sources cannot be ruled out in such cases, dust explosions must be considered a possibility [8].

Contact with correspondingly **hot surfaces**, or under certain circumstances exothermal decomposition (see 2.2.1 and [1]), can cause the product to heat up to its glow temperature (see [1]) if insulation conditions are also favorable, as they may be if caking is present. The occurrence of flammable low-temperature carbonization gases is also possible. Depending on the properties of the product and the environmental conditions, this can initiate a fire or explosion.

**Friction heat** (caused, for example, by hot-running bearings or trapped foreign matter) can cause local overheating of the product being dried. Depending on

the properties of the product, this can initiate a dust fire or dust explosion.

**Smoldering agglomerates**, which may only act as an ignition source for a dust fire or dust explosion in downstream apparatus, are only possible for products with a *combustion number* (see [1]) greater than 3. For a dust explosion to be initiated by a smoldering agglomerate of the same dust, a certain ignitability is necessary. Current knowledge indicates that for this to occur, the product must have a *minimum ignition energy* (see [1]) of less than 1 joule.

In dryers with moving agitators, there may be mechanical stress on the product. At corresponding relative speeds of the moving parts, incendive **mechanical sparks** must be considered a possibility if faults occur. Mechanical sparks can only cause dust explosions of products with a minimum ignition energy less than 1 joule and an *ignition temperature* (according to BAM) (see [1]) below 500 °C.

Ignitions hazards from **electrostatic discharges** can arise from both *non-conductive* and *conductive*, but ungrounded, plant components. Under certain circumstances, incendive electrostatic discharges can also be emitted from non-conductive products being dried. (See also 2.2.5, [2] and [9].)

### 2.2.3 Self propagating decomposition<sup>6</sup>

With corresponding product properties and under strongly insulating conditions, **local overheating** arising, for example, from friction heat can cause a locally initiated decomposition reaction which propagates independently even under the exclusion of atmospheric oxygen. This occurs if the energy of decomposition, whose extent is at first limited, heats up adjacent product particles until they also spontaneously decompose. Details are given, for example, in the corresponding section of ESCIS Booklet 1, "Safety Tests for Chemicals" [1]. As well as the energy of decomposition, which in some cases is substantial, large quantities of possibly flammable decomposition gases may also be spontaneously released.

### 2.2.4 Hazards from hybrid mixtures

If the product being dried contains a **flammable liquid**, the occurrence of *hybrid mixtures* must be considered a possibility. Hybrid mixtures are easily ignitable and explode with greater violence than solvent-free dust-air mixtures. This hazard can already become significant if the solvent vapor concentration attains approximately 20 % of the lower explosion limit of the solvent (see [8]). Mathematically, this is the case at a temperature about 30 °C below the flashpoint.

<sup>6</sup> The expression "self propagating decomposition", formerly known as "deflagration", is interpreted in various different ways. Within the scope of this booklet, the following definition applies: Self propagating decomposition is decomposition initiated locally by an external ignition source which in contrast to combustion propagates autonomously even in the absence of atmospheric oxygen, the rate of propagation never exceeding the speed of sound.

Because the presence of solvent substantially reduces the minimum ignition energy of the mixture, which in the limiting case can fall to the value of the more readily ignitable component, greater attention must be paid to the hazard of ignition by electrostatic discharges (see [2]).

If a product being dried can release flammable low-temperature decomposition gases, it must be treated in a similar manner to one which contains flammable liquid at corresponding temperatures.

### 2.2.5 Hazards in dust separators

When dust separators are in operation, the occurrence of explosive dust-air mixtures – and under certain circumstances even of hybrid mixtures – must be considered a possibility. In association with the drying processes, the increased hazard from operation of the filter at frequently **high temperatures** must be given special attention. With increasing temperature, the minimum ignition energy of the products may decrease and their flammability increase.

Electro-filters must always be regarded as ignition sources, and only used after consultation with explosion protection specialists.

### 2.2.6 Hot discharging

The hazards of hot discharging are primarily product-specific and not determined by particular drying processes. For an assessment of these hazards, see also the section on “**Exothermal decomposition and conditions of heat concentration**” in ESCIS Booklet 1 “**Sicherheitstests für Chemikalien**” (“**Safety Tests for Chemicals**”) [1].

## 2.3 Hazards of specific dryer types

The sections that follow draw attention to hazards specific to particular dryers. It should be borne in mind that the assessments in the tables assume that the respective dryers are operated as intended. It is clear that these assessments no longer apply in the case of faults or other exceptional situations (for further information, see the footnotes to the tables).

### 2.3.1 Contact dryers

Fig. 3 shows several typical contact dryers. To explain the situation regarding hazards, a number of important process characteristics of contact dryers are summarized in Table 1. From the criteria stated in Table 1, indications of possible explosion hazards or the occurrence of explosive mixtures in contact dryers can be deduced: these are summarized in Table 2. To permit assessment of the associated explosion risks, Table 3 shows ignition sources for the same situations which must be taken into consideration as necessary. (Depending on the specific operational conditions, further ignition hazards must also be considered.)

Since not all the individual fields of these tables contain clear-cut hazard situations applying to all operating conditions, attention must be paid to the additional information in the respective footnotes.

The thermal stresses on the product being dried shown in Table 1 result in the thermal hazards shown in Table 4.

### 2.3.2 Convection dryers

Fig. 4 shows schematically several well-known convection dryers. To explain the hazard situations typical

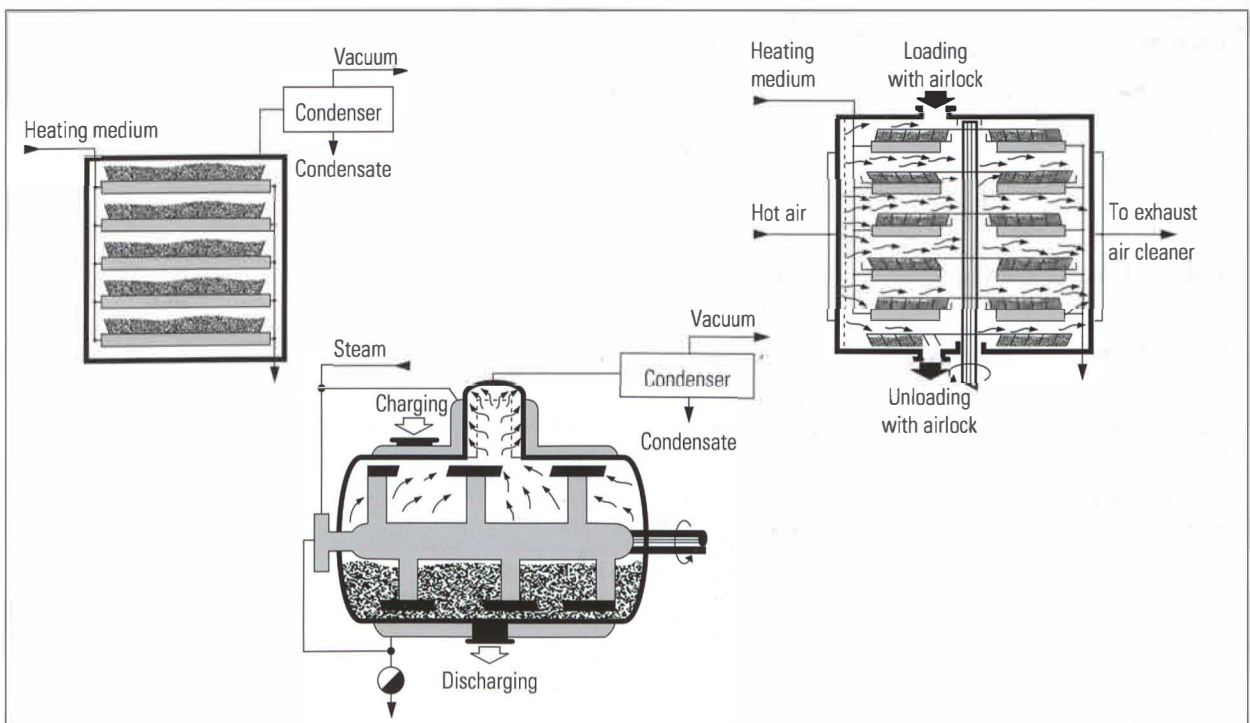


Fig. 3: Vacuum drying oven (left), paddle dryer, and tray dryer (right) [6]

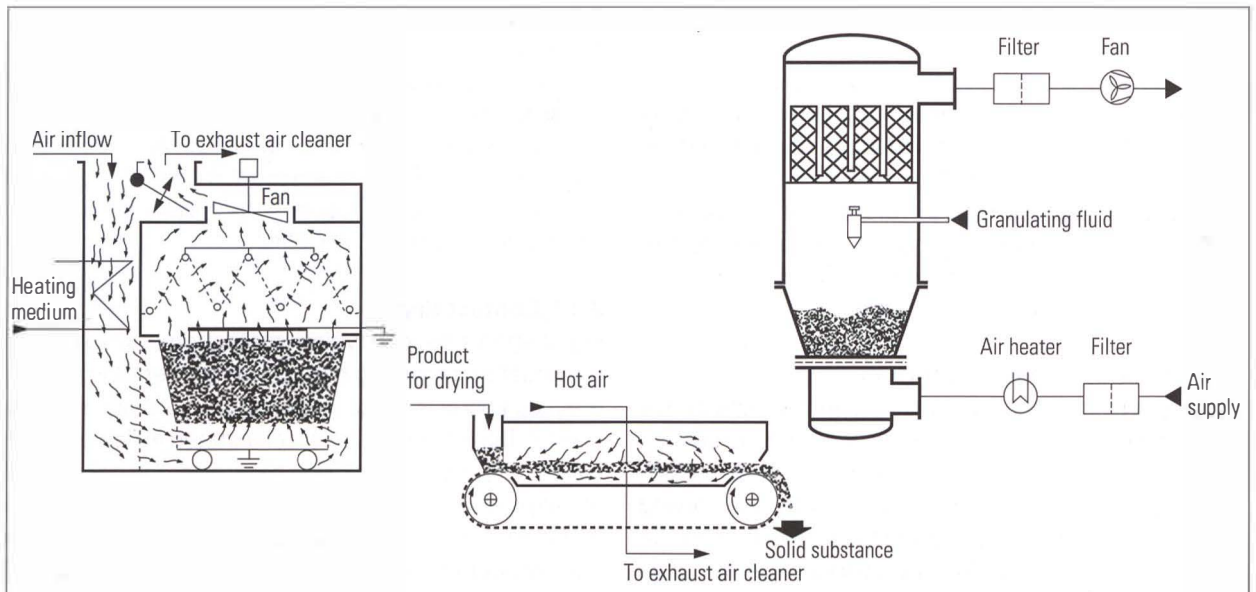


Fig. 4: Fluid-bed dryer for discontinuous operation (left), belt dryer [6], and fluid-bed dryer (right).

Table 1: Important safety-relevant processing characteristics of contact dryers

Processing Characteristic	Dryer Type				
	Drying oven	Paddle dryer	Nutsch dryer	Tray dryer	Thin-film dryer
Typical operating pressure	Vacuum/atmospheric	Vacuum	Vacuum	Vacuum/atmospheric	Vacuum/atmospheric
Normal time in dryer [h]	~24	~24	~24	~1	~0,1
Quantity of product <sup>①</sup> in dryer [kg]	<1000	≤5000	<3000	<300	<100
Product movement	None	Slow, mechanical	Slow, mechanical	Slow, mechanical	Slow, mechanical

① Product after drying

Table 2: Potential explosion hazards in contact dryers

Explosion Hazard	Dryer Type				
	Drying oven	Paddle dryer	Nutsch dryer	Tray dryer	Thin-film dryer
Dust explosion <sup>②</sup>	Practically impossible	Unlikely, see <sup>③</sup>	Unlikely, see <sup>③</sup>	Possible, only in filter	Possible
Solvent vapor explosion <sup>④</sup>	Possible, see <sup>⑤</sup>	Possible, see <sup>⑤</sup>	Possible	Possible	Possible
Hybrid mixture explosion <sup>⑥</sup>	Practically impossible	Unlikely, see <sup>⑥</sup>	Unlikely, see <sup>⑥</sup>	Possible, only in filter	Possible

② Dust explosions – unlike solvent-vapor explosions – require a fluidized cloud of dust.

③ A hazard of dust explosions is only possible with fluidized products and inadequate inerting or poor vacuum (e.g. greater than 100 mbar [abs.]). However, the corresponding effects are hardly relevant for safety. Under certain circumstances, a dust explosion hazard can arise when discharging.

④ When drying solvent-wet product.

⑤ Under vacuum operation, solvent-vapor explosions are only conceivable when charging/discharging, but under atmospheric conditions always.

⑥ Explosions of hybrid mixtures are only possible with fluidized products and inadequate inerting or poor vacuum (e.g. greater than 100 mbar [abs.]). However, the corresponding effects are hardly relevant for safety. The discharging process must be considered separately.

Table 3: Explosion risks from ignition sources in contact dryers

Ignition Source	Dryer Type				
	Drying oven	Paddle dryer	Nutsch dryer	Tray dryer	Thin-film dryer
Electrostatic discharge <sup>⑦</sup>	Practically impossible	Unlikely, see <sup>⑧</sup>	Possible	Practically impossible	Practically impossible
Mechanical spark	Practically impossible	Unlikely, see <sup>⑨</sup>	Practically impossible	Unlikely, see <sup>⑩</sup>	Possible
Impact stress	Practically impossible	Unlikely, see <sup>⑪</sup>	Practically impossible	Practically impossible	Possible
Overheating by foreign material	Practically impossible	Possible	Possible	Practically impossible	Possible

<sup>⑦</sup> This line contains only those electrostatic ignition hazards which still remain despite correct grounding of all conductive parts.

<sup>⑧</sup> When charging (loading) highly-insulating products (see ESCIS Booklet 3 "Inerting", Section 4.7).

<sup>⑨</sup> The hazard of mechanical sparking is only conceivable when beaters are used.

<sup>⑩</sup> Only significant for atmospherically operated dryers where there is no monitoring of residual dust before the air circulating fan.

<sup>⑪</sup> Impact stress is only possible when beaters are used.

Table 4: Thermal hazards in contact dryers

Thermal Hazard	Dryer Type				
	Drying oven	Paddle dryer	Nutsch dryer	Tray dryer	Thin-film dryer
Heat accumulation	Unlikely	Possible with agitator failure	Possible with agitator failure	Practically impossible	Practically impossible
Caking	Practically impossible	Possible	Possible	Practically impossible	Possible
Hazard from residual product	Practically impossible	Possible	Possible see <sup>⑫</sup>	Practically impossible	Practically impossible
Self propagating decomposition ("Deflagration")	Unlikely	Possible	Possible	Unlikely	Unlikely
Overheating by excessively hot inlet gas	Practically impossible	Practically impossible	Possible, only with recirculating gas	Possible	Practically impossible
Overheating by excessively hot surface	Possible	Possible	Possible	Possible	Possible

<sup>⑫</sup> A base layer up to 3 cm thick may result from the apparatus.

Table 5: Important safety-relevant processing characteristics of convection dryers

Processing Characteristic	Dryer Type					
	Spray dryer	Fluid-bed dryer/ granulator	FSD-dryer	Mikron dryer	Spin-flash dryer	Belt dryer
Normal time in dryer	<1 min <sup>⑬</sup>	~1 h	<1 h	~15 min	~15 min	~1 h
Quantity of product <sup>⑭</sup> in dryer [kg]	<100	<1000	<500	<300	<100	<2000
Product movement	Sprayed	Fluid bed	Fluid bed, transition layer <sup>⑮</sup> , spraying	Transition layer <sup>⑮</sup>	Transition layer <sup>⑮</sup>	None

<sup>⑬</sup> When post-drying with a downstream fluid-bed dryer, the time in the dryer can last up to 12 minutes.

<sup>⑭</sup> Product after drying.

<sup>⑮</sup> Transition layer = transition between fluid bed and riser (pneumatic transportation).

Table 6: Explosion risks from ignition sources in convection dryers

Ignition Source	Dryer Type					
	Spray-dryer	Fluid-bed dryer/granulator	FSD dryer	Mikron dryer	Spin-flash dryer	Belt dryer
<b>Electrostatic discharge</b> <sup>Ⓐ</sup>	Unlikely	Possible	Possible	Possible	Possible	Possible, see <sup>Ⓒ</sup>
<b>Mechanical spark</b>	Possible, only in disk atomizers	Practically impossible	Practically impossible	Possible	Possible	Unlikely, see <sup>Ⓒ</sup>
<b>Impact stress</b>	Practically impossible	Practically impossible	Practically impossible	Possible	Possible	Practically impossible
<b>Overheating by foreign material</b>	Practically impossible	Unlikely	Practically impossible	Possible	Possible	Unlikely
<b>Formation of smoldering agglomerates</b> <sup>Ⓓ</sup>	Possible	Possible	Possible	Unlikely, except in <sup>Ⓔ</sup>	Unlikely, except in <sup>Ⓔ</sup>	Practically impossible

<sup>Ⓐ</sup> This line contains only those electrostatic ignition hazards which still remain despite correct grounding of all conductive parts.

<sup>Ⓑ</sup> With non-conductive belt.

<sup>Ⓒ</sup> Only significant where there is no monitoring of residual dust before the air recirculation fan.

<sup>Ⓓ</sup> The hazard of smoldering agglomerates only arises for products with a combustion number greater than 3.

<sup>Ⓔ</sup> The hazard of smoldering agglomerates only arises if the product enters the heating area.

Table 7: Thermal hazards in convection dryers

Thermal Hazard	Dryer Type					
	Spray dryers	Fluid-bed dryer/granulator	FSD dryer	Mikron dryer	Spin-flash dryer	Belt dryer
<b>Heat accumulation hazard</b> <sup>Ⓐ</sup>	Possible, with deliberate hold-up in cone	Possible	Possible	Possible	Possible	Practically impossible
<b>Thermal decomposition in caking</b>	Possible	Possible	Possible	Possible	Possible	Practically impossible
<b>Hazard from residual product</b>	Practically impossible	Practically impossible	Practically impossible	Practically impossible	Practically impossible	Unlikely, see <sup>Ⓒ</sup>
<b>Self propagating decomposition</b> <sup>Ⓐ</sup> ("Deflagration")	Unlikely	Possible	Possible	Possible	Possible	Practically impossible
<b>Overheating by excessively hot inlet gas</b>	Possible	Possible	Possible	Possible	Possible	Possible
<b>Overheating by excessively hot outlet gas</b>	Possible	Possible	Possible	Possible	Possible	Possible
<b>Thermal decomposition in recirculation zones</b>	Unlikely	Possible	Possible	Unlikely, see <sup>Ⓒ</sup>	Unlikely, see <sup>Ⓒ</sup>	Practically impossible

<sup>Ⓐ</sup> If fan fails, e.g. because of a power outage.

<sup>Ⓑ</sup> Only for deposits on the housing.

<sup>Ⓒ</sup> Only arises if the product enters the heating area.

to them, Table 5 shows several important processing characteristics of the convection dryers considered. Because convection dryers are operated atmospherically, the occurrence within them of fluidized, explosive dust-air mixtures must be considered a possibility<sup>7</sup>.

When drying products wet with solvent, hybrid mixtures can occur. This can already be significant when the solvent-vapor concentration reaches approximately 20% of the value of its lower explosion limit. Because there is always an explosion hazard in convection

dryers, a table of such hazards (similar to Table 2 for contact dryers) is unnecessary.

Table 6 summarizes typical ignition sources for the individual dryers which should be taken into consideration in the evaluation of explosion protection measures in convection dryers: e.g. in the case of a mechanical defect in disk atomizers, the atomizing disk unit can act as an ignition source.

Table 7 gives an overview of the main thermal hazards of the various convection dryers:

- In the case of spray dryers, in normal operation the thermal stress on the products is relatively low even at high incoming air temperatures (vaporization heat, cooling effect) and only of short duration, provided no caking forms on the wall and upper part of the spray dryer. The safety measures whose description follows relate to this normal operation without caking. Otherwise, long-term thermal stress on the product must be assumed.
- However in fluid-bed apparatus, the products are generally thermally stressed for a longer period of time (up to several hours). This is also the case for continuously operating apparatus, because experience has shown that even in a fluidized bed with agitators, dead zones can be formed. In many cases, electrostatic charging of the product can also not be avoided.

### 3. Assessing the product to be dried

#### 3.1 Product information required

For every product to be dried, the protective measures to be taken must be specified before operational drying operations start. The foundation for the protective measures are the results of safety tests, from which the necessary safety notes and the limit values which must be observed (e.g. heating-medium temperature) as well as further restrictions on the selected drying procedure are derived, or can be deduced without ambiguity. The tests necessary for this purpose consist on the one hand of basic tests which are identical for all products, and on the other hand of more detailed dryer-specific tests.

In what follows, only the tests which are necessary in

each case are stated. Scope, execution, meaningfulness, and application area of the tests are described fully in [1].

In the case of products which are being dried for the first time, before the first charge is dried, these tests should be performed by gently drying a laboratory sample<sup>8</sup> and then repeated on product dried under operational conditions.

Repeating the tests should be considered whenever:

- There are changes in manufacturing methods, including post-drying processing
- There are irregularities in manufacturing
- There are changes in the source or specification of the primary materials and additives
- Production is relocated
- 5 years have elapsed since the last test, so that gradually occurring changes can also be detected.

To assist in performing the tests efficiently and in a manner adapted to the specific problem, the following items of information are generally helpful:

- Reason for testing
- Identity of the samples (if possible with structure formula[s] and data regarding their composition)
- Documentation of safety tests already performed
- Relevant physical/chemical data
- Special properties of the substance, such as toxicity, hygroscopicity, etc.
- Indications of any suspicion of other, especially dangerous, properties
- Any content of flammable solvents
- Target degree of dryness.

The documentation should also specify the drying method(s) to be used, the type of dryer selected, and the desired drying temperatures, and whether multi-stage drying methods are used.

If no desired drying temperature is specified, a relatively low value for the maximum allowable temperature (with corresponding safety margin) can be determined by a simplified method. If drying at a higher temperature would be more efficient, more detailed testing is necessary.

#### 3.2 Basic tests

- For all products to be dried, the following characteristics which are of basic importance for safety when drying are first determined:
- **Combustion behavior** (at 100 °C)
- **Thermal stability:**
  - *Testing for exothermic decomposition and/or self-ignition/self-heating are imperative for all products.*
  - *The testing conditions should be appropriate for the envisaged drying method.*
  - *The respective test methods, their performance, application, and meaningfulness are described in ESCIS Booklet 1, "Safety tests for chemicals" [1].*

<sup>7</sup> In spray dryers the occurrence of explosive dust-air mixtures at least in the lower part of the atomizer must always be considered a possibility. This applies even if rough calculations (solid substance throughput divided by air throughput) yield an average dust concentration which lies below the lower explosion limit of the dusty product.

<sup>8</sup> Gentle drying (at 50 – 60 °C in vacuum) is important, because otherwise important thermal information could be lost.

Table 8: Additional product characteristics requiring assessment for contact dryers

Characteristic for Assessment	Dryer Type				
	Drying oven	Paddle dryer	Nutsch dryer	Tray dryer	Thin-film dryer
Dust explosion behavior	Not required	Not required	Not required	Not required	Required
Minimum ignition energy	Not required	Not required	Not required	Not required	Required
Ignition temperature (BAM)	Not required	Not required	Not required	Not required	Required
Impact sensitivity	Not required	see <sup>29</sup>	Not required	Not required	Required
Self propagating decomposition	Not required	See 3.2	See 3.2	Not required	Not required
Heat accumulation in Dewar	See 3.2	See 3.2	See 3.2	Not required	Not required
Hot-storage test	See 3.2	Not required	Not required	Not required	Not required

<sup>29</sup> Only required for paddle dryers with beaters.

Table 9: Additional product characteristics requiring assessment for convection dryers

Characteristic for Assessment	Dryer Type					
	Spray dryers	Fluid-bed dryer granulator	FSD dryer	Mikron dryer	Spin-flash dryer	Belt dryer
Glow/ignition temperature <sup>25</sup>	See <sup>25</sup>	Not required	See <sup>25</sup>	Not required	See <sup>25</sup>	Not required
Dust explosion behavior <sup>26</sup>	Required	Required	Required	Required	Required	see 4.4.1
Minimum ignition energy	See <sup>27</sup>	Required	Required	See <sup>27</sup>	See <sup>27</sup>	Not required
Ignition temperature (BAM)	Required	Required	Required	Required	Required	Not required
Impact sensitivity	Not required	Not required	Not required	Required	Required	Not required
Heat accumulation in Dewar container	Not required	See <sup>28</sup>	See <sup>28</sup>	Not required	Not required	Not required
Self propagating decomposition	See 3.2	See 3.2	See 3.2	See 3.2	See 3.2	See 3.2
Hot-storage test	See 3.2	See 3.2	See 3.2	See 3.2	See 3.2	See 3.2

<sup>25</sup> Determination of the glow temperature is only necessary if an air inflow temperature close to a distinct decomposition is desired and caking cannot be ruled out. If the product to be processed contains a flammable liquid, the ignition temperature of the liquid must also be determined (it is generally shown in the usual tables).

<sup>26</sup> For a first screening, testing in the modified Hartmann Rohr is sufficient. Depending on the results, additional measurements in the 20 l sphere may be necessary. The comments in this line relate only to the actual drying operation itself. Discharging, filtering, etc. are not taken into consideration!

<sup>27</sup> Determination of the minimum ignition energy is necessary for an assessment of electrostatic hazards in the downstream filter.

<sup>28</sup> Power outage failure can lead to safety problems through heat accumulation conditions.

For all substances, the quantity of gas evolved with complete de-gassing is specified, with indication as to whether the gases evolved are flammable. This information is important for determining the possible consequences of smoldering fires and decomposition reactions (quantities of gas, occurrence of explosive mixtures).

Whether or not it is necessary to test for heat accumulation or conduct a hot-storage test, must be decided depending on the results of the aforementioned tests for exothermy.

Whether or not testing for self propagating decomposition (formerly "deflagration") is required, is decided depending on the chemical structure or the results of the decomposition test. Because of the higher temperatures when drying, this test is performed at 100 °C.

A decision as to the necessity of testing for autocatalytic decomposition is also made depending on the results of the tests for exothermy.

### 3.3 Dryer-specific tests

#### 3.3.1 Contact dryer

For drying in contact dryers – depending on the type of dryer envisaged – the product characteristics summarized in Table 8 can be of significance in addition to the aforementioned tests.

#### 3.3.2 Convection dryer

For drying in convection dryers – depending on the type of dryer envisaged – the product characteristics summarized in Table 9 can be of significance in addition to the tests stated in 3.2.

## 4. Recommended protective measures

In addition to the measures listed here – which relate only to the drying process itself – it is assumed that the basic requirements for ensuring safety and occupational hygiene when handling flammable dusts and/or solvents are fulfilled. These also include, among others, the regulations regarding explosion protection in the area surrounding the apparatus (division into zones) and the explosion protection of downstream apparatus (dust separators, filters, transportation containers, mixers). Experience has shown that the hazards of static electricity – e.g. through insufficiently grounded personnel – must be given special attention.

Any exceptions or deviations from the measures stated below may only be made by specialists and after careful testing of the specific case. If necessary, the opinion of external specialists must also be obtained.

### 4.1 General safety measures for drying operations

**Periodic checks:** All safety installations must be periodically checked, maintained, and, where necessary, tested. These checks must be documented.

**Cleaning:** The dryers must be checked to ensure that they are completely empty, and at suitable time intervals (at least every time the product is changed) they must be checked for cleanliness (encrustation) and cleaned if necessary. The plant manager must specify the frequency of the checks and the criteria for cleanliness. Consideration must also be given as to whether special installations or measures are required or necessary to prevent large accumulations of material in the dryer such as, for example, caking, blockages in the discharging equipment. For products which can decompose autocatalytically, these measures must be given special attention.

**Hot discharging:** If dried product is transported hot into large containers (e.g. larger than 0.2 – 0.5 m<sup>3</sup>), consideration must be given as to whether under the prevailing conditions measures are necessary against the product-specific occurrence of heat-accumulation conditions or against the subsequent occurrence of smoldering fires (e.g. quarantine storage, temperature monitoring). For further information, see ESCIS Booklet 8 “Thermal Process Safety” [5].

**Power failure, emergency switchoff:** Suitable procedures must also be provided to prevent the occurrence of hazardous heat-accumulation situations under these conditions.

### 4.2 Product-specific safety measures for drying operations

**Very fast-burning products,** i.e. products with combustion number 6 (at 100°C), may only be dried under specially defined conditions after further tests and after special evaluation by experts.

**Impact-sensitive products** must be handled in the same way as those with combustion number 6. They must not be dried in thin-film dryers or in paddle dryers with beaters. Drying in micron and spin-flash dryers must also be avoided.

**Products capable of self propagating decomposition** where large quantities are involved must, in principle, never be dried in paddle dryers, nutsch dryers, fluid-bed dryers, FSD dryers, or Mikron dryers. If there are compelling reasons why this must be done nevertheless, special safety installations on the dryer, further tests to determine the speed of decomposition, and testing of means of terminating decomposition are required, because inerting and vacuum are ineffective for self propagating decomposition.

**Solvent-wet products** may normally only be dried in apparatus which is inerted or under vacuum. In this connection, attention must be paid to the general rules of explosion protection applying also to the area around the apparatus (separation into zones) and to the open handling of solvent-wet products (especially open charging of paddle dryers with solvent-wet product).

### 4.3 Specification of temperature limits

As is well known, exothermic (decomposition) reactions do not only start at a certain “starting temperature”, but in accordance with Arrhenius’ Law also take place at lower temperatures, though correspondingly more slowly. Their speed – or more precisely, the associated rate of heat production – is designated “non-critical” if the heat produced can be removed sufficiently quickly for no safety-critical (i.e. self-accelerating) temperature increase to occur. To determine the so-called “critical” or “starting temperature”, the reaction-dependent rate of heat production must be compared with the speed of heat removal, which depends on the geometry of the apparatus as well as the properties of the substance and the characteristics of the surround-

ings. Such “critical” or “starting temperatures” are therefore never properties of the substance itself, but parameters specific to the individual case which depend greatly on the volume and geometry of the apparatus in question.

The mathematical treatment of such generally dynamic processes is very complex. With certain simplifications – which for practical applications are entirely realistic – the critical heat production ( $\dot{q}_{crit}$ ) for non-agitated substances in a non-insulated apparatus can be calculated according to Frank-Kamenetskii [7]. The applicable formula stated in [5] is:

$$\dot{q}_{crit} = \delta_c \cdot \frac{\lambda \cdot R \cdot T_0^2}{\rho \cdot r^2 \cdot E_a} \quad [\text{W kg}^{-1}] \quad (1)$$

		typical values
$\lambda$	thermal conductivity	0,1 W m <sup>-1</sup> K <sup>-1</sup>
$R$	8,314 J mol <sup>-1</sup> K <sup>-1</sup>	
$\rho$	density	1 000 kg m <sup>-3</sup>
$r$	radius [m]	
$E_a$	activation energy	100 000 J mol <sup>-1</sup>

$\delta_c$  is a dimensionless criticality parameter which takes one of the following values depending on the shape in question:

- $\delta_c = 3,32$  (highest value possible) for a sphere with radius  $r$
- $\delta_c = 0,88$  (smallest value possible) for a layer insulated on one side whose thickness  $r$  is much less than its other two dimensions
- $\delta_c = 2,5$  for a cube (where  $r =$  half the length of one edge)
- $\delta_c = 2,37$  for a cylinder with height = 3×radius  $r$

Fig. 5 Critical heat production in a solid body [5]

For example, if the same product is to be transferred hot into another container (hereafter designated with index 1 instead of 0), for example into a 200 l drum instead of a 5 l cylinder), the critical heat production in the new container is calculated from:

$$\frac{\dot{q}_{crit,1}}{\dot{q}_{crit,0}} = \frac{\delta_{c,1}}{\delta_{c,0}} \cdot \frac{r_0^2}{r_1^2} \quad (2)$$

However, according to the Arrhenius equation, the heat production  $\dot{q}$  produced by the (undesired) decomposition reaction depends on the temperature. If  $\dot{q}_0$  is known for  $T_0$ , it follows that for  $\dot{q}_1$  at temperature  $T_1$ :

$$\dot{q}_1(T_1) = \dot{q}(T_0) \cdot \exp\left[-\frac{E_a}{R} \cdot \left(\frac{1}{T_1} - \frac{1}{T_0}\right)\right] \quad (3)$$

For the new critical heat production  $\dot{q}_{crit,1}$  derived above (from the new container geometry), the new “critical” temperature  $T_1$  can be calculated which is the maximum temperature at which the product can be stored

without occurrence of a critical increase in temperature. However, to perform this calculation, the activation energy ( $E_a$ ) of the decomposition reaction in question must be known or estimated.

The problem when specifying temperature limits for drying processes now becomes that of extrapolating “starting temperatures” measured in the laboratory with small sample quantities and in an apparatus of a different sensitivity to practical situations.

The practice known in industry of specifying “safety margins” was also not only empirical (i.e. based on actual occurrence), but was also based on scaling up from laboratory to operational conditions in the manner stated above. This is illustrated by the following example from [5]:

*Suppose the heat output of a decomposition to exceed the detection limit (sensitivity) of the experiment of 500 mW kg<sup>-1</sup> (corresponding approximately to a 200 ml Dewar experiment). Under somewhat conservative assumptions, this value corresponds approximately to the critical heat production of an uninsulated cylindrical volume of 5 l (Formula (1) in Fig. 5). This means that the detection limit of our experiment – the calculated “critical temperature” – corresponds directly to the volume of a cylinder with a capacity of 5 l.*

*If now the stability of the same substance in a 200 l drum is to be evaluated, a lower heat output is already critical. According to equation (2), for identical geometry it is inversely proportional to the square of the radius of the cylinder. In our case, this therefore results in a “thermal scale-up factor” of about 12, although the critical heat output is only 40 mW kg<sup>-1</sup>.*

*By applying equation (3), the associated (critical) temperature is now determined above which the possibility of a thermal explosion must be considered in the 200 l drum. This is illustrated in Fig. 6 (taken from [5]).*

*For this extrapolation, the activation energy of the decomposition reaction in question must be known, but this is often not the case. Therefore, as an assumption, a rather lower value is used for the activation energy. Fig. 6 shows this calculation for three different values. The result indicates clearly that substances with a low activation energy (flat curve) are more hazardous in relation to heat accumulation than those with a high activation energy!<sup>9</sup> The three selected values of 40, 70, and 100 kJ mol<sup>-1</sup> result in critical temperatures for the 200 l drum of 60, 100, and 110 °C respectively.*

However, Fig. 6 also shows clearly that the scaling-up covered by this “safety margin” is limited. For larger scaling-up factors, the conservatively estimated critical

<sup>9</sup> On the other hand, substances with a high activation energy are sensitive to temperature fluctuations (e.g. of heating medium temperatures) but less susceptible to heat accumulation hazards [1].

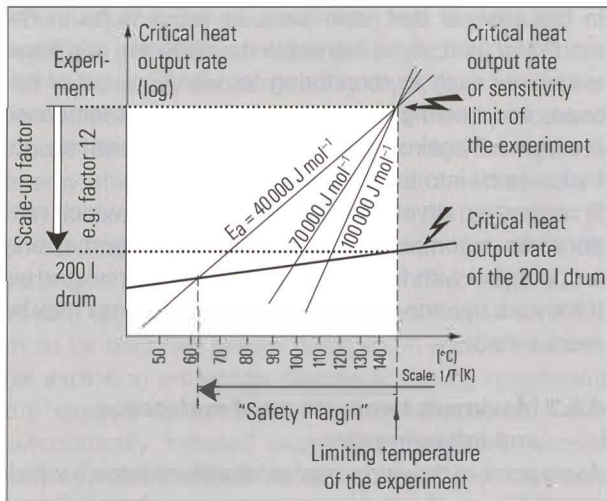


Fig. 6: Comparison of critical heat output rates in different volumes [5]. (The slope of the straight line of the critical heat output rate of the 200 l drum stems from the fact that in accordance with equation (3) this is also temperature-dependent under otherwise identical conditions.)

temperatures – and therefore also the maximum allowable drying temperatures – are unrealistically low [5]. In this connection, [1] states:

“Interpretation of the test results must be based on solid knowledge of the fundamentals of chemistry and physics, and the assessor must be thoroughly familiar with the operational conditions. Decisions including a factor of judgment and experience, and the acceptance of corresponding responsibility, are therefore unavoidable.”

For readers less familiar with the testing instruments, the detection limits of the most common testing arrangements for determining exothermic decomposition characteristics over the required range of sample sizes are summarized in Fig. 7 (taken from [10]).

Operators of drying equipment themselves will be the best judges of which radii and layer thicknesses  $r$ , and which criticality parameters  $\delta_c$ , are realistic for converting the test results to their operational situation using equation (1), and also of where such a conversion is not meaningful because of the short period spent in the dryer or other operational factors.

Annex A contains an example of the procedure used for determining the temperature limits for drying taken from a chemical company in Basel.

## 4.4 Additional measures for contact drying

### 4.4.1 Temperature of heating medium

Based on the measurements, the testing laboratory recommends a maximum allowable temperature for the heating medium. When doing so, it is usual to assume that the period of thermal exposure of the product to this temperature of the heating medium is less than two days.

If no desired drying temperature is specified, a rela-

tively low value for the maximum allowable temperature can be determined by a simplified testing procedure involving less effort. If a higher temperature of the heating medium would make drying more efficient, more detailed testing is necessary.

The maximum temperature of heating medium allowed in the specific plant must always be adhered to and must be safeguarded by corresponding measures (including redundancy if indicated by the risk assessment).

### 4.4.2 Heat accumulation

According to experience, heat accumulation conditions only occur relatively infrequently in normal operations because of the limited layer thickness and/or on account of the mixing that takes place during drying. The heat accumulation conditions which can accompany common faults (e.g. power outage) must be taken into consideration when specifying the maximum allowable temperature of the heating medium. However, more extreme heat accumulation conditions may occur when discharging the product (see above, and 2.2.6 Hot discharging).

### 4.4.3 Exposure to oxygen

The testing procedure for air drying covers exposure to oxygen. Conditions may be permitted for vacuum drying which would be critical if air were to enter. This situation must be taken into account by correspondingly securing the vacuum (relief with nitrogen; possibly cooling before discharging).



Fig. 7: Sensitivities of measuring apparatuses for decomposition tests after [10]. (For full descriptions of the instruments and abbreviations, including names of manufacturers, see [5].)

#### 4.4.4 Vacuum drying

Vacuum dryers must only be heated after evacuation. As protection against possible overpressure resulting from decomposition of the product, the doors or other openings of vacuum dryers can be unlocked after evacuation and before heating.

For products in burning class 4 or 5 which evolve perceptible quantities of decomposition gases, the following recommendations also apply:

- The dryer should only be charged when cold. At the end of the drying process, the vacuum should always be broken with nitrogen.
- Vacuum dryers should only be aired and discharged when the temperature of the dried product is lower than the allowable temperature for air drying. If this is not known, the dried product should be cooled to below 40 °C before discharging.<sup>10</sup>
- If the vacuum is broken, an alarm must be triggered. When this happens, the dryer must be filled with inert gas (either manually or automatically) and cooled.<sup>11</sup>

### 4.5 Additional measures for convection drying

#### 4.5.1 Belt dryers: a special case

Belt dryers are a special case of convection drying in that there is no fluidization of the product in the actual drying area. In relation to safety, they are treated as drying ovens, so the only safety measures applying to them in addition to those stated in 4.1 are those in 4.2. Any fluidization of dust in the discharge area must be considered separately.

#### 4.5.2 Explosion and fire protection measures

Convection dryers for drying flammable dusts must be protected against possible explosions of dust or hybrid mixtures by inerting, explosion venting, or explosion suppression according to the state of the art (see [8]). As a general rule, spray dryers should be equipped with a water-deluge system which is independent of the product stream.

Any exceptions for existing apparatus which is still unprotected must be reviewed on a case-by-case basis with safety experts.

In the case of disk atomizers, to avoid faults in the atomizing unit, it is advisable to consider additional measures such as monitoring for vibration, out-of-balance, and bearing temperature, as well as increased protection against penetration of lubricating or hydraulic oil into the atomizer.

If convection dryers are used for products which may generate decomposition gases (see 3.2), they may only be operated with fresh air and not with recirculated air. If they are operated with inert gas, the inert gas may be recirculated.

#### 4.5.3 Maximum temperature of surfaces and inflowing air

At no point in the dryer may temperatures occur which are as high as the ignition temperature of the fluidized dry dust. This applies even to sources of heat which are only temporarily present (e.g. handheld lamps at observation windows). If the product to be processed contains a flammable liquid, or if flammable gas may be evolved, the temperature must also not be allowed to attain their ignition temperatures.

#### 4.5.4 Avoiding heat-accumulation situations

(for general notes, see 4.3)

Convection dryers must be connected in such a way that when the heating is switched off, the fan continues to run for a sufficiently long time to prevent occurrence of a heat-accumulation situation. If the fan fails, the heating must switch off automatically and an alarm must be triggered. If necessary, additional measures to prevent heat accumulation must also be triggered automatically. In each individual case an assessment must be made as to the necessity of providing special measures for when power outages occur.

#### 4.5.5 Temperature limits for spray drying

The temperature limits stated here relate to normal operation of the dryer without caking. Each installation must therefore be assessed for the necessity of providing special equipment (such as observation windows, level sensors, fluidization, vibrators, etc.) to prevent large amounts of material building up in the dryer – e.g. heavy deposits on the sides and top, blockages in the discharging equipment.

The air inlet temperature (gas inlet temperature if operated with inert gas) is therefore selected taking into account the decomposition temperature (product quality) determined in the test for exothermy in air (inert gas). However, if deposits on the sides are a possibility, the maximum allowable inlet temperature must be determined according to the procedure described in 4.3.

To take account of the thermal stress of the (sometimes desirable) hold-up in the discharger, the maximum allowable air outlet temperature is specified as described above, but now based on the 24-hour hot-storage test.

<sup>10</sup> The temperature of the dried product can normally only be measured after it is discharged. The time required for cooling depends, among other things, on the size of the apparatus, the type and quantity of the dried product, the residual moisture, and the temperature of the heating medium. The cooling time can be determined by experiment under operational conditions. It must then be noted in the process documentation. Typical empirical values for paddle dryers between 4 and 6 m<sup>3</sup>:

- After 30 min cooling time the product temperature is approximately  $\frac{1}{2}$ – $\frac{2}{3}$  of the heating medium used in °C.
- Cooling to 40–70 °C takes about 3 hours.
- Cooling to 25–30 °C can take more than 4 hours.

<sup>11</sup> If in the case of a power outage, for example, effective cooling is no longer possible, switching over to cooling at least turns off the heating.

However, the temperature must not be allowed to reach the maximum temperatures specified in 4.5.3. If it is necessary to open a spray dryer, (e.g. for inspection purposes) in which a product is being processed whose dust-air mixture has a low minimum ignition energy, this must be done with great care because it may cause additional fluidization in the presence of smoldering agglomerates. As a minimum measure, cooling to below 40 °C is recommended, but the procedure for ensuring that sufficient cooling takes place must be specified before doing so. In addition, technical explosion protection devices (including monitoring the oxygen concentration in inerted apparatus and automatically initiated protective measures) should only be made ineffective after cooling to room temperature has taken place. It is also recommended, if conditions permit, thoroughly to dampen the product remaining in the apparatus with cold water before opening the apparatus or switching off the safety devices.

#### **4.5.6 Temperature limits for fluid-bed processes**

The method described in 4.3 can be applied here to determine the critical temperature of deposits on the walls. However, if the method is applied to heat-accumulation in the settled fluidized bed (caused, for example, by fan failure) it yields impracticably low drying temperatures. For this reason, the following procedure has proven itself in practice: By means of tests for exothermy in the presence of a fresh air supply, and a 24-hour hot-storage test, the lowest temperature is determined at which exothermy can still be observed. (For inerted apparatuses the test is repeated in a mixture of 92 % N<sub>2</sub> and 8 % O<sub>2</sub>.) The maximum allowable air-inlet temperature is then specified so that in the 24-hour hot-storage test no exothermy occurs. (It should normally be below the melting point or melting range of the product). However, the maximum temperatures specified in 4.5.3 must not be reached.

It is advisable to keep the surface temperature of the air heating elements as low as possible and to provide the heating by means of a heat-transfer medium (steam, hot water, etc.). If electrical-resistance heating is used, the heating elements should not be located in the apparatus itself, but at some distance from the sieve bottom. It is also advisable to build in a metallic air filter between the heating unit and the product room to prevent the transportation of hot particles (e.g. rust).

#### **4.6 Considerations relating to integrated drying methods**

Integrated drying processes generally combine two stages of convection drying: a spray-drying stage and a fluid-bed stage. The safety measures necessary are those given by the respective criteria for convection dryers.

#### **4.6.1 Explosion protection measures**

Installations of integrated drying processes for drying flammable dusts must be protected according to the state of the art by inerting, explosion venting, or explosion suppression (see [8]) against possible dust explosions or explosions of hybrid mixtures. They should also be equipped with a water-deluge system which is independent of the product flow.

#### **4.6.2 Temperature limits**

Two temperature limit values are determined: the maximum allowable temperature of the air leaving the spray-drying stage (see 4.5.5) and the maximum allowable temperature of the air entering the fluid-bed stage (see 4.5.6). The lower of the two values is taken as the maximum allowable air temperature both for entry to the fluid-bed stage and for leaving the spray-drying stage. The temperature should not be higher than 150 °C.

Within the limits stated in 4.5.3 and 4.5.5, the temperature of the air entering the spray-drying stage may be freely selected.

#### **4.7 Considerations relating to drying with integrated grinding and mixing operations**

In pulverizer and spin-flash dryers, convection drying, size reduction, and size classification operations are integrated into a single processing stage. At high temperatures, the drying-related thermal stress is increased by the additional mechanical stress.

For processing flammable products in these dryers, the measures stated in 4.1 and 4.5 must be fulfilled completely. The exceptions stated as possibilities in 4.5.2 are not allowed here.

If the product to be dried contains a flammable liquid, non-inerted dryers must be operated in such a way that the average concentration of flammable vapors in the air stream does not exceed 20 % of the lower explosion limit of the solvent. As already mentioned, at a temperature of approximately 30 °C below the flashpoint this is mathematically the case.

The temperature of the air on entering can be selected taking into account the decomposition temperature determined in the laboratory and the melting point. (As a rule, the maximum temperature of the outflowing air should not exceed 150 °C).

#### **4.8 Considerations relating to drying integrated with other basic operations**

The safety measures necessary for processes in which drying is performed in parallel with other basic physical operations must be determined for each separate case. When doing so, as well consulting the responsible safety specialists it may also be necessary to involve other experts.

## 5. Terms and definitions

### Burning number

The result of the combustion test is expressed by the allocation of a burning number (the burning number of common salt is 1, gunpowder 6).

### Combustion behavior

The combustion behavior describes the appearance (and speed of propagation) of a fire in a dust layer [1], [13].

### Conductive / non-conductive

In the context of static electricity, liquids whose specific resistance is less than  $10^8 \Omega \cdot m$  are defined as conductive, whereas those with a higher specific resistance are defined as non-conductive.

For dusts/powders the respective limit is  $10^{10} \Omega \cdot m$ .

Solid bodies are defined as conductive if their specific resistance is less than  $10^6 \Omega \cdot m$  and as insulating if the respective value is higher than  $10^{11} \Omega \cdot m$ ; if the respective value lies between these two limits, they are described as "dissipative".

### Deflagration

An explosion which propagates at less than the speed of sound (ISO 8421-1, 1987-03-01;1.11) [13].

Spontaneous decomposition also refers to the combustion of an explosive substance in open air without detonation [14]. See also footnote 6.

### Detonation

Explosion which propagates faster than the speed of sound, characterized by a shock wave (ISO 8421-1; 1987-03-01, 1.13) [13].

### Exothermic decomposition

A reaction which occurs in the absence of atmospheric oxygen which can cause spontaneous heating and, if accompanied by the release of gas in an enclosed apparatus, can cause an increase in pressure (volume-dependent) [12].

### Explosion

Process of rapid combustion with perceptible increase in pressure [12].

Sudden reaction involving oxidation or decomposition and an increase of temperature or pressure, or both concurrently [13].

### Explosion point, upper/lower

The temperature of a flammable liquid at which the concentration of the saturated vapor in air is also the upper/lower explosive limit [13].

### Explosive atmosphere

A mixture of air and flammable gases, vapors, mists, or dusts under atmospheric conditions in which the combustion process, once ignited, spreads to the entire unburnt mixture [13].

### Flashpoint

The flashpoint is the lowest temperature – at a pressure of 1013 mbar – at which under defined conditions vapor is evolved by the product being tested in such quantity as to form a mixture of vapor and air capable of ignition by an external ignition source [1].

### Glow temperature

The lowest temperature of a hot exposed surface at which dust deposited on the surface in a 5-mm-thick layer is ignited within 2 hours [12].

### Hazardous explosive atmosphere

An explosive atmosphere which causes damage if it explodes [13].

### Heat accumulation

The occurrence in practice of a hazardous accumulation of heat depends on the ratio of heat dissipation to heat production (from the decomposition reaction). As long as the heat dissipation capacity under working conditions (storage conditions) is greater than the rate at which heat is produced, no hazardous situation arises. However, if the energy released by exothermic decomposition cannot be dissipated, self-heating occurs which causes the decomposition reaction to be accelerated to the maximum heat production rate. This may result in a thermal explosion.

### Hybrid mixture

A mixture of air and combustible materials in different states of aggregation [13].

### Ignition temperature

The ignition temperature is the lowest temperature of a hot surface on which a flammable liquid just ignites, or which ignites fluidized dust with which it comes into contact [1].

### Limiting oxygen concentration

The maximum concentration of oxygen in a mixture of a flammable substance with air and inert gas at which no explosion occurs, determined under specified experimental conditions [13].

### Minimum ignition energy

The lowest value of electrical energy stored in a capacitor which when discharged by varying the parameters of the discharge circuit just suffices to ignite the most ignitable mixture of dust and air at atmospheric pressure and room temperature [12].

### Minimum ignition temperature of an explosive atmosphere

The ignition temperature of a flammable gas or of the vapor of a flammable liquid, or the minimum ignition temperature of a dust cloud, in each case under prescribed test conditions [13].

### Self-ignition

A process in which a layer of dust heated from all directions in the presence of air heats spontaneously and then ignites (volume-dependent) [12], [1].

### Self-ignition temperature

The temperature of the environment (storage temperature) at which spontaneous oxidative heating and then self-ignition occurs.

### Self propagating decomposition

Self propagating decomposition (formerly known as "deflagration") is a decomposition reaction initiated locally by an external ignition source which in contrast to combustion propagates autonomously even in the absence of atmospheric oxygen, the rate of propagation never exceeding the speed of sound.

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## Examples of proven practical procedures for specifying temperature limits

The procedure described below is taken from the internal regulations of a chemical company in Basel, Switzerland. It shows one possible way of implementing the recommendations of this booklet in practice.

### A1 Contact drying

#### A1.1 Drying in a vacuum or under inert gas

##### Basic testing (screening)

By means of isoperibole testing for **exothermic decomposition in an open container** (after Lütolf, see [1]) starting at 270 °C and then at decreasing temperature intervals of 10 °C, the limit temperature is determined at which for 8 hours the substance just displays no overheating (and no endothermic behavior).

A temperature 100 °C below this limit temperature is proposed as the highest allowable temperature for the heating medium. (The safety margin of 100 °C is based on the experience that when testing for heat accumulation in a Dewar container (see [1]), decomposition may be observed down to 100° C below this limit temperature.) However, the temperature should not exceed 170 °C (see 4.3.1) and should be safeguarded at 10 °C below the melting point of the substance being tested.

If this value is not acceptable for operational purposes, more detailed testing is performed.

##### Detailed testing

By means of **heat-accumulation tests in a Dewar container** at decreasing temperature intervals of 10 °C (starting, for example, 50 °C below the limit temperature determined above, but not higher than 180 °C), the temperature is now determined at which the temperature rise (in the case of an exothermic process) is less than 5 °C (and no endothermic decomposition is observed). The measuring period for this experiment should last at least three days. (If during this period a rise in temperature is observed, measurement is continued until the peak is passed.)

If at 10 °C above the temperature now determined there is overheating of more than 10 °C, or if reaction mass appears, a value 10 °C below this temperature is

proposed for the maximum allowable temperature for the heating medium. Otherwise, this temperature itself is the highest allowable temperature for the heating medium.

If the temperature of the heating medium desired for operational purposes is higher than the value of 170 °C stated in 4.2.1, the testing temperatures are increased by the amount of the difference.

#### A1.2 Air drying

##### Basic testing (screening)

Temperature-programmed testing for **exothermic decomposition in an air current** (after Grewer, see [1]) is performed up to 270 °C.

(If a signal is detected during testing in the range up to 50 °C above the limit temperature determined for vacuum drying, this must be stated in the test report if an assessment was ordered only for vacuum drying. Attention must be drawn to the hazard of air entering, and the need for additional testing for air drying must be stated.)

A temperature 110 °C below the decomposition temperature determined by testing for exothermic decomposition in air is proposed as the highest allowable temperature for the heating medium.

If this value is not acceptable for operational purposes, more detailed testing must be performed.

##### Detailed testing

By means of a **hot-storage test in a 400 ml wire basket** (starting, for example, 50 °C below this temperature, but not higher than 180 °C), at decreasing temperature intervals of 10 °C the basket limit temperature is now determined at which the temperature rise (in the case of an exothermic process) is less than 5 °C (and no endothermic decomposition is observed). The measuring period at the limit temperature should last at least 24 hours (and if necessary, until the peak temperature is passed).

If the overheating observed in the test at 10 °C above the basket limit temperature is more than 50 °C, the maximum temperature for the heating medium should not exceed a value of 10 °C below the basket limit temperature; otherwise, it should not exceed the basket limit temperature.

For certain products, experience has shown that the decomposition test under low-oxygen conditions in the Dewar container yields a lower limit temperature than the basket test. For this reason, the test for heat accu-

mulation in the Dewar container described above is performed in addition. The lower of the two temperatures is taken as the highest allowable temperature for the heating medium.

## A2 Convection drying

The temperature limits that follow are for normal non-caking operation of convection dryers for drying flammable dusts which are protected by inerting according to the state-of the art, and by explosion venting or explosion suppression against possible explosions of dust or hybrid mixtures.

### A2.1 Spray drying

#### Inflowing air temperature

The temperatures of the inflowing air can be selected by the plant manager, but they must be safeguarded below the ignition temperature of the fluidized dry dust or the flammable liquid contained in it.

#### Outflowing air temperature

##### Simplified method (screening)

Temperature-programmed testing for exothermic decomposition in the air current (after Grewer, see [1]) is performed. A temperature 60 °C below the temperature determined in this manner for the first exothermy is proposed for the maximum temperature of the air outflowing air. However, this temperature should not exceed 150 °C (from experience).

The test report must draw attention to the fact that this value was determined by a simplified testing method, and that more detailed tests may yield a higher temperature for the outgoing air.

If the value obtained is not acceptable for operational purposes, a more detailed test is performed.

##### Detailed testing

By means of hot-storage test in a 400 ml wire basket at decreasing temperature intervals of 10 °C, the basket limit temperature is determined at which the temperature rise (in the case of an exothermic process) is less than 5 °C (and no endothermic decomposition is observed). The measuring period at the limit temperature should be at least 24 hours (and if necessary, continue until the peak temperature is passed).

If the overheating observed in the test at 10 °C above

the basket limit temperature is more than 50 °C, the maximum temperature for the outflowing air should not exceed a value of 10 °C below the basket limit temperature, and otherwise should not exceed the basket limit temperature.

### A2.2 Fluid-bed drying

#### Simplified method (screening)

Temperature-programmed testing for exothermic decomposition in the air current (after Grewer, see [1]) is performed. A temperature 110 °C below the temperature determined in this manner for the first exothermy is proposed for the maximum temperature of the inflowing air.

The test report draws attention to the fact that this value was determined with a simplified testing method and more detailed tests may yield a higher temperature for the inflowing air.

If the value obtained is not acceptable for operational purposes, a more detailed test must be performed.

#### Detailed testing

By means of hot-storage test in a 400 ml wire basket at decreasing temperature intervals of 10 °C, the basket limit temperature is determined at which the temperature rise (in the case of an exothermic process) is less than 5 °C (and no endothermic decomposition is observed). The measuring period at the limit temperature should be at least 24 hours (and if necessary, continue until the peak temperature is passed).

If the overheating observed in the test at 10 °C above the basket limit temperature is more than 50 °C, the maximum temperature for the inflowing air should not exceed a value of 10 °C below the basket limit temperature, and otherwise should not exceed the basket limit temperature.

## Extracts from the 1985 edition of ESCIS Booklet 6, “Drying Solids in Drying Ovens in Air or Vacuum, or in Paddle Dryers”:

- Safety assessment of the product to be dried
- Protective measures on dryers
- Tests for assessment of the product to be dried
- Safety classes for drying (SCD)

### 3. Assessing the substance to be dried<sup>12</sup>

#### 3.1. Basic principles

For each product to be dried, the protective measures to be taken must be determined before the respective drying operations are started in the production environment. The criteria for the protective measures to be taken are a number of tests in relation to specific properties which are applied to the products. The tests of specific properties are divided into two categories: basic tests and supplementary tests. The test results are summarized in a test report.

#### 4. Assessment of the product to be dried and assignment to a safety class

The criteria for determining whether, and under what conditions, a product may be dried in a drying chamber in air or vacuum, or in a paddle dryer, are the maximum allowable temperature of the heating medium and the safety class of the product for drying purposes.

#### 4.1 Temperature of the heating medium

Based on the test results, the procedure described in the Annex can be used to determine the maximum allowable temperature of the heating medium for drying in air or under low-oxygen conditions.

If the request for testing does not specify a desired temperature for the heating medium, a relatively low maximum allowable temperature can be specified after

a simplified testing procedure and correspondingly noted in the test report. If a higher temperature for the heating medium is required by the production department because it would, for example, increase the efficiency of the drying process, they should then request a more detailed assessment (which also involves greater effort). See also the preliminary remark in the appendix.

#### 4.2 Assignment of products to safety classes for drying (SCD)

Depending on the results of the basic tests, products are assigned to one of three safety classes for drying (see Table 1). The three classes are:

SCD 0: A product is assigned to SCD 0 if all five criteria for this class shown in Table 1 are fulfilled.

SCD 1: A product is assigned to SCD 1 if at least one of the criteria of SCD 0 is exceeded but non of the criteria of SCD 2 is fulfilled.

SCD 2: A product is assigned to SCD 2 if it fulfils at least one of the three criteria for this class.

Criterion \ Safety Class	SCD 0	SCD 1	SCD 2
<b>Burning test at 100 °C in air (burning number)</b>	Max. 3	4 or 5	6
<b>Self propagating decomposition test at 100 °C (see footnote<sup>13</sup>)</b>	Negative	Negative	Positive
<b>Impact sensitivity (for paddle dryers with beaters only)</b>	Negative	Negative	Positive
<b>Test for volume of decomposition gases up to 220 °C [1 kg<sup>-1</sup>]</b>	Max. 10	> 10	
<b>Content of flammable solvent before drying (% by weight)</b>	Max. 0.1%	> 0.1%	

Table 1: Assignment of products to protection classes

<sup>12</sup> For companies which only carry out such tests relatively infrequently and therefore do not wish to acquire the necessary equipment, Suva, Section Chemistry, CH-6002 Lucerne, is willing on request to provide contacts with companies which perform such tests on a fee-paying basis.

<sup>13</sup> The expression “Self propagating decomposition”, formerly known as “deflagration”, is interpreted in various different ways. Within the scope of this booklet, the following definition applies: Self propagating decomposition is decomposition initiated locally by an external ignition source which in contrast to combustion propagates autonomously even in the absence of atmospheric oxygen, the rate of propagation never exceeding the speed of sound.

## 5. Drying conditions and protective measures

### 5.1 Significance of the drying-medium temperature

Irrespective of the safety class to which the product is assigned, care must be taken to ensure that the maximum allowable temperature for the heating medium determined in the tests is never exceeded. Wherever possible, this must be safeguarded by technical measures. For critical products it is advisable to monitor and record the temperature of the heating medium as a function of time.

### 5.2 General measures

After evacuation and before heating of vacuum dryers, as a precaution against possible overpressure resulting from decomposition of the product, it is advisable to unlock the doors or other openings of the dryer, and possibly to seal the workroom. This is unnecessary if the apparatus is equipped with constructional protective measures (e.g. a sufficiently large bursting disk), or if a paddle dryer is being used for solvent recovery (distillation).

If dried product is filled into metal containers, the containers must be grounded.

It is self-evident that adherence to the principles of Good Manufacturing Practice (GMP) also contributes to increasing the safety of drying operations:

- Workplace regulations and product-specific information must be present at the workplace.
- The dryer must be regularly and sufficiently cleaned; paddle dryers must be specially checked for noticeable encrustation. It is the responsibility of the plant manager to specify the frequency of the checks, the criteria for cleanliness, and the cleaning methods to be used. In the case of thermally sensitive products, consultation with the testing department regarding inspection and cleaning intervals may be advisable with respect to the long-term thermal exposure of product residues.

### 5.3 SCD 0 products

Products in SCD 0 have only a low hazard potential. Adherence to the general measures stated in 5.2 and the maximum allowable temperature determined for the heating medium are sufficient to ensure safe drying.

### 5.4 SCD 1 products: additional measures

Vacuum dryers shall not be heated before they have been evacuated. In exceptional cases, for example with strongly foaming (but solvent-free) products, pre-heating of a drying chamber or paddle dryer may be per-

formed with the door/cover slightly open, but only up to a maximum of 80 °C (heating-medium temperature) and only if the combustion number of the product at 100 °C is not higher than 3.

At the end of the drying process, the vacuum must always be relieved with inert gas. Vacuum dryers may only be aired and unloaded when the temperature of the dried product is below the allowable temperature for air drying. In exceptional cases where this temperature is not known, it is advisable to cool the dried product to below 40 °C before discharging it.<sup>14</sup>

Breaking of the vacuum – for example as a result of a power outage or a fault in the vacuum generating equipment – must be indicated by an alarm. The dryer must be immediately flooded with inert gas (manually or automatically) and cooled.<sup>15</sup>

When drying products which contain flammable solvent (more than 0.1 % by weight), or which generate decomposition gases, the regulations regarding separation into hazardous zones must be observed (SEV 3307, 1983, and Suva, Form 2153).

Air dryers for products containing solvent (more than 0.1 % by weight) should preferably be operated with fresh air, not recirculated air. If operation with recirculated air is necessary for economic reasons, the quantity of air must be so regulated as to ensure with certainty that the concentration of flammable gases or vapors at the outlet of the dryer does not exceed 50 % of the lower explosion limit (cf. Suva, Form 1733).

Dryers for products containing solvents (more than 0.1 % by weight) shall only be charged when cold and only be heated after they have been closed and evacuated.

The open charging of paddle dryers with solvent-wet product (more than 0.1 % by weight) at a temperature which is not at least 5 °C below the flashpoint of the solvent is not recommended because of the ignition hazard from static electricity.

<sup>14</sup> The temperature of the dried product can normally only be measured when it is discharged. The time required for cooling depends among other things on the size of the apparatus, type and quantity of the dried product, residual moisture, and temperature of the heating medium. The cooling time can be determined in the plant by trials. It should then be noted in the process documentation. Typical empirical values for paddle dryers in the range 4 to 6 m<sup>3</sup> are:

- After 30 minutes cooling time, the product temperature is approximately ½ to ⅔ of the temperature in °C used for the heating medium.
- Cooling to 40–70 °C takes about 3 hours.
- Cooling to 25–30 °C can take more than 4 hours.

<sup>15</sup> If, for example, a power outage makes further effective cooling impossible, when changing over to cooling at least the heating is switched off.

## **5.5 SCD 2 products**

Products in SCD 2 are potentially very hazardous because they

- have highly hazardous combustion properties
- have a tendency to self propagating decomposition (see footnote 2 p. 24)
- are impact-sensitive.

The test and assessment procedure described in the Annex is inadequate to determine safe drying conditions for these products. Further tests and a special evaluation of the test results in the light of the envisaged drying conditions are necessary to decide whether, and under what conditions, drying can be allowed.

## Expert Commission for Safety in the Swiss Chemical Industry (ESCIS)

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- Video Part 3 Constructional explosion protection
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- Video Parte 3 Protección constructiva contra explosiones
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