Report

Inerting Methods and Measures for the Avoidance of Ignitable Substance-Air Mixtures in Chemical Production Equipment and Plants

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Inerting

Methods and Measures for the Avoidance of Ignitable Substance-Air Mixtures in Chemical Production Equipment and Plants

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Translation of 2nd, revised German edition
The assurance of safety and environmental protection in a chemical processing plant is based to a large extent on the identification and determination of hazards, the implementation of appropriate measures to avoid and lower the hazard potentials, as well as estimation and assessment of the residual risks. Systematic risk analysis has proved to be an important tool to tackle these tasks and problems. Its basic methodology is described in ESCIS booklet No. 4 “Introduction to risk analysis”.

Risk analyses must embrace all possible critical areas. These can be summarized as follows:

- Chemicals
- Chemical reactions, physical processes
- Various types of energy
- Plant items, instruments, technical aids
- Personnel, organization

The present ESCIS booklet discusses the use of inerting to lower the risks associated with the handling of substances which can form ignitable mixtures with air. Inerting is suitable in numerous cases to exclude the formation of explosive mixtures from the outset.

The booklet describes a selection of proven inerting methods with application examples corresponding to the latest technological developments. Ecological aspects are not dealt with in detail, but plant design must take into account the safe and environmentally acceptable disposal of inert gas mixed with organic vapors.

In comparison with the first edition, this second edition offers not only editorial changes, but also a complete revision of the fundamental principles. Illustrative application examples which have been incorporated in industrial practice since the first edition are also provided.

Basle, April 1994

Expert Commission for Safety in the Swiss Chemical Industry (ESCIS)
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Methods and measures for the avoidance of ignitable substance-air mixtures in chemical production equipment and plants

1. Introduction

In the handling of flammable gases and liquids and combustible dusts, there is a danger that explosible mixtures can form and be ignited. This risk makes special protective measures necessary. While inerting of the gas space, in other words displacement of the atmospheric oxygen by a nonreactive gas, is a reliable preventive explosion protection measure, it requires adherence to certain basic rules and the assurance that the required degree of inerting actually obtains. This ESCIS booklet is intended as an aid in the selection of possible inerting alternatives and indicates the conditions which have to be met in each case. The procedures described have been tested in operational practice.

2. Fundamentals

2.1 Ignition and explosion behavior of flammable gases, vapors and dusts in air

Flammable gases and combustible dusts as well as the vapors and aerosols of flammable liquids form ignitable and hence explosible mixtures with air within certain concentration ranges. These concentration ranges are specified by the lower and upper explosibility limits. With pure liquids, the equilibrium concentration of their vapors in air is described by their vapor pressure curve. Here, specification of the temperature can thus be used to describe the vapor concentration. This is shown in Figure 1 using ethanol as an example: At the lower explosion point (8 °C), the vapor concentration (in thermodynamic equilibrium) corresponds exactly to the lower explosibility limit. In practice, however, it has become customary to use the flash point (here 11 °C) to describe the explosion hazard due to the

![Figure 1: Explosion characteristics of ethanol](image-url)
vapors of flammable liquids. As this is determined by a dynamic procedure (by heating the liquid in a closed vessel), it is always above the lower explosion point. The flash point can not be used to draw conclusions regarding the ignitability of aerosols in the spraying of liquids.

To describe the development of an explosion in closed equipment, both the explosion overpressure (\(p\)) and the rate of pressure rise (\(\frac{dp}{dt}\)) are used as a measure of the severity of the explosion. As both characteristics depend on the composition of the mixture, their maximum values \(p_{\text{max}}, \frac{dp}{dt}_{\text{max}}\), which have to be determined experimentally, are always used as the basis for safety considerations. Whereas the maximum explosion overpressure is practically independent of the volume of the explosion vessel, the explosion severity depends on the vessel volume \(V\) in accordance with the “cube-root law” (sometimes called the cubic law):

\[
(p_{\text{max}}) \cdot V^{\frac{1}{3}} = \text{constant} = K_{\text{max}}
\]

This maximum value for the material-specific constant "\(K_{\text{max}}\)" is often characterized by the index "\(G\)" for flammable gases and "\(St\)" (St is an abbreviation of the German Staub, which means dust) for combustible dusts. In practice, it has become customary to classify combustible dusts into so-called dust explosion classes based on their \(K_{\text{max}}\) values and to design constructional explosion protection measures according to these dust explosion classes\(^2\). These dust explosion classes only predict the severity of a possible explosion, they say nothing about its probability of occurrence or possible effects!

When explosive dusts and flammable gases or vapors are present at the same time, the resulting hybrid mixtures can be exploisible even when the concentrations of the individual components are below their respective lower explosibility limits. The minimum ignition energy (see below) of the hybrid mixtures is also lowered to that of the gas or vapor. Moreover, the explosion severity of the hybrid mixtures is generally greater than that of the individual components. Typical examples of hybrid mixtures are combustible powders with residual contents of flammable solvents in granulators, fluid bed and spray dryers, centrifuges, etc.

To describe the ignition behavior of exploisible mixtures, various parameters are used which have been matched to the ignition characteristics of different types of ignition sources. The two most important are the minimum ignition energy and the minimum ignition temperature. The minimum ignition temperature represents the lowest temperature of a hot surface at which the exploisible mixture ignites. The minimum ignition temperatures of flammable liquids lie between 102 °C (carbon disulfide) and 630 °C (aniline), those of dust clouds are, for example, 235 °C for sulphur and 530 °C for zinc, whereas those of methane and hydrogen are 595 °C and 560 °C, respectively. The minimum ignition energy is the lowest energy of an electrical discharge spark which is just capable of igniting the most readily ignitable concentration of the exploisible mixture under standard conditions. The minimum ignition energies of common gases and vapors are around 0.2 mJ; hydrogen, acetylene and carbon disulfide can be ignited with just 0.01 mJ. The minimum ignition energies of combustible dusts are generally in the range 1 mJ to 100 J. Combustible dusts designated as readily ignitable have a minimum ignition energy less than 10 mJ. Ignition sources found in practice – such as electrostatic discharges, mechanically generated sparks and hot surfaces – are easily capable of igniting these exploisible mixtures.

2.2 Measures of explosion protection
For an explosion to occur, all three elements of the hazard triangle shown in Figure 2 must be present.

Preventive explosion protection is designed to exclude at least one of the sides of the hazard triangle with certainty. Depending on the operational conditions, however, this is not always possible with sufficient reliability. For example, in the processing of combustible dusts, the avoidance of ignition sources is employed mainly as an additional, secondary measure as the absolute exclusion of electrostatic discharges is not always possible.

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\(^1\) see ESCIS brochure 1 "Sicherheitstests für Chemikalien"

\(^2\) see ESCIS brochure 5 "Milling of Combustible Solids"
The avoidance of explosible concentrations, i.e. safe working below the lower explosibility limit usually requires continuous monitoring of the combustible product concentration in order to initiate emergency measures should the specified limit values be reached. In the case of flammable liquids, the difference between the flash point and the lower explosion point should again be noted (see Figure 1): The rule usually employed today “working temperature at least 5 °C below the flash point” must be subjected to critical scrutiny, depending on the slope of the vapor pressure curve. With combustible dusts, the hazards posed by secondary explosions due to the airborne dispersion of dust layers should be noted. Inerting to lower the oxygen content is treated in detail below.

Measures of constructional explosion protection are used in cases where those of preventive explosion protection are insufficient by themselves. As the occurrence of explosions is anticipated, plant or plant items so protected must have a certain explosion resistance (see Figure 3). They are designated as explosion pressure resistant when the overpressure allowed by the construction corresponds to the maximum possible explosion overpressure. Explosion pressure shock resistant vessels withstand the expected maximum explosion overpressure without rupturing, but may suffer permanent deformation in the event of an explosion. (Guidelines also exist today for the construction and calculation of “pressure shock resistant” vessels.) Whether the pressure used in the design is the full or a reduced explosion overpressure depends on whether one of the following explosion protection measures has also been employed.

In explosion (relief) venting (see Figure 4), the attainment of a preselected response pressure releases openings which vent the remaining explosion development to the open air and thus lower the resulting explosion overpressure to a value corresponding to the vessel strength.

![Figure 3: Explosion-resistant construction](image)

![Figure 4: Explosion (relief) venting](image)

Explosion suppression (see Figure 5) operates according to the principle of an “automatic fire extinguisher”: Pressure sensors detect an incipient explosion, suppressant is injected into the vessel under high pressure and extinguishes the flame front. The vessel strength is adequate for the remaining reduced explosion overpressure. The explosion protection of a complete production plant normally comprises a combination of several explosion protection measures. In other words, wherever possible the preventive protection measure avoidance of ignition sources is used, where necessary, however, constructional explosion protection measures must be planned. Isolation methods must then be employed to ensure that “controlled” explosions can not be propagated as undesired ignition sources into areas which are protected by the “avoidance of ignition sources”.

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**Figure 3: Explosion-resistant construction**

- pressure resistant
- pressure shock resistant

**Figure 4: Explosion (relief) venting**

- release of specified openings at $p_{\text{stat}}$
- flame escape in nonhazardous direction to open air $\rightarrow$ vent ducts
- $p_{\text{red,max}} \leq$ vessel strength

**Explosion suppression** (see Figure 5) operates according to the principle of an “automatic fire extinguisher”: Pressure sensors detect an incipient explosion, suppressant is injected into the vessel under high pressure and extinguishes the flame front. The vessel strength is adequate for the remaining reduced explosion overpressure. The explosion protection of a complete production plant normally comprises a combination of several explosion protection measures. In other words, wherever possible the preventive protection measure avoidance of ignition sources is used, where necessary, however, constructional explosion protection measures must be planned. Isolation methods must then be employed to ensure that “controlled” explosions can not be propagated as undesired ignition sources into areas which are protected by the “avoidance of ignition sources”.

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2.3 Mode of action and types of inerting

The principle of inerting is based on lowering the oxygen content of the combustible atmosphere to a level where fires or explosions are no longer possible. There are basically two methods to achieve this:

Partial inerting involves lowering the oxygen concentration in the mixture so that it is no longer exploisable. The decisive value here is the limiting oxygen concentration: the highest oxygen concentration at which flame propagation (explosion) is just no longer possible. This value depends primarily on the flammable substance, the inert gas and also on temperature and pressure. For industrial practice, the decisive parameter is the maximum permissible oxygen concentration, which is obtained by subtracting a safety margin (in general 2 vol% oxygen) from the experimentally determined limiting oxygen concentration. It must be ensured that the maximum permissible oxygen concentration is not exceeded. This can occasionally be checked by calculation, but usually measurements (continuous or at intervals) are employed. Warning: If air enters or escapes from the system, the mixture may become exploisable again!

In total inerting, on the other hand, the ratio of the inert gas content to that of the flammable substance is so high that even with the admixture of any amount of air (or escape from the system) the mixture is no longer exploisable. Total inerting is used in practice only in connection with flammable gases and vapors, it must be accorded particular attention with equipment from which vapors escape sporadically or form part of the exhaust gas. Owing to the high ratio of inert gas to flammable substance, these plant items are often operated under high pressure.

2.4 Gases for inerting, residual oxygen content

In the chemical industry, nitrogen (with max. 0.5 vol% oxygen) is used as inert gas in numerous cases. But other gases, for example carbon dioxide, flue gases or water vapor can also be employed if it can be established that they actually exhibit inert behavior under the given operating conditions. Noble gases are occasionally used for the inerting of small-scale apparatus. The effectiveness of a gas as an inert material (the maximum permissible oxygen concentration) depends mainly on its specific heat capacity.

However, the use of inert gases is also associated with certain hazards: most inert gases are odorless and thus give no warning of their presence. If the equipment leaks and nitrogen or carbon dioxide is used as inert gas, this can lead to a danger of suffocation or poisoning in the vicinity of the installation. Carbon dioxide from pressure vessels or low-temperature tanks should not be used without additional safety measures as on escape crystals (carbon dioxide snow) can be formed which can be electrostatically charged to such a high level that incendive discharges are possible. In inerting with water vapor, due consideration must be given to condensation.

Assuming powerful, operational ignition energies such as mechanically generated sparks or hot surfaces, according to our present knowledge the following holds when nitrogen is used as inert gas:

- Most gases and vapors (cf. Table 1, Appendix) are no longer ignitable at room temperature and standard pressure when the oxygen content in the inerted mixture is less than 10 vol% (with hydrogen and carbon monoxide this critical parameter is 5 vol%). With increasing temperature, this limit decreases, the decrease being of the order 0.5 to 1 vol% for every 100 °C rise in the temperature. If no data specific to the material are available, an oxygen content of maximum 8 vol% (with hydrogen and carbon monoxide maximum 4 vol%) must be maintained.

- For organic dusts, with an oxygen content of ≤ 12 vol% in nitrogen there is generally no longer a danger of ignition. Exceptions exist, however, and include paraformaldehyde, 2-naphthol and biphenyl for which the permissible residual oxygen content is only 4 vol% (cf. Table 2, Appendix). With increasing temperature, the limiting oxygen concentration sinks by around 1.6 vol% per 100 °C rise in temperature.

- For aerosols and metal dusts, the permissible residual oxygen content must be determined for each specific case.
For hybrid mixtures, the limiting oxygen content is determined by the combustible material with the lowest limit value.

In all cases where doubt exists, the maximum permissible oxygen concentration must be determined experimentally.

An important aspect of each type of inerting is its monitoring with suitable measuring instruments. The sensitivity of oxygen analyzers may cause problems as they frequently need special preparation of the sample gas and always require regular maintenance and calibration programs. Alarm thresholds with a sufficient margin below the maximum permissible oxygen concentration should be specified which, if attained, must initiate protective measures or emergency functions.

2.5 Limits of applicability
Inerting is ineffective with products capable of deflagration, which can burn or decompose in the absence of atmospheric oxygen. It is also ineffective against thermal decompositions.

31 see ESCIS brochure 1 “Sicherheitstests für Chemikalien”
3. Methods and measures

The first step in inerting involves lowering the oxygen concentration in the area to be inerted to below the limiting oxygen concentration. It is then necessary to prevent the ingress of oxygen into the inerted area during operation.

3.1 Pressure change methods

3.1.1 Vacuum method

This method is used in numerous plants and involves evacuating the equipment to be inerted to a specified pressure value and then releasing this vacuum with an inert gas (e.g. nitrogen). It must be ensured that the vacuum pumps used for the evacuation can not act as an ignition source (explosion protection, mechanical friction). If need be, special safety measures must be installed (e.g. flame barriers). Further, the capability of the equipment to withstand vacuum must be taken into account.

3.1.2 Overpressure methods

The area being inerted is pressurized with inert gas to a certain overpressure and this is then released. Clearly, it must be ensured that the plant item is not stressed above its admissible operating pressure.

3.1.3 Pressure change and inert gas consumption

For both pressure change methods, Figure 6 shows the theoretical number of pressure changes \( n \) for a specified pressure (vacuum and overpressure method) needed to achieve a specific residual oxygen concentration. Further, the diagram can also be used to determine the inert gas consumption \( f \) as a multiple of the equipment volume for one pressure change (intersection point with vacuum or overpressure curve).

When this is done, it is clear that more inert gas, in some cases appreciably more, is required for the overpressure method. For this reason and also as a consequence of clean air regulations, the overpressure method is used rather seldom in actual practice.

The following holds:

\[
y_s = \left( \frac{P_1}{P_2} \right)^n \cdot (y_0 - a) + a
\]

where:

- \( y_s \) = O\(_2\) concentration after \( n \) pressure changes (vol%)
- \( y_0 \) = initial O\(_2\) concentration (vol%)
- \( a \) = O\(_2\) concentration in inert gas (vol%)
- \( \frac{P_1}{P_2} \) = pressure ratio, less than 1
- \( n \) = number of pressure changes

Examples:
- Vacuum region
  - Evacuating to \( P_1 = 0.5 \) bar and releasing to \( P_2 = 1 \) bar with nitrogen results in a residual oxygen content \( y_s \) of 10.8 vol% and a nitrogen requirement of \( \frac{1}{2} \) of the vessel volume.
- Pressure region
  - Two pressurizations to \( P_2 = 2 \) bar and two releases to \( P_1 = 1 \) bar produce a residual oxygen content \( y_s = 5.6 \) vol% and a nitrogen requirement of \( 2 \times 1 = 2 \) times the vessel volume.

3.2 Purging

With equipment which can be used for neither vacuum nor overpressure operation, inerting by means of purging can be employed. Here, optimum arrangement of the inert gas inflow and outflow and selection of a sufficiently large inflow rate must be used to attain the best possible mixing effect in the equipment.

Figure 7 shows the theoretical inert gas quantity as a multiple of the vessel volume needed to achieve a specified residual oxygen content with ideal mixing. As ideal mixing is never achieved in practice, the inert gas quantity actually needed must be checked by periodic oxygen measurements.
3.3 Maintenance of inerting during operation

The following factors influence the degree of inerting of a plant item during operation and thus warrant special attention in operations in inerted equipment:

- Convection
- Density difference air – inert gas
- Diffusion
- Equipment openings (e.g. manhole or charging opening)
- Leaks
- Working pressure (vacuum)
- Temperature
- Operating errors

Example:

In an agitated vessel (2500 liters), after two evacuations and two releases with industrial nitrogen with closed manhole and stationary agitator an oxygen concentration of 3.5 vol% was measured at the bottom at room temperature. When the manhole was opened, the oxygen concentration rose to 9 vol% after 4 minutes. After 10 minutes this increased to 12 vol% and to as high as 16 vol% after 20 minutes.

This example clearly shows that to maintain the inerting in equipment during the operations involved in the process (charging, discharging, sampling, etc.), suitable measures must be taken, e.g.

- Inert gas blanketing with pressure control and possible oxygen monitoring at intervals
- Inert gas purging with periodic or permanent oxygen monitoring
- Gas return lines to supply vessels
- Feed lock systems

3.3.1 Charging and discharging of liquids into and from inerted equipment

Problems:

- In the charging of liquids using drum pumps, there is a danger that air is entrained when the drum is almost empty.
- In the addition of liquids from noninerted, elevated supply vessels, air can enter the system when such vessels are completely empty.
- When vessels are discharged, air can enter the system.

Possible measures:

- Inerting of supply vessel
- Incomplete emptying of supply vessels
- Gas return lines between inerted equipment and inerted supply vessel
- Assurance of inerting by make-up with nitrogen.

3.3.2 Charging of bulk material into inerted equipment

Problem:

- In the charging of solids through the open manhole, air enters the inerted equipment.

Possible measures:

- Charging through a feed lock
  The bulk material is charged by means of a feed lock (rotary air lock, drop pipe with interlocking shutoff devices) which is tightly connected to the inerted equipment. As a result of leaks in the feed device (especially if the equipment is under vacuum) and through the product feed (air in bulk material or in the feeder), air can still enter the inerted equipment. Usually, this must be diluted by addition of inert gas to ensure that no inadmissable oxygen concentrations can appear despite the ingress of air.
  Simple estimations can be used to determine the ingress of air and hence also the amount of inert gas required for purging. This must also be monitored by periodic or continuous measurements of the oxygen concentration.

- Charging from inerted container
  The bulk material is held in an inerted container tightly coupled with the equipment and falls under its own weight into the inerted equipment.
  With poorly flowing bulk material, fluidization devices operated by inert gas or mechanical vibrators can be used to facilitate the flow.
  However, if pokers have to be used, the same rules as for charging through an open manhole apply.
  When noninerted containers are used, the amount of air entrained must be estimated and diluted by inert gas purging if necessary.

- Charging by means of pneumatic conveying
  In frequently occurring charging operations of relatively large amounts of bulk solids, it can be advisable to perform these under closed conditions using pneumatic conveying. In such cases, the inert gas is used as the conveying medium, preferably in a closed circuit.

- Closed charging
  The solid is added to an intermediate container attached to the equipment. After its inerting, the bulk solid can be added to the vessel in a closed system.

- Charging through open manhole
  Charging through the open manhole continues to be used, particularly with older installations or when small amounts of bulk solid have to be added. To keep the ingress of air during these charging operations through the open manhole to a minimum, in practice various time-tested methods are used such as ventilated feed funnels with simultaneous inert gas purging, selection of an inert gas that is clearly heavier than air (e.g. carbon dioxide) or limiting the opening time of the manhole. As air can still enter the apparatus with all these methods, their effectiveness must be checked by periodic or continuous oxygen measurements.
3.3.3 Sampling
Sampling can lead to problems when inerting is employed, primarily with batch processes. If samples are taken through the open manhole, the same rules as for charging through the open manhole apply. In practice, however, many sampling systems which allow a sample to be taken from the inerted equipment through a closed system have proved their worth. These range from the simple Teflon tubing, which, for example, can be inserted in the equipment via a tapered plug cock or a ball valve to take a sample using a suction bottle up to the sampling device permanently installed in the apparatus (e.g. in the baffle).
4. Illustrative examples

Preliminary remarks
It is not possible to give directions on how to perform inerting successfully for the numerous processing operations in the wide range of different plant types. The application examples described in what follows have proved their worth in practice and are intended to stimulate the search for solutions to comparable problems. The highly simplified schemes include only those installation parts important for inerting. They represent the basic requirements for the inerting and must be adapted to the individual characteristics of the respective plant items.

4.1 Reaction vessels
The charging of solids into a reactor in which ignitable gases/vapors can occur, the following inerting measures must be implemented:
- inerting of the entire installation before charging (preinerting),
- continuous supply of inert gas during charging.

Examples:
If the plant item is equipped with suitable installations for vacuum inerting as described in section 3.1.1, in preinerting this method should always be given preference over purging (section 3.2) and overpressure methods (section 3.1.2).
Quantities of air entrained during the charging of solids must always – for permanent maintenance of inert conditions – be diluted by a continuous supply of inert gas to the reactor.

In the individual examples, the procedure is as follows:

4.1.1 Charging of bulk material in counterflow
After the vessel (Figure 8) has been preinerted (V7 and V2), a constant stream of inert gas (by means of F1 and V3a or orifice plate) is supplied via valve V3 in a manner such that the incoming bulk material flows through the outgoing inert gas stream (in counterflow). If the gas flow is set too high, dust can be discharged, a situation which must clearly be avoided. On the other hand, the gas flow must be high enough to ensure that inerting is always maintained.
The drop pipe of the feed device should generally be immersed in the vessel as far as possible. This ensures that the mixing zone is displaced further into the inerted part of the vessel. It also results in an improved product distribution in the pipe.
To ensure that inerting is maintained, the vessel should not be operated under vacuum. An exception to this rule is toxic gases, which must not be removed into the room. If need be, an O2 monitoring system must be planned here.
The required pressure level in the system can be set using valve V4 (suction effect of exhaust pipe) and pressure relief valve V5. The suction effect in the feed funnel is set using valve V6.

4.1.2 Bulk material charging using a mobile supply vessel
Before connection of the supply vessel and charging, the reactor (Figure 9) is inerted with closed charging opening F1. After the supply vessel has been connected to the charging device of the reactor, flaps F1 and F2 are opened.
If the bulk material has unfavorable flow properties, the bulk solids container must be opened and discharged by "prodding".
The supply vessel is connected here to the exhaust system by opening valve V7 so that good occupational hygiene conditions are maintained. It must be ensured that air does not enter the system in an uncontrolled manner.
This feed device is being replaced in an increasing number of cases by the devices described below.

4.1.3 Charging of bulk material via feed locks
A double flap system DF as shown in Figure 10 or the rotary vane feeder R shown in Figure 11 can be used as the feed device. Although the amount of entrained air can be reduced with these systems, it must be determined for every application case.
The feed systems shown in Figure 11 (screw conveyor and rotary vane feeder) must also be inerted (V4).
An increase in the oxygen concentration in the reactor during charging is prevented by controlled, simultaneous supply of inert gas (FI and V3a or orifice plate) by means of valve V3.

4.1.4 Closed charging of bulk material
The closed funnel is inerted, filled with bulk material and connected via flap F1 to the inerted reactor (Figure 12). The entire system is blanketed by a small inert gas pressure (V1 and V2).

Preferably, the upper part of reactor and funnel is provided with a gas return line GL so that the bulk material is not adversely affected by the solvent vapors backflowing into the funnel (e.g. caked deposits).
In principle, this arrangement is the most favorable. It has the following advantages:
- inert gas addition necessary at only one location,
- small amounts of exhaust,
- high level of safety.

4.2 Pressure filter with agitator
As shown in the illustrative example in Figure 13, the following operations are performed with agitated pressure filters:

- Inerting
  by evacuation with opened valve V1 and release with inert gas using valve V4.
  Before product discharge, the product truck is inerted by purging with inert gas using valves V6 and V7. The time of inerting and the required amount of inert gas are determined by means of periodic oxygen measurements.

- Loading product
  with opened pressure control valve V2 and exhaust valve V3.

- Filtering
  through the addition of inert gas (overpressure) by opening valves V5 and V6. The filtration operation is monitored by the flow indicator (FI). The measurement is primarily used to determine the end of filtration, which is characterized by a sudden increase in the volumetric rate of inert gas flow. This leads to an appreciable reduction in the inert gas consumption and the emissions.
4.3 Top discharge centrifuge
As Figure 14 shows, very simple inerting of a top discharge centrifuge can be implemented as follows:

- **Before centrifuging (purging phase)** a constant inert gas stream (FI) specified in the purging instructions is passed through the centrifuge for a given time by means of control valve V2. The pressure control valve V5 is used to maintain a slight overpressure of 10 to 20 mbar in the centrifuge. This pressure must always be less than the hydrostatic pressure of the liquid column in siphon S1 to ensure that no gas can escape into the filtrate vessel. In the purging operation, the gas escapes via pressure control V5 into the exhaust line. When the required purging time has elapsed, centrifuging can be started.

- **During the centrifuging operation** inert gas is introduced at constant pressure using V2 and the flow indicator FI, particularly to compensate the volume of the separated filtrate. Further, during the entire operating time it must be ensured that no solvent vapors reach the bearings. This can be achieved by blanketeting with a small inert gas overpressure (compared with the pressure inside the centrifuge). If the lower limit of this pressure is violated, the centrifuge switches itself off automatically. However, a more reliable method involves inert gas “breathing” using permanent pressure or oxygen measurement in the centrifuge.

- **For product discharge** the centrifuge is opened with the inert gas supply interrupted. This stops inerting. Extreme care is needed, particularly with products moist with solvent which are not electrostatically conducting (resistivity > 10^8 ohm - m, cf. footnote46), as electrostatic discharges (ignition sources) can not be excluded.

With top discharge centrifuges, the inerting method described in section 4.4 can also be employed.

46 see ESCIS brochure 2 “Static Electricity”
4.4 Bottom discharge centrifuge
The bottom discharge centrifuge shown in Figure 15 is operated as follows:

- During the purging phase, the top part of the centrifuge, the product drop pipe and the product truck are filled with inert gas. The product truck can also be inerted before the product discharge. The purging time of the installation is checked by means of periodic oxygen measurements and programmed in the control unit.
- Before centrifuge startup, an overpressure of 20 to 20 mbar is maintained inside the centrifuge through a gassing control system and the pressure control valve V2.

This is also monitored via a control unit. To prevent solvent vapors penetrating the centrifuge bearings, inert gas is supplied separately to the bearings at a slight overpressure (compared with the pressure inside the centrifuge). The separate inert gas flows are determined by means of periodic oxygen measurements.

The safety fitting V1 is used as a vacuum safeguard. To prevent contamination of the pressure control valve V2 (this could lead to serious leaks or sticking), a mist separator is built in before the valve should the application demand it.
- The product discharge is effected in two stages with:
  - a closed system and continued inert gas supply through “ploughing” of the product into the product truck and
  - an open system and interrupted inert gas supply (for scraping off the remaining product).

The last operation is performed under conditions which are no longer inert – as with the top discharge centrifuge. This is thus also a case where care is needed (e.g. electrostatic discharges as ignition sources could appear).

4.5 Horizontal filter-bag centrifuge
The inerting of horizontal filter-bag centrifuges as illustrated in Figure 16 is similar to that of bottom discharge centrifuges.

- Before the start, the centrifuge is purged with inert gas at 3 positions simultaneously according to the appropriate operating instructions:
  - housing (V4)
  - basket (V2)
  - product discharge pipe (V1)
- During the operation, the pressure in the centrifuge is kept constant (PIC). As long as the system is tight, no inert gas enters the housing (V4).
- Labyrinth and bearings are permanently supplied with inert gas by means of valve V3. The maximum operating pressure in the centrifuge is preset using the pressure control valve V6.

The complete product discharge is effected in a closed system (under inert conditions).

Note
Compared with top and bottom discharge centrifuges, the horizontal filter-bag centrifuge offers the following advantages:
- lower inert gas consumption
- smaller amount of exhaust gases
- good occupational hygiene conditions
- increased safety during the (entire) product discharge operation

4.6 Mills
4.6.1 Multi-purpose mill
Before the installation shown in Figure 17 is put into operation, the following installation parts must be inerted with the fan switched off and flap F1 for circulating gas closed:
- Closed circuit line CL by means of valve V1
Supply vessel by means of valve V2 via inlet nozzles or injectors to attain good mixing

All bearings by means of valve V3

After attainment of the specified oxygen concentration (or if applicable inerting time), valves V1 and V2 are closed. Valve V3 always remains open. The installation is then ready for operation.

Before startup of the milling operation, the inert gas is led into the closed circuit using a blower. Control valve V2 is used to supply the inert gas needed to achieve the protection goal (oxygen concentration ≤ 12 vol% or if applicable the values in section 5) to the installation. If deviations are detected, an alarm message is outputted by the oxygen analyzer.

On attainment of a specified system overpressure, recycle gas is discharged into the exhaust line by means of the PIC pressure regulation system through flap F2.

4.6.2 Cold mill

With the cold milling installation shown in Figure 18, liquid nitrogen is used in addition to inerting of the mill to achieve, among other things,

• an improvement in the milling properties of the product and

• a cooling effect in the grinding chamber.

The product is first precrushed in the crusher and then led to the rotary vane feeder R by means of the feed device. For both the crusher and the feed device, provision must be made to supply the bearings with inert gas.

The rotary vane feeder R is followed by the liquid nitrogen supply. In the mill, the “frozen” product is then milled without additional inert gas supply and the resultant heat development causes the liquid nitrogen to

Figure 17: Multi-purpose mill

Figure 18: Cold milling installation
evaporate and at the same time inert the grinding and silo chambers. All the mill installation needs is a separate inert gas supply to the bearings. As far as the silo is concerned, temperature (TIA) and pressure monitoring (PIA) systems are the sole requirement. As an additional safety measure, an O₂ monitoring system can be built into the exhaust gas stream and an inert gas supply into the exhaust filter. Backflows in the exhaust line due to disturbances (O₂ entrainment) are prevented by rapid action flaps F1 and F2.

4.7 Paddle dryer
A different hazard situation can result during charging of product moist with solvent into (empty) paddle dryers as shown in Figure 19. Evaporation of the solvent leads to the buildup of an explosible mixture near the surface of the bulk material. If the moist product can be electrostatically charged (powder volume resistivity > $10^8$ ohm · m), which is generally the case with organic powders and apolar solvents, the bulked product in the paddle dryer is electrostatically charged. On approach of the rotating, grounded paddles to the bulked product, incendive brush discharges\(^\text{6}\) can appear exactly where the explosible mixture is found. This phenomenon has already led to several accidents in operational practice. As here

\(^{6}\) see ESCIS brochure 2 “Static Electricity”

the ignition source and explosible atmosphere are not in the vicinity of the charging opening but below in the paddle dryer, inerting with CO₂ is advisable in this case. Solid carbon dioxide is added to the empty paddle dryer. Owing to its higher density, the CO₂ vapor formed displaces the air originally present through the charging hole. The residual oxygen content is monitored at the top in the discharge pipe. If this is below the admissible value, adequate inerting is also ensured in the region of the possible ignition source.
5. Appendix

This Appendix contains data on limiting oxygen concentrations as well as details regarding oxygen measurement and a list of references.

5.1 Limiting oxygen concentration

5.1.1 Gases and vapors

The following Table is taken from [5] and shows the limiting oxygen concentration for different gases and vapors at 20 °C and 1 bar, ignition energy = 10 J.

<table>
<thead>
<tr>
<th>Combustible material</th>
<th>Partial inerting</th>
<th>Total inerting</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Limiting oxygen concentration (mole content) in entire mixture of flammable material/air/inert gas on inerting with:</td>
<td>Minimum values of the ratio of the mole fractions of inert gas (N$_2$ or CO$_2$) and air (A) for inerting at any admixture of flammable material</td>
</tr>
<tr>
<td></td>
<td>N$_2$</td>
<td>CO$_2$</td>
</tr>
<tr>
<td>Benzene</td>
<td>11.2</td>
<td>13.9</td>
</tr>
<tr>
<td>Butadiene</td>
<td>10.4</td>
<td>13.0</td>
</tr>
<tr>
<td>Butane</td>
<td>12.1</td>
<td>14.5</td>
</tr>
<tr>
<td>Carbon monoxide</td>
<td>5.0</td>
<td>5.0</td>
</tr>
<tr>
<td>Carburetor fuel</td>
<td>~11.8</td>
<td>~14.5</td>
</tr>
<tr>
<td>Cyclopropane</td>
<td>11.7</td>
<td>13.9</td>
</tr>
<tr>
<td>Ethane</td>
<td>11.0</td>
<td>13.3</td>
</tr>
<tr>
<td>Ethylene</td>
<td>10.0</td>
<td>11.7</td>
</tr>
<tr>
<td>Hexane</td>
<td>12.1</td>
<td>14.5</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>4.8</td>
<td>4.8</td>
</tr>
<tr>
<td>Methane</td>
<td>11.6</td>
<td>14.1</td>
</tr>
<tr>
<td>Pentane</td>
<td>11.6</td>
<td>14.4</td>
</tr>
<tr>
<td>Propane</td>
<td>11.8</td>
<td>14.2</td>
</tr>
<tr>
<td>Propylene</td>
<td>11.5</td>
<td>14.1</td>
</tr>
</tbody>
</table>

Table 1: Limiting oxygen concentration for various gases and vapors
5.1.2 Dusts
The following Table is taken from [5] and shows the limiting oxygen concentration for various dusts at 20 °C and 1 bar, ignition energy = 10 kJ.

<table>
<thead>
<tr>
<th>Dust type</th>
<th>Mean value M (µm)</th>
<th>Limiting oxygen concentration (mole content in the gas phase) [vol%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium</td>
<td>22</td>
<td>5</td>
</tr>
<tr>
<td>Barium stearate</td>
<td>&lt;63</td>
<td>13</td>
</tr>
<tr>
<td>Cadmium laurate</td>
<td>&lt;63</td>
<td>14</td>
</tr>
<tr>
<td>Cadmium stearate</td>
<td>&lt;63</td>
<td>12</td>
</tr>
<tr>
<td>Calcium stearate</td>
<td>&lt;63</td>
<td>12</td>
</tr>
<tr>
<td>Carbon black</td>
<td>13</td>
<td>12</td>
</tr>
<tr>
<td>Cellulose</td>
<td>22</td>
<td>9</td>
</tr>
<tr>
<td>Cornstarch</td>
<td>17</td>
<td>9</td>
</tr>
<tr>
<td>Hard (fat) coal</td>
<td>17</td>
<td>14</td>
</tr>
<tr>
<td>Lignite</td>
<td>63</td>
<td>12</td>
</tr>
<tr>
<td>Methyl cellulose</td>
<td>70</td>
<td>10</td>
</tr>
<tr>
<td>Organic pigment</td>
<td>&lt;10</td>
<td>12</td>
</tr>
<tr>
<td>Paraformaldehyde</td>
<td>23</td>
<td>6</td>
</tr>
<tr>
<td>Pea flour</td>
<td>25</td>
<td>15</td>
</tr>
<tr>
<td>Polyethylene (HDPE)</td>
<td>26</td>
<td>10</td>
</tr>
<tr>
<td>Resin</td>
<td>&lt;63</td>
<td>10</td>
</tr>
<tr>
<td>Wood</td>
<td>27</td>
<td>10</td>
</tr>
</tbody>
</table>

Table 2: Limiting oxygen concentration for various dusts

5.1.3 Hybrid mixtures
As Figure 20 shows, the limiting oxygen concentration of hybrid mixtures is determined by the combustible material with the lowest limit value.

5.1.4 Various inerting gases
In addition to nitrogen, carbon dioxide and water vapor are used as inert gases. With regard to the limiting oxygen concentration, carbon dioxide has the best inerting properties (see Figure 21).

![Figure 21: Influence of the oxygen concentration on the explosion characteristics of lignite dust for different inert gases](image)

5.2 Oxygen measurement
In conjunction with inerting, both stationary apparatus for continuous measurement and portable devices for periodic measurement of the oxygen concentration are used.

The measurement method most frequently employed is based on the high paramagnetism of oxygen. Unfortunately, it is not possible to make statements of general validity regarding the optimum oxygen analyzer. Rather, the best instrument for the application in question should be selected in cooperation with the supplier. Here, the following points warrant special attention: type and location of use, other components of the gas, temperature and pressure, required availability and maintenance, as well as the explosion protection needed by the device.
5.3 References dealing with inerting

[1] VDI 2263, part 2, Staubbrände und Staubexplosionen; Gefahren – Beurteilung – Schutzmassnahmen; Inertisierung, 1992


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April 1994

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