

# Transfer of powders into flammable solvents overview of explosion hazards and preventive measures

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## Abstract

This paper evaluates and analyses the explosion hazards associated with transfer operations of powders into large containers, hoppers and reactors, which may also contain flammable solvent vapours. The formation and characteristics of explosive atmospheres and the occurrence of process inherent ignition sources, such as static electricity are described. The most commonly applied preventive measures and technical equipment are outlined and discussed including their advantages and disadvantages.

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## 1. Introduction

The transfer of powders into flammable solvents is a very common operation frequently applied in the different branches of the process industry. Typically additives, pigments, catalysts, or any kind of reactants have to be added in powder form into reactors, large containers and hoppers. Very often the recipient container contains a flammable solvent either already charged in a large amount or as residue from the previous operation or residue from an intermediate washing operation.

Depending on the flashpoint of the solvent as well as the liquid and ambient temperature, an explosive vapour atmosphere may be built up in the container and in the surroundings of the container. In case the powder transferred to the container is also combustible in addition an explosive dust/air mixture may be formed in the container as well as in the surroundings.

Together with the vapour from a flammable solvent, the explosive dust/air mixture may form a hybrid mixture. Even if all precautions to exclude the common and trivial effective ignition sources, such as ignition sources related to electrical equipment, mechanical load, cutting, welding,

open flames, smoking, etc. have been considered, there remains the hazard of electrostatic ignition sources associated with the transfer process itself.

Based on the explosion hazards outlined above it is not at all astonishing, that this type of operation is one of the most hazardous in the process industry. Also the incident statistics clearly demonstrate this fact. Not only the probability, but especially also the severity is extremely high. Normally at least one and very often even several operators are involved in such transfer operations. In case of an explosion they will be directly exposed to the blast wave and the fireball, which, especially in case of a dust cloud or hybrid mixture explosion, causes serious if not life threatening burns.

For all these reasons listed above, measures of explosion prevention are of utmost importance. As clearly stated in the ATEX directives ([Directive 94/9/EC, 1994](#)) and ([Directive 1999/92/EC, 1999](#)), the employer shall take technical and/or organizational measures appropriate to the nature of the operation, in order of priority and in accordance with the following basic principles:

- The prevention of the formation of explosive atmospheres, or where the nature of the activity does not allow that,

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- the avoidance of the ignition of explosive atmospheres, and
- the mitigation of the detrimental effects of an explosion so as to ensure the health and safety of workers.

These measures shall, where necessary, be combined and/or supplemented with measures against the propagation of explosions and shall be reviewed regularly and, in any event, whenever significant changes occur.

Thus, also in the transfer operations most attention should be paid to the exclusion of explosive atmospheres. In the following sections the formation, characteristics and probability of an explosive atmosphere, the build-up and efficiency of static electricity as an ignition source as well as the possible preventive measures will be outlined more in detail.

## 2. Formation, probability and minimum ignition energy of explosive atmospheres

For an assessment of the explosion hazard during the transfer of powders into a reactor preloaded with a flammable solvent, the ignition sensitivity of the explosive atmosphere and the probability of its occurrence at the different locations (within the reactor above the solvent surface and in the region of the manhole) are of great importance. Based on this information it can be assessed, whether an ignition source, such as e.g. static electricity will become effective. The ignition sensitivity of the explosive atmosphere is characterized by the minimum ignition energy (MIE) and its occurrence depends among other things on the vapour pressure of the solvent, the dispersibility of the powder and the adjustment of the ventilation.

The well-known relationship between the vapour pressure curve of a flammable liquid, the explosive range of the vapour and the flashpoint is shown in Fig. 1. Most of the commonly used solvents in the process industry, such as

white spirit, toluene, acetone, ethyl acetate, ethanol, methanol, isopropanol, etc. show a flash point far below room temperature.

Furthermore, it must be kept in mind that the explosive range increases with temperature.

In addition, an explosive dust cloud may be formed in the reactor, in the region of the manhole and in the surroundings. This depends on the nature of the powder, that is to say the particle size distribution of the powder, the moisture content, the concentration and the explosibility of the whirled up product characterized by the LEL, MIE, the minimum ignition temperature, etc.

At least within the reactor and in the region of the manhole, the dust cloud will become mixed with the solvent vapours and will form a hybrid mixture. The characteristics of hybrid mixtures have extensively been investigated in the past (Bartknecht, 1993). The most relevant properties are as follows:

- Even if the concentration of both components, of the dust cloud and of the solvent vapour lie below their own lower explosion limit, the hybrid mixture as a whole may be within the explosive range.
- The MIE of the hybrid mixture usually lies between the MIE of the components. Since the MIE of the solvent vapour is usually lower than the MIE of the powder, the MIE of the hybrid mixture is usually much lower than the MIE of the pure powder, even if the flash point of the solvent is above ambient temperature.

If the gas or vapour concentration is below 20% of the lower explosion limit, the effect of the gas or vapour can be neglected (BGR 132, 2003). This means that the MIE of the pure powder represents the proper value. Vapour pressure curves of common solvents show, that a concentration of 20% lower explosion limit is commonly reached at a temperature of about 30–40 K below the flash point as shown in Fig. 2. This is of course only true, if the flash point is close to the temperature corresponding to the lower explosion limit according to the vapour pressure

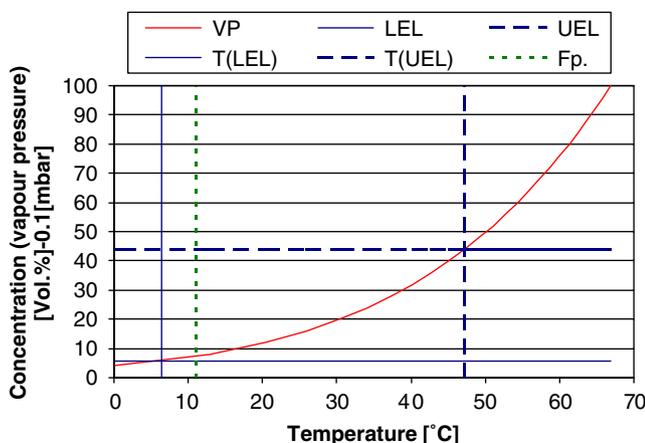


Fig. 1. Relationship between the vapour–pressure curve, the explosive range and the flashpoint of methanol.

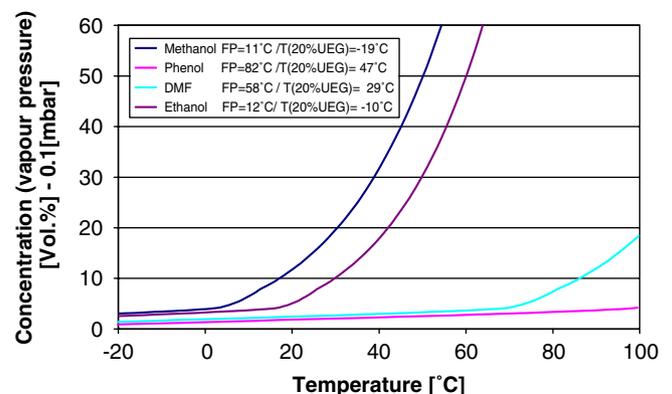


Fig. 2. Vapour–pressure curve for different solvents at concentrations below the lower explosion limit.

curve, which is commonly the case. Thus, this “30–40 K rule” can be used as a rule of thumb to judge the probability of the formation of a hybrid mixture.

In the absence of a vapour atmosphere formed by residues of a preloaded solvent in the reactor, residual solvent within the powder may also form the vapour atmosphere. As a rule of thumb no hybrid mixture has to be anticipated, if the content of residual solvent within the powder is below 0.5% by weight (BGR 132, 2003). This rule does, however, no longer apply, if the powder containing the solvent is ground (formation of new surfaces and subsequent desorption of the solvent).

With respect to the occurrence of the most probable possible ignition sources (see Section 3) the presence of an explosive atmosphere at the following locations has to be evaluated:

1. Close to the surface of the solvent.
2. In the upper part of the reactor.
3. In the region of the manhole inside and outside of the reactor.

If the flash point of the solvent is very low (high-vapour pressure at room temperature), the atmosphere is most probably over saturated within the reactor (locations 1 and 2), whereas in the region of the manhole, it will most probably be within the explosive range. If, however, large amounts of powder are transferred into the reactor air will be entrained and the atmosphere may also become explosive inside the reactor.

If the flash point is only slightly below the ambient temperature as in the case of toluene or methanol, the whole gas phase in the reactor from the liquid surface to the manhole may be filled with an explosive atmosphere, which—particularly for toluene—is at its most ignition sensitive concentration.

Depending on the dispersibility of the powder and the way of its transfer, an explosive dust cloud may be present within the whole reactor and in the region around the manhole. Thus in combination with the presence of solvent vapours, hybrid mixtures can be formed throughout the reactor and the region of the manhole.

### 3. Formation and probability of effective ignition source

#### 3.1. Mechanical sparks and hot surfaces

During most transfer operations of powders into a liquid phase in the reactor the agitator is running to prevent the formation of lumps. In case of any mechanical defect of the agitator mechanical sparks or hot surfaces may be generated which could ignite an explosive atmosphere within the reactor.

Another possible ignition source is the rotating mechanical seal of the agitator axis. Hot surfaces cannot be totally excluded at this location.

#### 3.2. Static electricity

Discharges due to the build-up of static electricity may be generated at different locations and during different transfer steps. Their occurrence also depends on the way of transfer and on the way of addition of the powder to the reactor. Typical possible discharges are (see (CENELEC, 2003) for definition of the terminology):

- Spark discharges from the operator, if he is not reliably earthed.
- Spark discharges from any conductive but not earthed auxiliary device used in the transfer procedure, e.g. shovel, funnel, chute, pipe, etc.
- Brush discharges from any non-conductive auxiliary devices, e.g. shovel, funnel, chute, pipe, etc.
- Spark discharges from any conductive but not earthed bag, bin, drum, container, etc., from which the powder is transferred into the reactor.
- Brush discharges from any non-conductive bag, bin, drum, container, etc., from which the powder is transferred into the reactor.
- Spark discharges from any conductive but not earthed fixtures and fittings within the reactor.
- Brush discharge from the dust cloud formed within the reactor during the transfer of the powder.
- Brush discharges from the charged solvent, suspension or emulsion preloaded in the reactor.
- Brush discharges from the powder heap formed on top of the liquid phase within the reactor.
- Cone discharges from the powder heap formed on top of the liquid phase within the reactor.

Though, those electrostatic ignition sources associated with the equipment, the packaging and the operators, can in principle be removed by adequate measures (use of conductive materials and reliable earthing), the discharges associated with the products will remain. They cannot be removed without significant changes of the product properties. More details about the incendivity of the electrostatic discharges with respect to gases vapours, dusts and hybrid mixtures are given in the literature (BGR 132, 2003 and CENELEC, 2003).

### 4. Measures to exclude explosive atmospheres

When powders are transferred into a reactor preloaded with a flammable solvent in an open way, it is very difficult to prevent explosive atmosphere due to gas, solvent vapour, dust or hybrid mixtures, as outlined in Section 2. In addition, it is very dangerous only to rely on the measure “exclusion of effective ignition sources” to prevent explosions as outlined in Section 3. This applies not only to situations with flammable solvents but also to situations where very ignition sensitive powders with a MIE below 10 mJ are transferred into a solvent free container. Experience in practice demonstrates that also in this case

it is very difficult to exclude effective ignition sources (Glor, 2001 and Glor, 2004). In consideration of the fact, that in case of an explosion not only equipment and construction may heavily be damaged, but also life of personnel is in danger, this operation should nowadays no longer be performed in an open way. The transfer of powder into a flammable solvent should always be performed under inert conditions, i.e. the oxygen within the reactor must be reduced below its critical level called limiting oxygen concentration (LOC), below which an explosion is no longer possible (Bartknecht, 1993).

Reduction of oxygen is usually achieved by the addition of nitrogen, carbon dioxide or another inert gas to the reactor. A common method is evacuation of the reactor and subsequent filling with the inert gas. More technical details are described in the literature (ESCIS (Expert Commission for Safety in the Swiss Chemical Industry), 1993). Experience shows that when opening any access port of the reactor the inert atmosphere will no longer be maintained within the reactor (see Fig. 3). Due to diffusion, turbulence and entrainment of air, the oxygen concentration will immediately increase within the reactor and exceed the LOC after a short period of time. Thus, the transfer of the powder via any kind of a lock into the preinerted reactor is the only safe method of transfer and represents nowadays the state of the art of such an operation.

There exist different lock systems. The most commonly applied systems are schematically shown in Fig. 4.

Table 1 summarizes the characteristics of the different transfer systems. Inherent to all transfer systems, except the PTS-System, is the oxygen enrichment within the reactor with increasing amount of powder transferred. This is particularly a handicap in case of products with low-bulk density (apparent density in contrast to skeletal density) and/or transfer of large amounts (volumes) of powders, which will nearly fill the total free gas space within the reactor. Fig. 5 shows this effect of oxygen enrichment in the reactor due to the air contained in the bulked powder.

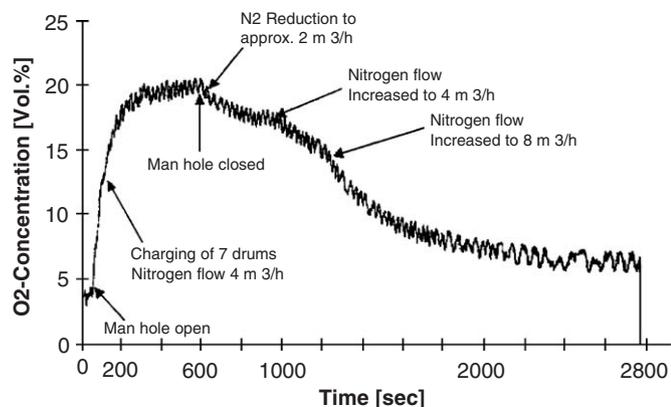


Fig. 3. Oxygen concentration in a preinerted reactor after opening the man hole and addition of powder (Hoppe & Jaeger, 2005).

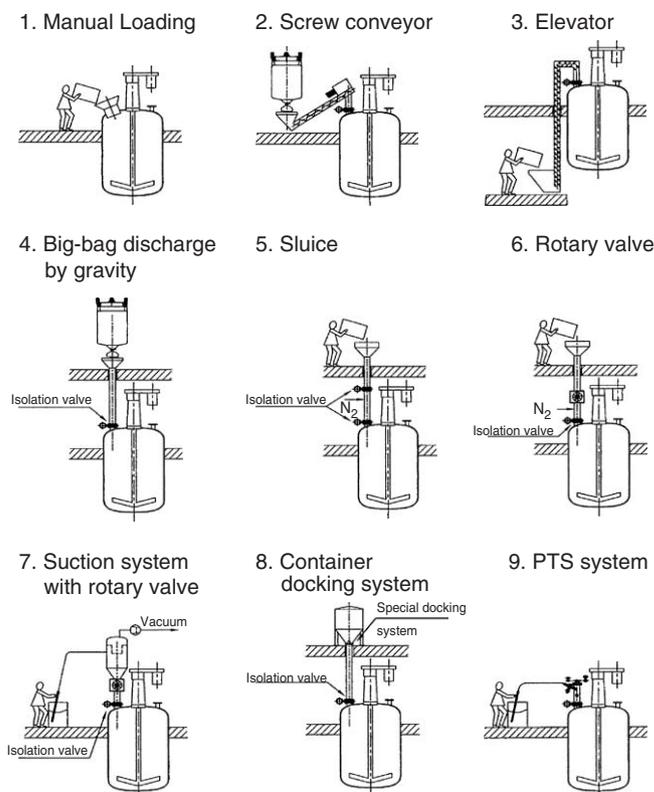


Fig. 4. Different methods of powder transfer into a reactor.

## 5. Practical aspects—containment and explosion protection

When considering the Pharmaceuticals Industry, in addition to the safety aspects related to the charging of powders into flammable atmospheres, the toxicity of the powders must also be taken into account. The constantly increasing toxicity and reactivity of products in recent years as well as the ever more demanding production standards with regard to quality have made containment an unavoidable factor and manual handling obsolete.

Few approaches have been implemented that eliminate the charging of powders through open manholes. Most methods use gravity to charge powder however, with an improved charging mechanism as shown in the Fig. 6. Most of these methods are based primarily on the need to improve the containment of powders and do not take into consideration the safety issues concerning the risks of explosion.

The choice between the various approaches is based on the toxicity of the powders, the amount to be charged and the type of packaging. Often different approaches coexist within the same production unit. Most frequently, the powder is positioned on a higher floor and falls through a chute into the production equipment (dryer, reactor, etc.) located on a lower floor. The loading zone can be confined, e.g. by a laminar flow booth, and the drums can be emptied with a drum lifting system. In this particular case, it is necessary to protect the operators with safety suits and gas masks with external breathing air.

Table 1  
Characteristics of the different powder transfer methods

	Manual transfer	Screw conveyor	Bucket/chain conveyor	FIBC discharge	2 valve system	Rotary valve	Vacuum transfer with lock	Docking station for containers	PTS- system
<i>Prevention of explosive atmosphere</i>									
Transfer to closed reactor, inerting possible	—	+	++	++	++	++	+	++	+++
Entrainment of air with powder transfer highly improbable	—	++	—	+	+	+	+	+	+++
Entrainment of air within bulked product excluded	—	—	—	—	—	—	—	+	+++
Repeated inerting not required for transfer of large quantities	—	++	—	+	+	+	+	+	+++
Inert atmosphere maintained after transfer	—	+	—	+	+	+	+	+	+++
Diffusion of flammable gases or vapours to surroundings excluded	—	+	+	+	+	+	+	+	+++
Formation of dust cloud in surroundings not expected	—	+	—	+	—	+	+++	+++	+++
<i>Other advantages</i>									
Required space (particularly above the reactor) low	+	+++	.	—	.	—	++	—	+++
Easy to clean	++	+	—	++	+	+	+	+	++
Mobile transfer system	+++	++	—	—	—	—	—	—	++
Transfer into pressurized systems	—	—	—	—	—	—	—	—	+++
Not depending on flow properties of powder	+++	—	+++	+	+	+	+	++	+++
GMP (good manufacturing practice) Conformity	—	+	—	+	+	+	+	+++	+++
Transfer over large distances	—	+	++	—	—	—	++	—	++
Investments	+++	+	—	+	+	—	—	—	+
Charge moist or solvent wet powder	+++	+	++	++	+	+	+	+	+++
For multipurpose applications	+	—	—	++	+	+	++	+	+++
Provides manufacturing flexibility	+	+	—	++	—	+	+	—	+++
Automated operation	—	+	+	+	+	++	++	++	++
Environmental health & safety	—	+	+	+++	+	+	+	+	+++

Another common approach is to use containers equipped with special automatic connecting valves (active and passive) or flexible intermediate bulk containers (FIBC) with a special docking device allowing the receiver to be connected and disconnected in an almost air-tight manner (see Fig. 7). This method makes it possible to deal with large quantities of powder (> 100 kg) while decreasing the need for manual handling. It is also useful when dealing with an intermediate product that needs to be stored or quarantined between different production stages.

One of the only solutions when dealing with very toxic products is to use a glove box (see Fig. 8). This provides good protection for the operator, the product and the environment.

However, since glove boxes are designed for specific tasks, they offer very little flexibility. They take up a lot of space and the investment costs are substantial.

With all these methods powder is loaded by gravity into various types of vessels. Due to the space required by these installations, it is usually necessary to provide a separate room on the upper floor just for the powder handling. The connecting tube between the loading zone and the equipment to be charged is often a source of problems. The well-known phenomenon of clogged-up (powder bridging) chutes occurs frequently when handling products with poor flow characteristics or when there is a lot of moisture present during loading. Neither cleaning nor validations of these tubes, which are often several meters long, are easy.

As explained previously, such systems, which charge by gravity can generate several problems concerning safety. It is generally difficult to render these installations inert and there is a risk of introducing oxygen into the vessels while they are being charged with powder. The nitrogen

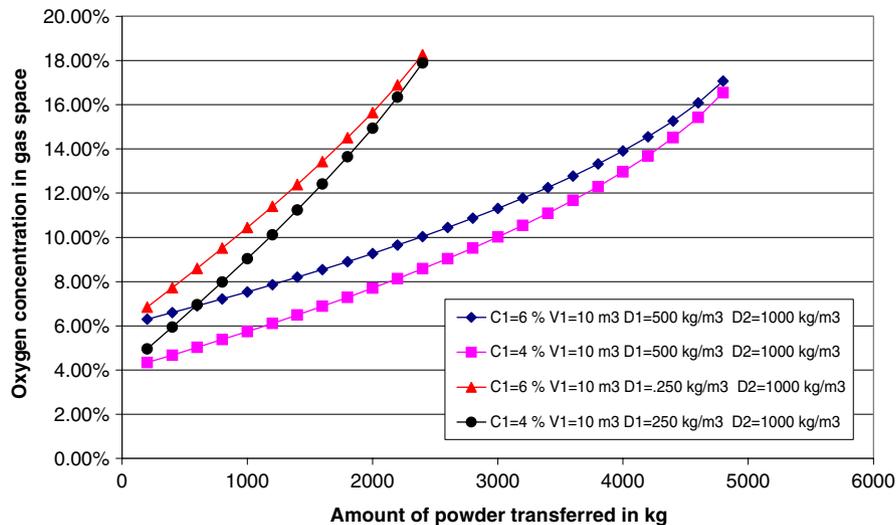


Fig. 5. Oxygen enrichment during transfer of powder into a preinerted vessel. C1: Oxygen concentration in the reactor before the transfer, V1: volume of the gas phase in the reactor before the transfer, D1: bulk density of the powder transferred, D2: skeletal density of the powder transferred.

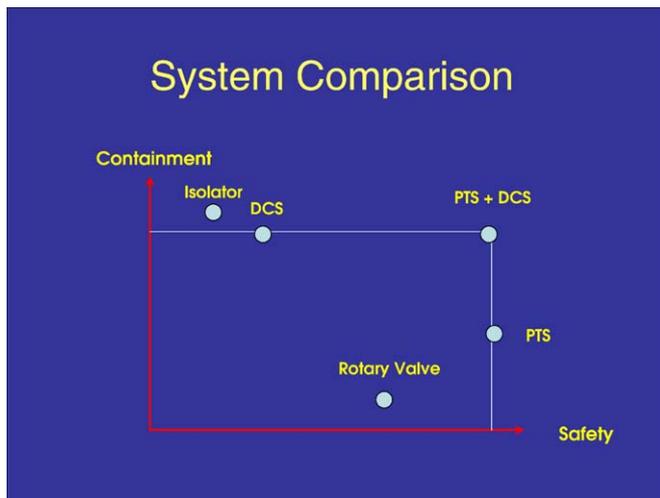


Fig. 6. Improver gravity charging mechanisms.

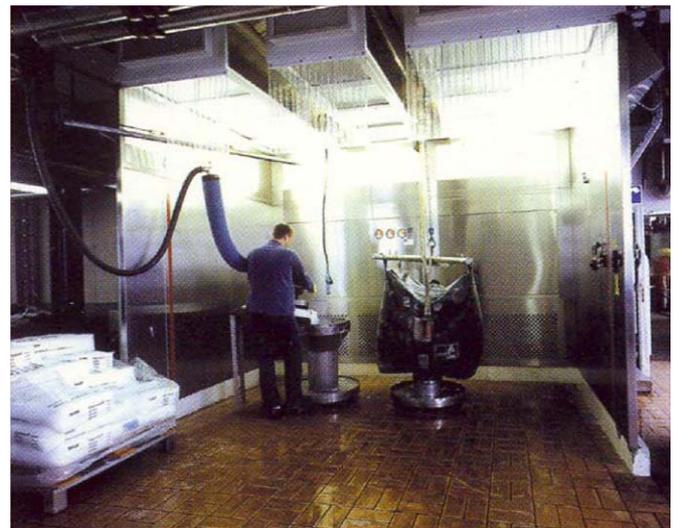


Fig. 7. Charging from FIBC in the upper floor.

consumption is usually very high and costly. In order to compensate for these inadequacies, it is often necessary to integrate complex instrumentation which can lead to a decrease in reliability of the process or can modify the manufacturing process e.g. charging an empty reactor without any solvent present, thereby jeopardizing the efficiency or lifetime of production equipment.

The disadvantages of charging an empty reactor, i.e. without the presence of solvents are various:

- Risk of damaging the agitator seal or the agitator itself due to the large amount of solids at the bottom of the reactor.
- Damage to the reactor lining through the abrasion of the powder.
- Long mixing time and problems with product homogenisation due to the formation of agglomerates.
- High static created by the introduction of powder in dry conditions.

A further important aspect of the ATEX standards is the delimitation of the various zones in a production unit. The choice of equipment and its set-up can influence the way the zones are determined, making it possible, for example, to downgrade certain zones and thus benefit from the direct economic and operational advantages.

Most powder handling systems are not designed neither to be pressure proof nor explosion proof. Yet, they can be directly connected to other equipment, which may be under pressure, contain an explosive atmosphere and run at high temperatures. In this case, as there is no physical barrier between the two systems during the powder loading and, due to the direct communication between systems, there is a potential risk of an explosive atmosphere developing in the powder-handling zone. Consequently, the powder-handling zone must be classified as an explosive zone.

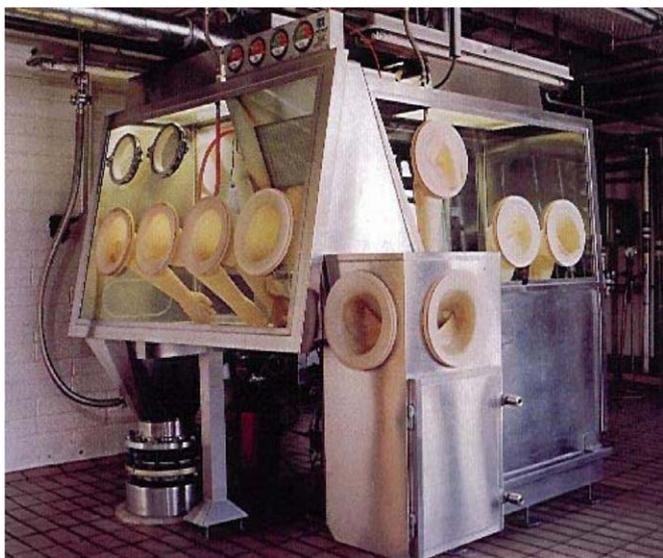


Fig. 8. Charging through a glove box.

Due to an increasingly difficult economic situation, the pharmaceutical and chemical industries face many challenges when designing production facilities. Production units must remain very flexible in order to adapt quickly to changes in the market and to conform to the very strict safety and quality control standards. The operation and maintenance costs must be kept to a minimum.

These industries are confronted with a dilemma. In order to increase their productivity they need to change their way of charging powders into reaction vessels, this however, is not possible in most cases due to the design of the commonly used powder handling equipment.

For example, in order to decrease batch time and energy consumption powder is preferably charged in a controlled manner into a reactor already filled with solvents. This occurs—if possible—already at the temperature where the reaction will take place. The other advantage of charging powder in the liquid phase is that it also increases the lifetime of the equipment and at the same time it decreases maintenance requirements.

As explained before, most of the systems that use gravity are not intrinsically safe and do not allow such operations. Therefore, manufacturers have to accept either a loss of productivity or to take unconsidered risks in order to improve their processes. In order to overcome these various problems and to comply with safety as well as process requirements, the charging system must be able to isolate the process equipment during the loading phase and to transfer powder in a contained manner.

One technology does comply with these requirements. This solution is based on the concept of transferring powder in an active way without the use of gravity. It is thus possible to view the handling of powder (dry and moist) like the handling of liquids. The heart of this concept is the PTS (Powder Transfer System, patented, see Fig. 9), which uses a source of vacuum and a source of pressure to transfer powder. Powder can be transferred



Fig. 9. Powder Transfer System (PTS).

from almost any receptacle (drum, big bag, container, process equipment, etc.) and over long distances, thus allowing for more flexibility when designing production units. The operating principle of the PTS is as simple as it is effective. Powder gets sucked into the PTS chamber by vacuum. A flat filtration membrane, installed in the upper part of the system, ensures that no fine product particles can enter the vacuum line. As soon as the chamber is full, the cycle is reversed and powder is discharged into the receiving vessel by compressed gas. At the same time, the filter membrane is cleaned by the reverse flow of compressed gas, thus ensuring its optimum performance.

The PTS, designed for pressure, is directly installed on the receiving vessel, which it can isolate during the loading phase. One of the major advantages of this technology is that it makes it possible to separate the air from the powder and to keep the receiving vessel inert while loading the powder by using, for example, nitrogen to discharge the PTS chamber. It is, therefore, possible to safely fill powder into a reactor, which contains solvents or is pressurized, without risking explosions or dangerous gas leaks.

## 6. Conclusions

During transfer operations of powders into reactors, containers or agitators preloaded with flammable solvents many fires and explosions have occurred in the past in different

branches of industry (Fishwick, 2003). Even in the absence of flammable gases or vapours, transfer operations may lead to fires and explosions, particularly if explosion sensitive powders (low MIE) are involved. Such operations are frequently still performed manually. As a consequence personnel are always affected in case a fire or explosion occurs.

According to the present state of the art such transfer operations should nowadays always be performed in a so-called closed way into preinerted reactors or containers. Care must be taken that the required low degree of oxygen concentration is maintained during and after the transfer. As outlined above, the safety of most of these systems, which use gravity for transfer is far from absolute and depends on powder characteristics (bulk density, etc.) and process conditions. In the worst case, a process, which is considered safe for a specific activity can become unsafe in case one of the process parameters is changed and a new risk assessment has to be carried out. The PTS, however, as is does not use gravity and can eliminate the air comprised in the powder, is particularly safe and functions independently of the process parameters and powder characteristics.

In view of the flexibility and frequent changes required of a production facility and due to the pressure on manufacturers for continuing process optimisation, it is important to select charging equipment that can guarantee full safety at all times, independently of the process parameters and powder characteristics.

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