

Prevent Explosions During Transfer Of Powders Into Flammable Solvents

Explanations of the development and characteristics of explosive atmospheres and the potential for an explosion to occur are given, along with the advantages and disadvantages of preventative practices

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Explosion prevention must be a primary objective wherever the process of transferring powders into flammable solvents is utilized, irrelevant of the industry and existing practices, in accordance with regulatory guidelines such as the ATEX directives (Directive 94/9/EC, 1994 and Directive 1999/92/EC, 1999), which prevail in Europe.

The addition of powders such as catalysts, pigments and other reactants into a reactor, hopper or large container is an extremely common operation within the chemical process industries (CPI). Frequently, the vessel into which the powder is being added will already be charged with flammable solvents in large amounts, or contain residual levels from previous use or intermediate cleansing procedures.

The presence of flammable solvents may create an explosive vapor atmosphere both within the vessel and in the immediate environment, depending on the flashpoint of the solvent, the temperature of the liquid and the ambient temperature outside the container. Equally significant are the nature of the powder and the act of transferring it. Whatever the combustibility of the powder, the combination of the powder's characteristics in addition to the transfer, increases the potential for formation of an explosive dust/air mixture, both in the container and in the immediate surroundings. The amalgamation of flammable solvent vapors and explosive dust/air

mixtures can form a volatile hybrid mixture.

The presence of such explosion hazards, further supported by incident statistics, corroborates the fact that this type of operation is clearly one of the most hazardous within the CPI. Even if all effective ignition sources generally considered common and insignificant, including those ignition sources related to electrical equipment, mechanical load, open flames, cutting, welding and smoking, have been excluded through the introduction of precautionary measures, the hazard of electrostatic ignition sources inherent in the powder transfer will still remain a viable possibility for causing an explosion.

In short, the probability of an explosion occurring within the transfer process is high. Furthermore the severity of such an explosion could be disastrous, especially when the number of operators that would be directly exposed to the initial blast wave and subsequent fireball are taken into consideration. Serious, if not life threatening, burns are possible, especially in the presence of a dust cloud or hybrid mixture explosion.

It is therefore paramount to ensure the appropriate organizational, operational and technical systems relating to the nature of the process and the materials in use are in place in conjunction with ATEX directives and the

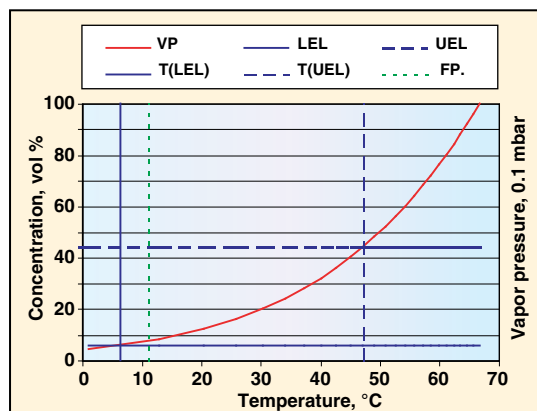


FIGURE 1. The affiliation between methanol's vapor-pressure (VP) curve, the explosive range of its vapor and its flashpoint (FP) is similar to that of most commonly used solvents. The majority of common solvents have flashpoints below room temperature. Explosive range of solvents also tends to increase with increasing temperature. Note T(LEL) and T(UEL) are the respective temperatures at methanol's LEL and UEL

following basic principles:

- Prevent the formation of explosive atmospheres

Where prevention due to the nature of the process and materials is precluded, then the following criteria apply:

- Ignition sources must be avoided
- Mitigation of the detrimental effects of an explosion must be a priority to ensure the health and safety of operators

In conjunction with the above factors, frequent reviews of the practices and processes in place must be carried out, the primary goal of which should be to avoid propagating explosions. If any part of the process changes, then additional reviews and precautionary measures should be introduced.

Probability and cause

Determining the risk of an explosion hazard is very important in assessing how a process should be carried out and whether adequate precautionary measures are in place. In order to calculate the explosion risk associated with charging a powder into a reactor already containing flammable solvents, two main criteria need to be established:

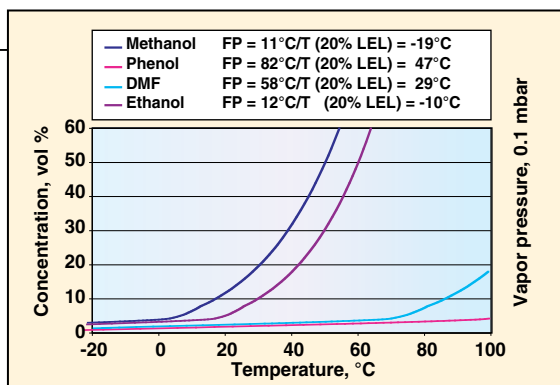


FIGURE 2. The vapor pressures of most commonly used solvents reach the concentration of 20% of the LEL at temperatures 30–40 K below the flashpoint

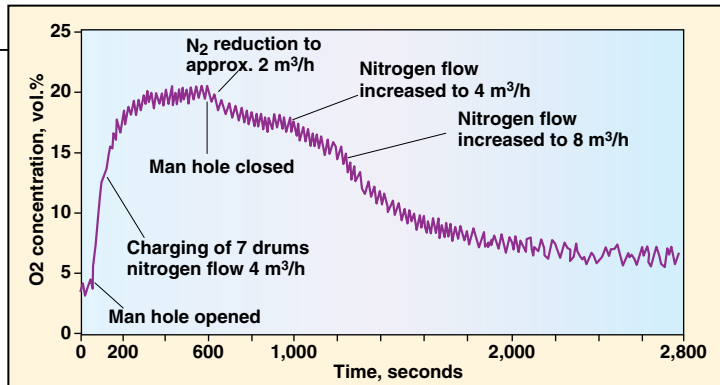


FIGURE 3. The opening of any access port and the addition of the powder itself will cause a previously inerted reactor atmosphere to be lost [10]

- The ignition sensitivity of the explosive atmosphere, which is categorized by the minimum ignition energy (MIE)
- The probability of an explosion occurring at different locations (highest probability during transfer of powder into a flammable solvent occurs in the reactor above the solvent surface, such as where vapors exist, and at the manhole where powder is charged into the reactor and turbulence is high coupled with vapor release from within the reactor).


Factors such as the solvent vapor pressure, powder characteristics and ventilation are key

Once these criteria have been established, the risk of a specific ignition source, such as static electricity, to create an explosion can also be determined.

For most commonly used solvents, the affiliation between their vapor-pressure curve, explosive range of their vapor and their flashpoints are well recognized (Figure 1). The majority of universal solvents, including white spirit, toluene, acetone, ethyl

acetate, ethanol, methanol and isopropanol, have flashpoints below room temperature. The explosive range of solvents tends also to increase with increasing temperature.


Explosive dust clouds formed during the transfer of powders can be located within the reactor or at the point of entry into the reactor, for instance at the manhole and its surrounding area. Particle size and distribution, moisture content, concentration and explosibility of the powder in its tumultuous state when being charged



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to the reactor, make up the powder characteristics, which can then be expressed in terms of the lower explosion limit (LEL), MIE and minimum ignition temperature (MIT).

When a dust cloud mixes with flammable solvent vapors, which can happen within the reactor or at the manhole, a hybrid mixture is formed. The explosion risks of hybrid mixtures have been extensively reported [7]. However, the most relevant points regarding their characteristics are as follows:

- A solvent's MIE is generally much lower than that of a pure powder; therefore, when a hybrid mixture is formed, its MIE as a rule will be somewhere between the two and tends to veer towards the lower range, even if the flashpoint of the solvent is above ambient temperature
- Irrelevant of whether the concentrations of the dust cloud and solvent vapor are below their own LELs, a

hybrid mixture is an entirely separate entity that may well be within the explosive range

There are exceptions regarding the explosive properties of a hybrid mixture, specifically the MIE. If for instance the vapor concentration is below 20% of the LEL of the solvent, the MIE of the pure powder would then be representative of the explosion risk for the hybrid. The vapor pressure, temperature, LEL and flashpoint of solvents are used in conjunction with each other to determine the probability of a hybrid mixture forming under any specific environmental conditions. The "30-40 K" rule applies to this calculation; in that the vapor pressure reaches a concentration of 20% of the LEL at temperatures 30-40 K below the flashpoint of most commonly used solvents (Figure 2).

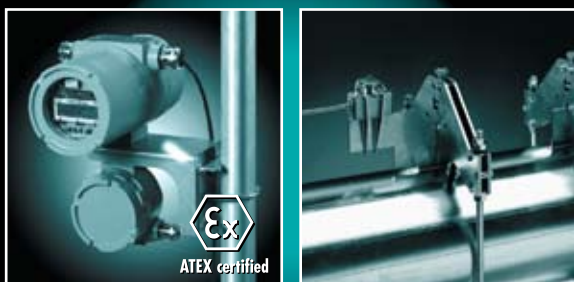
Vapor atmospheres can also be created by charging powder where solvents or solvent residue, and therefore

vapors are not already present in the reactor, as the powder itself may contain solvent residue capable of creating a vapor atmosphere. If solvent residue in a powder is present at less than 0.5% (by weight) the risk of a hybrid mixture being formed can be negated as a rule, the exception being when the powder is ground up, allowing the desorption of vapors and therefore creating a vapor atmosphere.

A powder's dispersibility and method of transfer will affect whether an explosive dust cloud will be present within the whole reactor and in the region around the manhole, consequently (and dependent on the presence of solvent vapors), hybrid mixtures may be formed throughout the reactor as well as in and around the manhole.

In the case of toluene or methanol, which are solvents with flashpoints only slightly below ambient temperature, the entire gas phase within the

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reactor — for instance, from the liquid surface to the point of entry (manhole) — can be filled with an explosive atmosphere. In their gas phases, solvents are at their most ignition-sensitive concentration. This is especially the case for toluene.

Conversely, if a solvent has a low flashpoint (high vapor pressure at room temperature) the environment within the reactor will tend to be saturated. In this instance the most explosive range will occur around the manhole. However, if large amounts of powder are conveyed into a reactor containing a solvent with a low flashpoint, the entrainment of air associated with the operation may also cause the atmosphere within the reactor to become explosive.

Process ignition sources

Static electricity. The occurrence of static electrical discharges at different locations and during distinct phases of the powder-transfer process, are dependent on the methods used for transferring the powder and charging it into the reactor. Although electrostatic ignition sources associated with the packaging, the equipment and the operators can in theory be removed with the use of conductive materials, reliable earthing and other such measures, discharges associated with the products will remain. Substantial changes to the product properties would be required in order to remove the electrostatic ignition sources within specific products.

Details regarding the incendiary properties of the electrostatic discharges related to gases, vapors, dusts and hybrid mixtures are given in ref. [2, 3].

Typical possible discharges include the following:

- Spark discharges from any conductive but not earthed (grounded) bag, bin, drum, container, etc., from which the powder is transferred into the reactor.

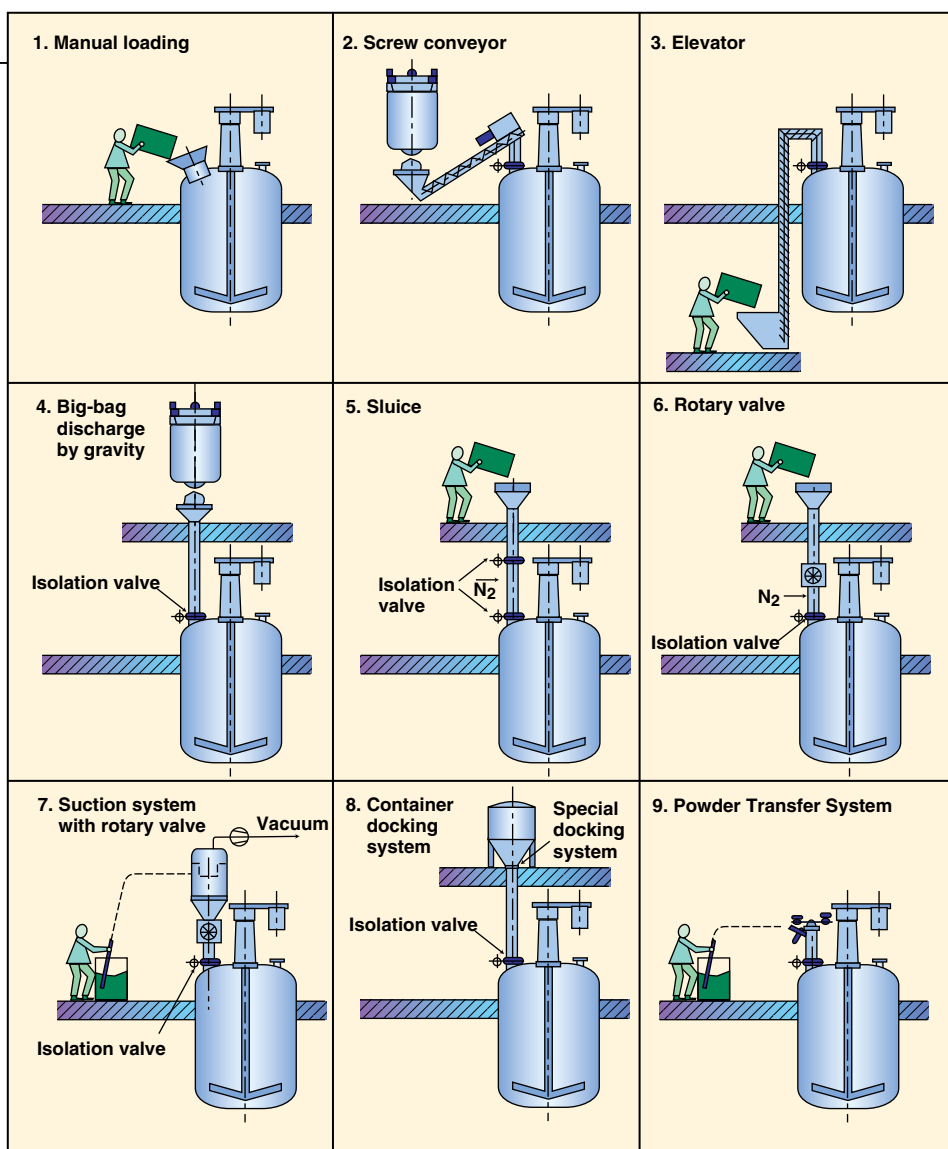


FIGURE 4. There are a number of common lock systems available for transferring powder into an inerted reactor

- Brush discharges from any non-conductive bag, bin, drum, container, and so on, from which the powder is transferred into the reactor
- Spark discharges from any conductive but not earthed auxiliary device used in the transfer procedure, including, but not limited to, shovels, funnels, chutes and pipes
- Spark discharges from the operator, if he is not reliably earthed
- Brush discharges from any non-conductive auxiliary devices, such as shovels, funnels, chutes and pipes
- Brush discharge from the dust cloud formed within the reactor during powder transfer
- Spark discharges from any conductive but not earthed fixtures and fittings within the reactor
- Brush discharges from the charged solvent, suspension or emulsion pre-loaded 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

Mechanical sparks and hot surfaces

A requisite agitator is normally running in the reactor during the transfer of powders into a liquid phase, to prevent lumps from forming and to blend the products together. The rotating mechanical seal on the agitator's axis is a potential ignition source, and a hot surface potentially present on the axis is another that cannot be ruled out. Additionally mechanical faults of the agitator, mechanical sparks caused by the operation of the agitator and hot surfaces that can be generated are also potential ignition sources of the explosive atmosphere within a reactor.

TABLE 1. ADVANTAGES AND DISADVANTAGES OF THE DIFFERENT POWDER TRANSFER METHODS

	Manual transfer	Screw conveyor	Bucket/chain conveyor	FIBC discharge	Two valve system	Rotary valve	Vacuum transfer with lock	Docking station for containers	Specially designed system
Prevention of explosive atmosphere									
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 vapors to surroundings excluded	-	+	+	+	+	+	+	+	+++
Formation of dust cloud in surroundings not expected	-	+	-	+	-	+	+++	+++	+++
Other advantages									
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	-	+	+	+++	+	+	+	+	+++

– (No); + (Sometimes); ++ (Usually); +++ (Yes)

Avoiding explosive atmospheres

If an explosion occurs, not only is it likely to cause significant damage to equipment and the infrastructure of the plant but even worse jeopardize personnel and expose them to possible injury or even death, which is unacceptable. It is clear, therefore, that operations where the transfer of powder into reactors containing flammable solvents, even where very sensitive powders with MIE's below 10 mJ are being transferred into solvent-free vessels, should no longer be carried out using open methods.

As previous sections have outlined, it is nearly impossible to prevent the formation of explosive atmospheres. Additionally, the exclusion of effective ignition sources from a process is not simple and can in no way be a guaranteed measure against explosion risks. In this day and age, it is necessary to utilize every possible precaution to

prevent explosions from happening in order to reliably protect both personnel and plant.

In order to attain the required level of safety for such transfer operations, powders must be conveyed under inert conditions, especially when the recipient vessel is preloaded with flammable solvents. Inert conditions exist where the oxygen content of the reactor is at a level below the limiting oxygen concentration (LOC), where explosions are no longer possible [1].

Reduction in the oxygen content of a vessel is achieved with the addition of carbon dioxide, nitrogen or any other inert gas (ref. [6] expands on the technical aspects of this practice). However, as illustrated in Figure 3, the opening of any access port and the addition of the powder itself will cause the previously inerted reactor atmosphere to be lost. The opening of the manhole allows the inert atmosphere within the reactor to diffuse

into the surrounding environment thus increasing the level of oxygen. The addition of powder also increases the oxygen level within the reactor due the entrainment of oxygen within the powder itself and as a result of the turbulence caused by the powder swirling around in the oxygen-rich atmosphere outside and at the manhole of the reactor. The LOC within the reactor is compromised and the risk of explosion is again present. Modern technology provides the solution to these problems; using any type of lock to transfer the powder into an inerted reactor is a safe operation.

Figure 4 illustrates some of the more common lock systems available today. Table 1 compares some of the existing lock systems against criteria for prevention of an explosive atmosphere. Notably, oxygen enrichment within the reactor is a fundamental problem associated with all the lock systems, with the exception of the PTS

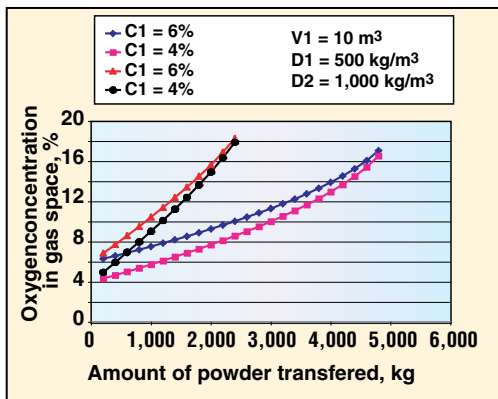


FIGURE 5. Oxygen enrichment during transfer of powder into a preinerted vessel. C1: Oxygen concentration in the reactor before the transfer, V1: volume of the gas phase in the reactor before the transfer, D1: bulk density of the powder transferred, D2: skeletal density of the powder transferred

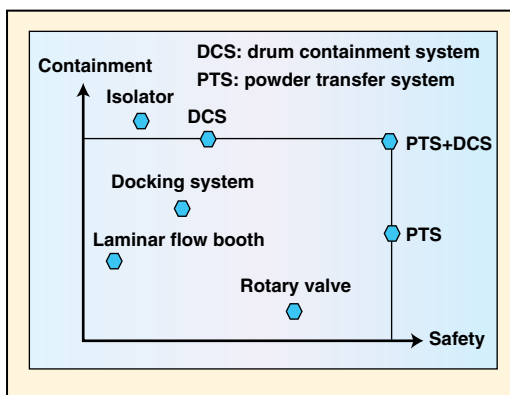


FIGURE 6. Unlike specially designed powder transfer systems, most contemporary methods for contained transfer of powders tend to be more focused on the containment aspect and do not incorporate the added need for improving the safety of the process with regard to explosion risks

system, as more powder is transferred into the reactor. Oxygen enrichment is increasingly highlighted when products with low bulk density (apparent density as opposed to skeletal density)

and/or large volumes of powders are being transferred. Figure 5 illustrates the effect of oxygen enrichment in the reactor due to the oxygen entrained within the powder.

ity and should also make manual handling obsolete.

Currently, the addition of powders into reactors through open manholes is still the norm in the CPI. The in-

Protection and containment

Consideration of the safety aspects related to the transfer of powders into flammable atmospheres must also incorporate measures that take the toxicity and the reactivity of the powder being transferred into account, especially within the pharmaceutical industry. These factors, in addition to the evermore stringent quality control and production standards, make containment an inevitability

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roduction of alternatives to this method (Figure 6) tend to be more focused on the containment aspect and do not incorporate the added need for improving the safety of the process with regard to explosion risks.

Most contemporary methods for contained transfer of powders use gravity as the impetus to charge the powder into a reactor, which requires that a multi-story facilities be built. The powder is delivered to a higher floor and falls through a chute (due to the natural effects of gravity) directly into the production equipment. The problem of containment around the loading zone is addressed by incorporating a laminar flow booth, for example, into the area and a drum-lifting system within that to prevent manual handling being required. In these instances, operators must still wear personal protective equipment including full body suits, masks and, depending on the toxicity of the powder, external respiratory apparatuses.

Alternatively, containers may be equipped with automatic connecting valves (active and/or passive) or flexible intermediate bulk containers (FIBC; Figure 7) fitted with docking devices that enable a receiver to be connected or disconnected in an *almost* airtight manner. These systems enable large quantities of powder, in excess of 100 kg, to be transferred and reduce the requirement for manual handling. This method is also suitable for processes where intermediate products are used in the process that require storage or isolation between phases.

A product with high toxicity will require more containment. Glove boxes offer one of the few practical solutions and protect the operator, product and environment (Figure 8). The cost, however, of this solution can often be prohibitive, especially considering that most glove boxes are rigidly designed for a specific use, require a large dedicated area within the plant and are not ergonomically designed therefore causing operators discomfort.

The chutes used for charging the powder into the reactors often get clogged up, so bridging can occur, especially if the powder has poor flow



FIGURE 7. Containers may be equipped with flexible intermediate bulk containers (FIBC) fitted with docking devices that enable a receiver to be connected or disconnected in an almost airtight manner

characteristics or high moisture content. Cleaning and validation is an inherent problem and increases proportionately with the length of the chute.

Gravity charging as a process, itself, can be a safety issue. The process cannot be rendered completely inert and the problems associated with increasing the oxygen concentration within the reactor are, as previously discussed, significant. The use of inert gases to reduce the oxygen content introduced to the reactor via the powder is costly as large volumes of such gases, such as nitrogen are required with this system. To counterbalance these inadequacies, more solutions are required. Sophisticated instrumentation may need to be incorporated into the system to monitor oxygen levels, for instance, but it in turn increases the cost, affects the reliability of the process by requiring calibration, maintenance and other repairs that necessitate downtime. Or, the system itself may have to be modified; charging the powder into an empty reactor may address most of the safety issues, but the efficiency of the process will be compromised. The following points illustrate the detrimental effects of charging powder into a reactor in the absence of solvents:

- Production of static electricity as powder is introduced under dry conditions
- Damage to the reactor lining due to abrasion or corrosion
- Risk of damage to the agitator seal or the agitator itself due to large amounts of solids at the bottom of the reactor
- Increased mixing cycle and problematic product homogenization due the formation of agglomerates

ATEX and other applicable standards determine the delimitation of



FIGURE 8. Glove boxes offer one of the only solutions to protect the operator, product and environment from a product with high toxicity

zones within a process environment; the choice of equipment, its setup and the methodology employed within a plant can directly impact on the determination of zones. Therefore certain zones may be downgraded, for example, where the plant would then benefit from operational advantages and associated economic benefits.

Common to the majority of powder handling systems is the lack of a physical barrier between the reactor and other production equipment thus rendering the peripheral units neither pressure nor explosion proof. The operating pressure of the recipient vessel, temperature and presence of flammable atmosphere are serious safety risks, especially when charging powder and even more so when powder is charged by gravity. There is an increased risk of the powder loading area developing an explosive atmosphere, which must therefore be classified as an explosive zone.

Economic constraints faced within the process industry create a diversity of challenges — from the conception and implementation of a flexible production unit, which complies with current quality control and safety legislation and is also capable of adapting to changing demands in the marketplace and future changes in policies, to maintaining the lowest possible overheads. Existing process systems that may need updating to comply with legislation and increase productivity face even tougher fiscal dilemmas as the nature of such systems lend themselves to modification and often stipulate entirely new systems.

Having established that gravity charging systems are fundamentally unsafe, manufacturers face the predicament of a loss of productivity versus significant costs associated with



FIGURE 9. Specially designed powder transfer systems specifically address the safety risks related to transferring powders into solvents

addressing the inherent operative risks. The solution for manufacturers is to use a system capable of isolating process equipment during the filling stage and transfers powder in a contained way.

A technology providing a total solution to the problems faced by manufacturers including safety, containment and productivity has to be chosen. The concept should lead to actively convey powder of any characteristics without using gravity, effectively in the same way liquids can be handled. The principle used has to allow the transfer of powder from any receptacle (container, drum, big bag, silo, process equipment and so on) over long distances (hori-

zontally and vertically). The problems of designing new plants or processes have to be solved by discussing and considering all arguments mentioned.

If a system is installed directly onto a reactor (or other process equipment), for instance, the two systems can be operated in isolation from each other. The technology should not only act to reduce the oxygen content of the powder before it is discharged into the reactor but also to keep the atmosphere within the reactor inert while powder is being charged into it by using nitrogen or other inert gas. Equipment should allow powder to be safely charged in to a reactor, even one that contains solvents or operates under pressure without the risk of explosions or gas leaks.

Conclusions

Historically, operations where powders are transferred into reactors have resulted most conspicuously in fires and explosions. The risks are increased significantly where flammable solvents are also present within the process. A large proportion of such operations are still carried out manually, thus exposing the personnel involved to immense safety risks.

Either in the presence or absence of flammable gases or vapors, the MIE of the powder and the method of trans-

fer can create the risk of an explosion taking place. In order to increase the safety of these processes, transfer of powders should be carried out in closed systems, the recipient vessel should be inerted, every precaution should be taken during and after the transfer to maintain the lowest possible oxygen concentrations within the reactor, the systems should be separated by a physical barrier.

Most gravity based transfer systems offer poor levels of safety overall, and risks are further compounded by the nature of the material being transferred and the process conditions. This is true to such an extent that an operation that is considered safe under one set of parameters can be destabilized by changing one small aspect of the system. A system that does not use gravity, eliminates oxygen from the powder, has a physical barrier between itself and the reactor during loading can provide a safe solution for powder transfer independent of the nature of the powder and the process parameters.

The importance of process optimization in conjunction with ever changing safety and quality criteria means that in order for manufacturers to effectively function in a competitive marketplace, the process technology they choose to apply must be flexible, guarantee full safety of their personnel, product and equipment regardless of the process parameters and powder characteristics. ■

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