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Inert Milling Systems

by Ruby Jacobson, P.Eng.

This article discusses the reasons for inerting a mill, what methods to employ to determine if a product is explosive, and the various factors used to define explosibility. It compares inerting to other protection strategies and concludes with a review of the different inerting methods.

In many of today's pharmaceutical processing plants, a high proportion of process materials assume a powder stage on their journey to the end-product. The uncontrolled handling of these powders during the process period can easily lead to the generation of electrostatic sparks and the risk of fire and explosion. As a result of this risk, inerting is being seriously looked at in order to further increase the safety of both plant personnel and the process line itself. Inerting is the process via which an inert gas is added to a combustible mixture to reduce the concentration of oxygen below the Limiting Oxygen Concentration (LOC). The inert gas is usually nitrogen or carbon dioxide. This article discusses the reasons for inerting a mill, what methods to employ to determine if a product is explosive, and the various factors used to define explosibility. It compares inerting to other protection strategies and concludes with a review of the different inerting methods.

Why Inert a Mill?

It is important for process engineers to realize that this is a question that must be answered by explosion experts, based on the characteristics of the product being milled and the condition of the product.

According to one professional process safety firm, "for a dust explosion to occur, the dust must be explosible, airborne, be in concentrations within the explosible range, have a particle size distribution capable of propagating flame, an ignition source is present, and the atmosphere must support combustion."[1]

Inerting is often chosen for processing explosible powders in mills because it is impossible to remove the ignition source from mills, impossible to reduce the dust concentration,

Figure 1. No oxygen monitoring.

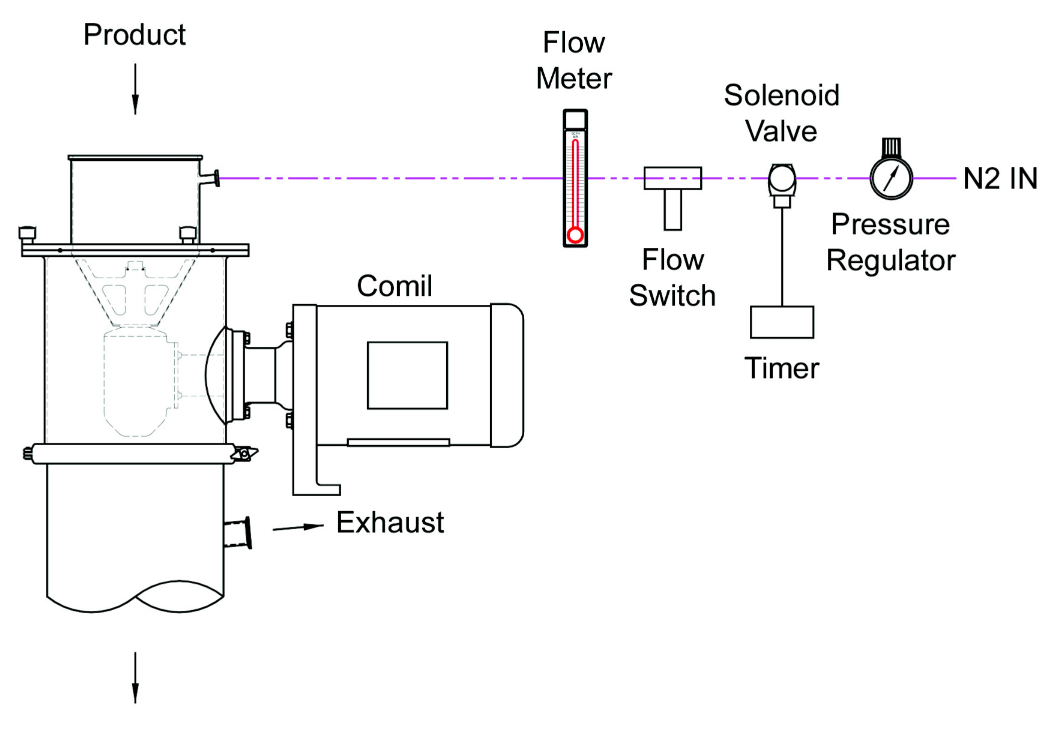

Figure 2. Mobile inert mill system with oxygen monitoring controls.

and generally, costs less than pressure-resistant processes, venting, or suppressant systems.

How do I know if my product is explosive?

Test Method - Dust Bomb

A dust bomb, 20L ($0.02m^3$) test vessel is used to determine whether the dust cloud is explosible as a result of the dust handling/milling conditions. A portion of the powder is injected into the test vessel, dispersed as a cloud by a blast of air, and exposed to an ignition source. Many federal standards such as ISO and ASTM govern the various procedures and dictate the apparatus to be used for these tests. In general, dusts, which ignite and propagate away from the ignition source, are considered "explosible."

Dusts, which do not propagate flame away from the ignition source, are considered "non-explosible."[2] However, non-explosible powders also are known to present a fire hazard and may be explosible at elevated temperatures.

Minimum Ignition Energy (MIE)

MIE of a flammable material is the minimum spark energy (in mJ) needed to ignite a material using a capacitive spark, under specific conditions. The apparatus is a modified dust bomb. An appropriate quantity of dust is placed in the dispersion cup at the bottom of a 1.2L plastic cylinder and dispersed by a blast of air. A spark is discharged across two electrodes located above the powder source. This spark is discharged at the same time as the dust cloud reaches the gap between the two electrodes. Starting with a value of spark energy that will reliably cause ignition of a given concentration of the dust being tested (note: dust concentration also is a variable), the test energy is successively halved until no ignition occurs during 10 successive tests. Then, starting at a lower energy where ignition does not occur, the energy is increased until an ignition does occur. The MIE is defined to lie between the highest energy at which ignition fails to occur in at least 10 successive attempts in order to ignite the dust/air mixture, and the lowest energy at which ignition occurs within 10 successive attempts. Powders with a MIE of lower than 10 mJ are highly sensitive to ignition. The majority of ignition incidents occur when ignition energy is below 25 mJ. The hazard from electrostatic discharges from dust clouds should be considered.[1]

Minimum Ignition Temperature (MIT)

MIT of a dust cloud or of a dust layer is a measure of its sensitivity to ignition by hot surfaces. An example of a laboratory test for MIT of a dust layer is as follows: a layer of dust is placed on a hot-plate and the temperature of the hot plate and the temperature of the dust layer are monitored. Ignition is defined as the point at which there is a significant rise in the dust temperature. The MIT is the lowest hot-plate temperature that can initiate ignition.

It is important to consider the MIT for a milling process because of the potential for heated surfaces in mills if the drive box is in the contact area, and heat is generated from inter-particle friction, screen blinding, or the impact force between the hammers, pins, or particles. A low energy mill, such as a Conical Screen Mill, generates almost no heat during the milling process. However, there are many other types of mills used in industry such as the Pin Mill or Hammermill that generate heat due to the high energy required for milling. The mill supplier would be the best source via which to determine the temperature that a powder would be exposed to, within a mill, under normal operating conditions and also, when something goes wrong. Special designs incorporating cooling jackets may be necessary for some applications/mills.

Limiting Oxidant Concentration (LOC)

The LOC is critical when designing an inert system. It dictates the Maximum Oxygen Content (MOC) allowable within the process area. Wiemann (1989) recommended that the MOC be set at 2-3% lower than the LOC determined by the lab test.[2]

The LOC is the Oxidant content measured in % volume, below which the product's dust cloud is unable to propagate a self-sustained flame. The LOC is dependent upon the material and type of inert gas used. Nitrogen is the most commonly used inert gas. Carbon dioxide and Argon also are used. Details regarding the type of inert gas to be used should be provided at time of testing.

The apparatus used for this test is similar to the one used for determining the MIE. However, the cylindrical test chamber where the dust cloud will be dispersed and where the electrodes are located is initially flushed with a specific concentration of inert gas/oxygen mixture. This same gas mixture also is used to disperse the dust sample into the test chamber tube. After a pre-set delay, a soft spark of approximately 3 J (this may vary depending on the dust) is discharged across the spark gap in the dust cloud within the tube. The test is repeated 20 times at each oxygen concentration. The LOC is basically the highest concentration at which no ignition occurred during 20 trials.

Explosion Severity

The Kst value is often cited in discussions regarding explosible powders. The Kst is a function of the maximum pressure rise during an explosion of a particular powder:

$$Kst = (dP/dt)_{max} \times V^{1/3}$$

Where Kst = material constant [bar m/s]
$(dP/dt)_{max}$ = maximum pressure rise [bar/s]
V = volume of the test vessel [m^3]

This was derived from theoretical work by Henrich and experimental work by Bartknecht.[3]

Lab test apparatus varies in size and shape. For example, the dust bomb test consists of a 1.2L cylindrical test chamber with a pressure transducer at the top with which to measure the pressure. The standard ISO vessel consists of a $1m^3$ sphere equipped with pressure sensors. The Siwek 20L sphere is similar to the ISO sphere, but on a much smaller scale. The dust sample is dispersed as a cloud into the test chamber and ignited to cause an explosion. The dust dispersion method and the igniters also vary from one test method to the next. Some dusts are dispersed from a nozzle; others are dispersed with a perforated ring in the test chamber.

This test provides the Pmax, which is the maximum pressure generated during an explosion. This information is important in the design of a mill if a pressure-resistant process is selected.

Other Tests

There are many other tests that deal with explosive materials and the severity of explosions. Some examples include Minimum Explosion Concentration, Propagating Brush Discharge, Conductivity, Charge Relaxation, Volume Resistivity, and Surface Resistivity. To test for your particular needs, it is recommended that professional process safety firms be contracted.

Inerting Versus Other Protection Strategies - When to Inert?

There are a few options available for processing explosible materials. Basically, there are five strategies that can be employed:

a) Containment: building the entire process train to withstand the Pmax of the explosion.

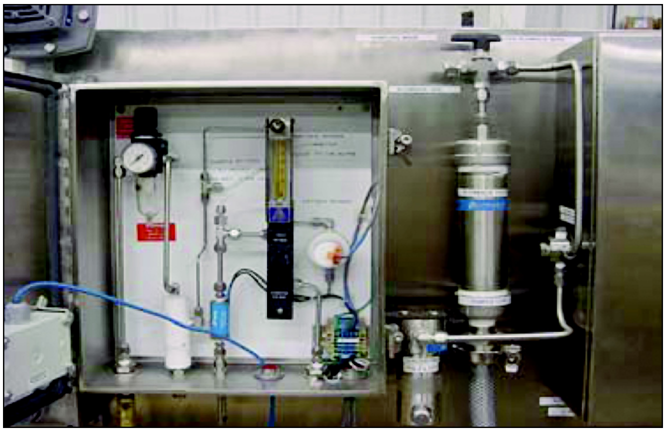

Figure 3. O_2 sampling panel.

b) Isolation: building critical components (such as a mill) as pressure rated vessels with pressure rated quick shut-off valves for isolating the mill.

c) Suppression: fire retardants are released at the onset of an explosion.

d) Venting: building vent panels on the process train to allow explosions to be directed to an external environment.

e) Inerting: inerting the process train

Suppression involves the use of water, sodium bicarbonate, or mono-ammonium phosphate that is injected at the onset of an explosion and a pressure sensor which monitors internal pressure. Venting is generally used on large, stationary equipment located close to the outside wall.

In our experience, we have found that these two methods have not practically met the needs of pharmaceutical manufacturers. Vent panels also may not be plausible due to size or location constraints. That leaves containment, isolation or inert systems.

Nitrogen costs approximately $\$0.02/ft^3$. This equates to a cost of approximately \$5/hr for a typical Inert Mill Control System.[4] Costs would vary if the entire process train was inerted, but this can be calculated based on the volume of the system and the number of air exchanges required. The operating costs and capital costs can be compared between an inert system and a pressure rated system in the selection of an appropriate strategy. Generally, the capital cost of an inert system is more cost effective than a pressure rated system. This can vary, depending on the equipment involved.

Some materials may actually generate oxygen during a process, which effectively renders inerting as not being economical or practical.

How to Inert?

Once the decision has been made to use inerting as the means via which to mill the explosible material, the next decision is what type of system is best suited to your process and budget. An inert mill can range from a very simple design to one with more precise controls. The system cost rises proportionately with the complexity of the controls. The process engineer uses risk analysis to determine the system most appropriate for his/her process. Following is a brief description of some available systems.

Types of Systems

Simple System

An example of a simple inert mill is shown in Figure 1. In this example, a conical screen mill is simply configured with a small port in the infeed chute and a vent port either in the lower portion of the mill or further downstream. When the N_2 gas enters the mill, it naturally flows downward through the mill. The N_2 infeed stream is equipped with a pressure regulator, solenoid valve (activated by a timer), flow meter, and flow switch to ensure that there is a constant flow of N_2 into the mill. If the flow of N_2 falls below a particular pre-set point, the mill is stopped. The mill is purged with N_2 before and during periods of operation. Simple calculations are done to determine the purge time and flow required for displacing the O_2 in the mill, prior to start-up.

This represents the most economical and straightforward solution. However, there are no controls on the amount of O_2 in the mill.

Inert Mill with Oxygen Monitoring

Inert milling with oxygen monitoring is the most common option employed. It monitors the oxygen content in the air/inert gas mixture and controls the amount of N_2 introduced into the system.

Gas sampling occurs on a continuous basis. A Venturi/aspirator draws the gas from the mill through a set of filters. This filtered gas mixture then flows through an O_2 sensor, which sends a 4-20mA signal to the analyzer. The analyzer converts the signal into an oxygen concentration in %.

In this system, there are two set-points: a warning level and a target/alarm level. The target/alarm level is the Maximum Oxygen Content (MOC) and the warning level is usually set at about 2% below the target level - *Figure 2*. If the O_2 concentration increases above the "warning level," the N_2 flow is increased to bring the mix back to the desired percentage. If the O_2 concentration reaches the target/alarm level, an alarm will sound and the mill shuts down. N_2 will continue to be purged into the mill until an operator turns the purge off. If nitrogen is used as the inert gas, it should not be exhausted into the process room because it may cause the asphyxiation of operators. For some systems, a local exhaust system is used to accept the vented nitrogen and the exhaust from the sampling panel. For others, the exhaust is recycled back into the process train. Some customers may opt for the additional nitrogen monitoring feature in the process room, as an added safety precaution - *Figure 3*.

Various types of HEPA filters can be used, dependent on whether the powder being milled poses a "bio-hazard" threat. This is one of the many examples of "specific requirements."

Conclusion

In conclusion, inert milling is becoming more important in today's pharmaceutical processing facilities, given the large amount of powder being processed. Before choosing to inert, there are several points that need to be considered. To answer the question "why inert?" the product has to be properly characterized.

To determine "when to inert,?" the economics and design of various explosion prevention/protection strategies ought to be considered. And finally, to determine "how to inert,?" the process should first be determined in partnership with your mill supplier. Overall, inert milling is a popular choice due to the lower costs associated with it when compared to building pressure-resistant process trains. Proper testing and process design by reputable and experienced firms are recommended.

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About the Author

Ruby Jacobson is Product Manager, Comil and Quadro Vac for Quadro Engineering, the originator of Conical Screen Milling technology. She has been involved with many projects in the pharmaceutical and food industries where the Comil is used. She graduated from the University of Waterloo with a chemical engineering Honours Bachelor of Applied Science degree in 1989 and has worked in the plastics and chemical industries prior to joining Quadro. She holds a Professional Engineers of Ontario license and is a member of the ISPE and IFT. She can be reached through Quadro's Web site, www.quadro.com.

Quadro Engineering, 613 Colby Dr., Waterloo, Ontario N2V 1A1, Canada.