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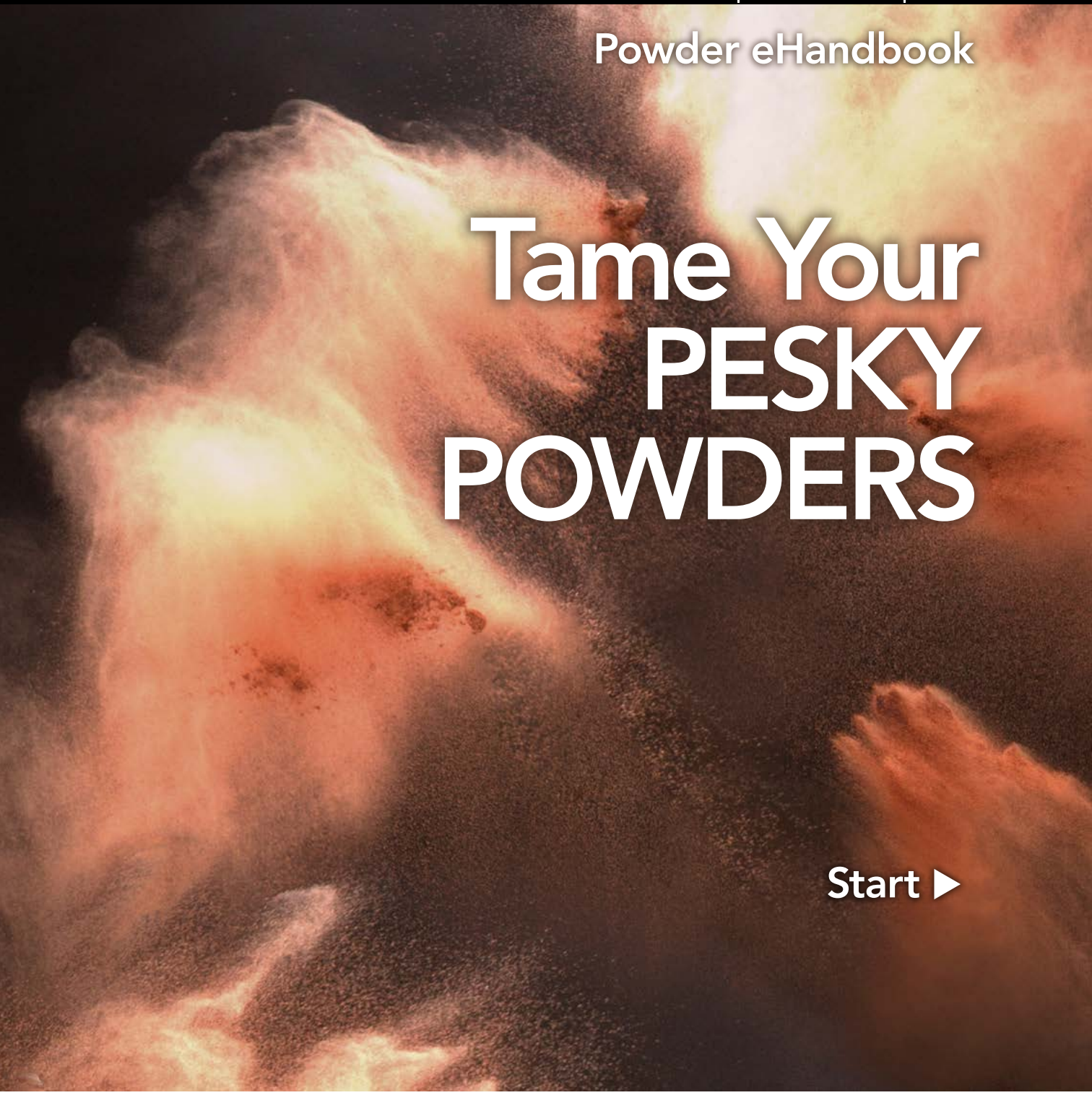

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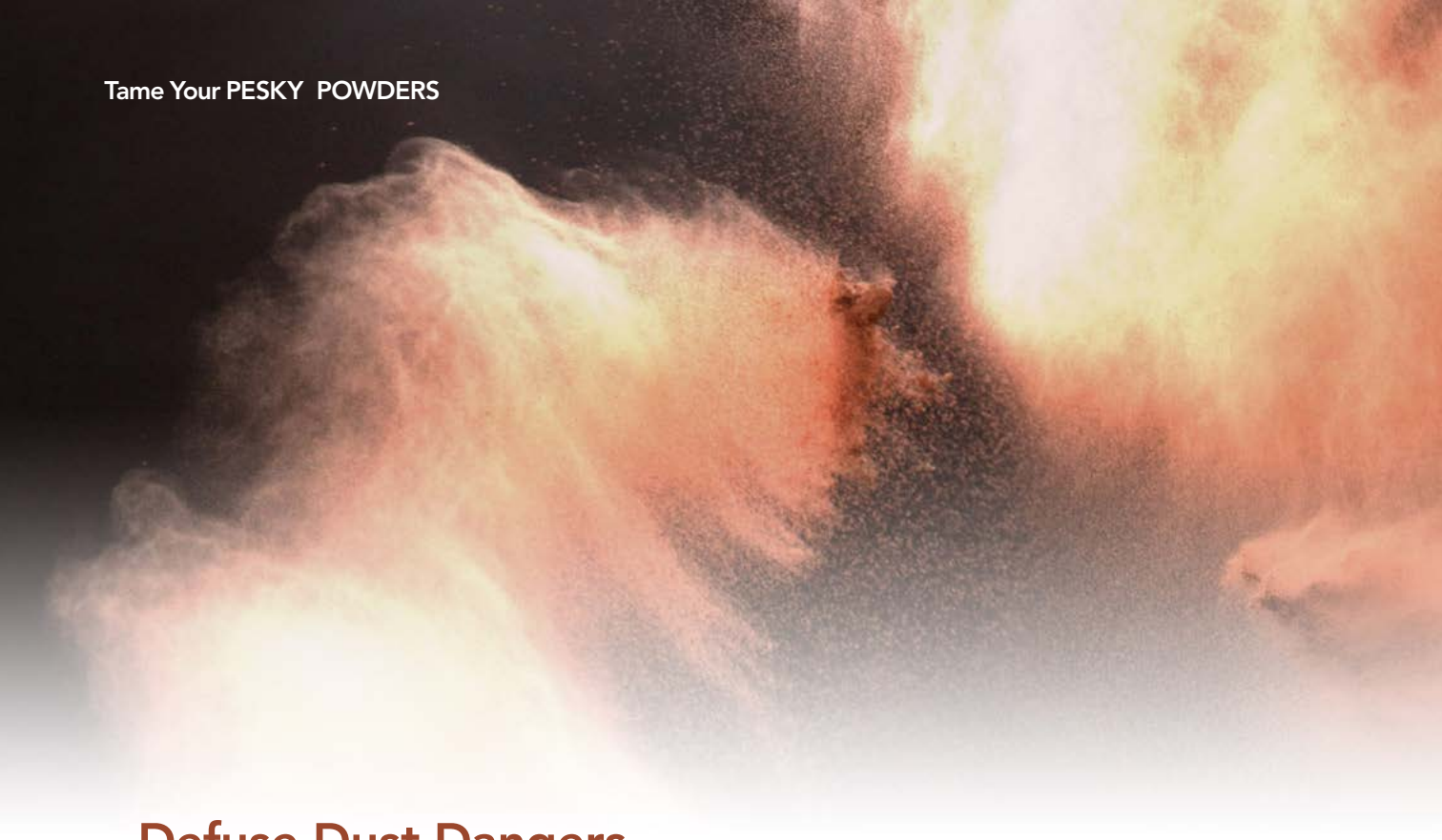

Defuse Dust Dangers

Carefully consider and then counter risks of fire and explosion

By Dirk Willard, Contributing Editor

WHEN THE West Fertilizer Company in West, Texas, blew up on April 17, 2013, killing 14 people, it must have taken Donald Adair, the owner of the plant, by surprise. In his 2011 emergency plan, Adair described the worst-case scenario for his plant as a 10-minute release of gas! Perhaps we chemical engineers don't appreciate the risks posed by dust as well as we do those of flammable fluids.

One way to bolster your understanding of dust's risks is to check out *Chemical Processing*'s free on-demand webinar "Dust Control: How to Identify & Manage Explosion Hazards," accessible via http://goo.gl/MRTKKM.

You must estimate the severity of the risk and then its probability. NFPA 499, "Recommended Practice for the Classification of Combustible Dusts and of Hazardous Locations for Electrical Installations in Chemical Process Areas," lists many compounds that produce combustible dusts. If yours isn't on the list, test according to ASTM E1226, "Standard Test Method for Explosibility of Dust Clouds."

In simplified terms, ASTM E1226 involves disturbing a small volume of dust with a pulse of air, followed, after a prescribed time delay, by ignition with a small electrical charge. The dust must contain < 5% moisture by weight and have particles smaller than 420 microns in diameter (i.e., ones that pass through a U.S. No. 40 standard sieve). The test takes place in a bomb of at least 20 liters at ranges of dust concentrations, fuel/air ratios, and electric charges. The goal of this test is to estimate the maximum pressure, the rate of pressure rise with time, and the dust deflagration index, K_{st}, a measure of relative explosive severity; these parameters also are useful in designing deflagration vents. OSHA defines a dust as a hazard if its K_{st} exceeds zero; this definition won't protect you if your process produces fines, especially those smaller than 15 microns, which easily are converted to an aerosol. A K_{st} between 0 and 200 (when measured in bar-meters/sec) indicates a weak explosive risk typical of sugar.

ASTM E1226 can pose several problems: measuring the dust density accurately; accounting for the pressure spike from the igniters; maintaining a dry sample; mixing issues affecting dust and air combustion; and, perhaps, comparisons between bombs of different volumes. So, get as much data as you can on dust properties, do more bomb runs, evaluate the equipment and procedure for systemic

faults, and compare your test data against a known standard.

With the severity estimated, it's time to consider the probability that a spark or heat could initiate a fire or explosion. Probability tests involve measurement of the minimum ignition energy (MIE), the minimum explosible concentration (MEC), the auto-ignition temperature (AIT), and the limiting O_2 content (LOC). Except for the AIT, tests are for dust clouds. ASTM E2019 covers measuring the MIE of a dust cloud; ASTM E1515 the MEC of a cloud; ASTM E1491 the AIT of a cloud; and ASTM E 2021 the AIT of layered dust. No LOC test is approved in the US; ASTM has WK41004 in the works but Europe has DIN EN 14034-4:2004.

The spark risk is measured in milliJoules (mJ). OSHA states that "materials that ignite above 0.50 joules (500 mJ) are not considered sensitive to ignition by electrostatic discharge." Between 500 and 100 mJ, equipment and people must be grounded to reduce the risk of ignition. An MIE less than 25 mJ is extremely hazardous, posing a risk during bulk operations, e.g., pneumatic conveying, silo storage, etc.

German data from 1965–1985 show that electrical discharge represents only 10% of the ignition sources in 426 accidents. Unfortunately, it's not as easy to assess the danger from heat. Fire caused by grinding or another physical action, drying or even self-heating represents the greatest potential, and is poorly understood. I couldn't find any correlation directly connecting MIE and fire risk; it's more of an article of faith that a low MIE is a fire risk.

So, let's move on to mitigation. Here're some ideas: 1) keep surface temperatures 170°F below the AIT; 2) avoid rubbing of rotating parts; 3) reduce rotating speed; 4) maintain strict grounding policy (see: "Move Against Static Electricity," http://goo.gl/CwHgBN; 5) measure and decrease available oxygen; and 6) cut the quantity of dust by good housekeeping. Also, check out "Dust Explosion Standard Gets Significant Revisions," http://goo.gl/V1CEQG, which highlights important revisions to that standard for prevention of fire and dust explosions.

Hopefully, by focusing on temperature as well as electricity we can avoid more surprises for plant managers.

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Clamp Down On Clumping

First understand what's really causing the problem

By Tom Blackwood, Healthsite Associates

POOR FLOW is one of the most common problems encountered in handling or storing solids. With liquids you open the valve and (hopefully) material runs out. With solids you often have to pray before opening the valve. Wouldn't it be nice to have an inexpensive well-established test method or procedure that would allow you to predict whether a fine powder will flow after a given time interval?

Often test methods take too long and generate results that are qualitative and very subjective. Also, material may be limited and quantitative test procedures are very expensive. If your plant has been working with solids you're probably familiar with this scenario. The basic issue is a balance of cost versus usable results. Clumping is a complicated issue that's difficult to quantify; so it's no surprise that finding a meaningful test is difficult. In most cases, clumping is unpredictable. However, a generalized procedure can be used to solve a clumping problem after it has occurred.

By clumping we really mean unintended agglomeration. While some types of agglomeration are desirable, e.g., to reduce dustiness or make a material easier to handle, most clumping isn't appreciated. The last thing you need in a pharmaceutical plant is for a bulk bag of acetaminophen to come in as a solid block (as has happened). Clumping is such a tricky issue due to its many causes. Before you can select a test method or procedure, you need to determine the root cause of clumping. Sometimes that can identify a solution without further testing.

CAUSES OF CLUMPING

The 10 most common sources of agglomeration in bulk solids are:

1. *Simple dissolution followed by drying of solids without any chemical reaction.* This is the fundamental problem with a lot of storage systems. Bags aren't sealed well enough or a solvent gets into the transport line. Several of the following causes also involve this process but in a roundabout way.
2. *Chemical reaction between particles and gases in voids in the bulk solid.* Most common is formation of hydrates, which changes particle density or creates bridges between particles. Oxidation or reduction of particles is less common but can release gases or yield condensable products that form a sticky film on the solids. Diffusion of soluble gases such as carbon dioxide can soften particles and make them susceptible to shear. In addition, particles can interact with wall material through abrasion, which can act as a catalyst to reduce the potential energy needed for a reaction to occur.
3. *Change of phase.* This is the most difficult problem to diagnose but often is easiest to prevent. Many people don't realize that a polymorph could be present. About one-third of all organics have at least one polymorph [1]; lots of

TESTING DEVICE

Figure 1. Distillation relies upon the differences in the boiling point or the vapor pressure versus temperature characteristics of substances to provide a mechanism for separation.

pharmaceuticals rely on chemicals that aren't in their most stable form. While transformation upon storage may take years a small amount of change can result in a big effect on flowability. For crystalline solids the problem usually starts with a very small amount of excess solvent and a temperature change. The unstable form dissolves and then recrystallizes into the stable form with solvent release. The process will repeat as solvent moves from particle to particle. A similar process can occur with amorphous organics because the crystalline form is at a lower potential energy.

4. *Recrystallization of solids during storage.* Often particles can pick up excess energy prior to storage though handling or milling operations. The latter is a very common culprit because attrition raises the surface potential energy of the solids and creates very fine solids, which have a much higher charge-to-mass ratio. It's rare for solid-state transformation to occur but it only takes a small amount of solvent to aid the crystallization process, similar to a polymorphic transformation.
5. *Viscous films on particles.* Interstitial solvent can prompt formation of such films. Heating or cooling solids can cause solvent to migrate and collect in one location. As the solvent partially evaporates, it leaves behind a sticky surface on the particle that can lead to bridging.
6. *Impurities in solids.* These can induce stresses in the particles, which can hasten chemical reaction, phase changes and recrystallization. Impurities can act as catalysts in a reaction or interact with wall materials to initiate one of the causes previously cited. Localized change in density due to an impurity can prevent normal transfer of shear force from particle to particle and put more stress on an individual particle, resulting in breakage. Location of the impurity — whether on the surface or interior of the particle — may even be critical and cause some batches of solids to behave much differently than others.
7. *Particle size and width of the particle size distribution (PSD).* These attributes often can't be changed but contribute to clumping. Finer particles have higher specific surface area and more particle/particle contact, resulting in higher shear forces. In addition, the van der Waals' forces increase rapidly with finer particles. The wider the PSD, the more likely voids around larger particles will fill in with fine particles and boost cohesion. While this is a major factor in clumping, it's very easy to identify in advance of a problem.
8. *Attrition.* This is more of a contributing factor for the previously mentioned sources of clumping. Breakage of particles releases energy that's confined to the solids' surface. In addition, finer solids will have poorer flowability and higher electrostatic charge. The increase in fines makes the PSD wider and solids easier to bridge.
9. *Mechanical deformation of solids.* This usually isn't the primary cause of agglomeration. The normal stresses in a bulk bag or fiber drum are fairly low. However, the ultimate formation of agglomerates often appears as a mechanical failure. Because a solid is defined as something that can support its own weight, most failures stem from shear forces that exceed the solid's strength. Many of the previous sources of clumping induce a failure that allows for compression of solids to form agglomerate.
10. *Vibration.* This often is overlooked as a cause of finer solids sifting into voids and increasing compression of bulk solids. When combined with temperature changes, vibration can make solids soften or plasticize, resulting in physical deformation and clumping. However, sometimes vibration can help to prevent mechanical deformation during transport.

As the above highlights, one major factor that repeatedly enters the equation is the presence of excess solvent or solvent vapor. If either afflicts solids, you must focus attention on the solvent source. Water is the most common solvent; moisture causes many clumping problems. Four major types of solvent in bulk solids contribute to clumping (see sidebar). No single test can detect all four; methods for determining the amount of solvent may not give a clear indication of the type.

USEFUL TESTS

Many bulk solids' tests focus on setting parameters for design of a bin or chute to keep product moving or to induce flow. Clumping is an afterthought of these methods. The tests can show how a material gains strength upon storage but can't predict outside of testing conditions (time or temperature) future increase in strength. In many cases the rise may be limited, at least to a solid block of solids. In addition, most methods require specialized and costly equipment, which is the major reason plants don't conduct such tests prior to experiencing a problem. However, these tests, by determining the time-dependent unconfined yield strength, are some of the best ways to determine when there'll be a problem.

The three major contenders for test equipment for this property are the Schultze (ASTM D6773) and Jenike (ASTM D6128) shear cells and the Johanson Indicizer. In addition, other devices have been developed for specialized industries. Some of these methods

CARR INDEX OF FLOWABILITY (ADAPTED FOR CLUMPING)*

Degree of Flowability	Flowability Index	Compressibility		Cohesion	
		%	Index	%	Index
Very Good	91–100	<5	25		
		6–9	24		
		10	22.5		
Fairly Good	81–90	11	22		
		12–14	21		
		15	20		
Good	71–80	16	19.5		
		17–19	18		
		20	17.5		
Normal	61–70	21	17		
		22–24	16		
		25	15	<6	15
Not Good	41–60	26	14.5	6–9	14.5
		27–30	12	10–29	12
		31	10	30	10
Bad	21–40	32	9.5	31	9.5
		33–36	7	32–54	7
		37	5	55	5
Very Bad	0–20	38	4.5	56	4.5
		39–45	2	57–79	2
		>45	0	>79	0

* Note: There are three conditions when the cohesion test should not be used:
1. Mean density (i.e., the average of tapped and aerated density from the Carr bulk density tests) is below 0.4 g/cm^3 and particles are greater than 150 µm;
2. Mean density is between 0.4 and 0.9 g/cm^3 and particles are greater than 75 µm; and
3. Mean density is above 0.9 g/cm^3 and particles are greater than 45 µm.

Table 1. The Flowability Index often can provide important insights.

can use samples as small as 20g — but several runs may be needed to account for product variability. These tests' major limitation is that, to be assured that material won't exceed a given strength in the future, trials must cover a wide range of temperature and humidity over the expected storage time. This may not be practical for solids kept in a bag or drum for many months and for replicating shipping conditions.

Carr [2-4] developed several tests in an attempt to determine the flowability and compressibility of bulk solids. His methods give indices that have had mixed reviews over the last 40 years. Many companies have devised their own internal methods and have published these for others to use — but they're often subjective and depend on the operator's scrutiny. Examples are observation of flow though different orifice sizes, lumping and compression as well as frangibility and friability tests.

TEN SOLUTIONS TO CLUMPING

	CAUSE	POTENTIAL SOLUTIONS
1	Simple dissolution	Shrink-wrapping pallets of bags can help. Choosing the correct liner or using multi-liner bags is another solution. Ensure that convey lines are dry and operators know how to properly store the material (i.e., good education on the product). Clamp down on procedures.
2	Chemical reaction	The material safety data sheet should provide an indication of potential reactivity of the solids. If not, this needs to be spelled out in the specification for the material. Consider potential chemical reactions before selecting a packaging method or container. If the material forms a hydrate, purge particles with dry gas prior to packaging. Also, don't package solids hot as this can speed up a reaction.
3	Change of phase	Know if the material has polymorphs. Molecular models can suggest and a DSC can verify that the material is the most stable form. Ensure there's no free solvent. In some cases a desiccant can be put into the container to pick up excess solvent. When an unstable material has to be packaged, use conditioned transport to minimize temperature changes.
4	Recrystallization	Avoid packing freshly milled solids unless there are no polymorphs — or minimize the intensity of the milling (i.e., use multiple millings rather than a single pass).
5	Viscous films	Avoid packaging solids when hot as this can drive solvent to voids in the top of the container where they can condense. Also, control humidity in the packaging area.
6	Impurities	These can be critical in promoting chemical reaction. They aren't a common source of clumping but can cause problems. When an impurity doesn't affect end use, improve product purity to remove it.
7	Particle size	This is the most difficult problem. When the customer wants a fine particle or a wide distribution, a problem is likely. Maybe a different specification can be used to make the particle larger and the distribution narrower.
8	Attrition	Design filling and handling equipment properly to limit attrition. Use an extended chute to minimize dropping distance. If the material is sensitive to attrition, use dense-phase conveyors.
9	Mechanical deformation	Consider not stacking bags or limiting the depth in a drum (use separators or sleeves). If a material has a poor initial strength, it's likely to deform and then clump. Design packages to limit the amount of compression by restricting the height of solids or inducing motion during transport.
10	Vibration	Vibration can work for or against clumping. Use specialized containers that limit transmission of motion to minimize the effect — or air-ride shipping methods.

Table 2. For each common cause of clumping, there's often a first line of defense.

Here's a highspotting of the pros and cons of the most common qualitative tests for evaluating clumping potential:

- *Carr Flowability Index* (ASTM-D6393) [5] — is quick and easy to do (simple equipment) and used extensively, gives a relative indication of how much a powder will compact and the strength of cohesive material, but can't provide quantitative results and isn't useful for time and temperature effects on solids;
- *BASF lumping and compression tests* (BASF Bulletin TPU 0402) [6] — uses small amount of material (10–15 g) to get qualitative results, but provides limited ability to study the effect of storage time and temperature on flow;
- *Flow though an orifice or down a surface* — offers qualitative results on how big an orifice needs to be for 50% of solids to flow out of a container (commercial instruments are available (Figure 1); however, many companies have constructed their own devices) or the necessary angle of slide (specific to wall material), but can't give the effect of storage time on flow; and
- *Frangibility* (sometimes called friability) tests — indicate relative strength of agglomerates, can be used to estimate crushing strength or how easily a clump can be broken, which may eliminate the need to solve a clumping problem,

STOP SOLVENT SNAGS

Solvents — gases as well as liquids — cause many clumping problems; understanding the nature of the solvent can be crucial in coming up with a solution. The solvents fall in four major categories (with the relevant one often related to how solids are dried):

1. *Free or surface.* These are easily removed by drying. However, in a production facility time may be limited or heat not uniformly distributed to solids. Most dryers are run based on contact time or exit temperature. While a longer time will give a dryer product that extra time may alter product color, taste or effectiveness.
2. *Bound.* Cohesive or electrostatic forces can cause physical or chemical adsorption of solvent onto particles. Some types of dryers are better at removing bound solvent but this can a difficult source to quantify.
3. *Inherent.* Solvent molecules trapped inside crevices of crystals or micropores of amorphous powders come into play when particles break. These molecules usually can't be removed by drying but you can minimize their effect, especially after a milling process. Solvents of hydration or crystallization are part of this group — but they're a stable form of the chemical that only would be released by a phase change or chemical reaction.
4. *Interstitial.* Vapor that fills voids in bulk material may total only a very small amount of solvent but can play an important role in caking mechanisms. When this source is identified as part of the problem it's fairly easy to fix by purging or fluidizing with dry gas.

Total solvent is the sum of all of the above.

In evaluating clumping it's important to know where solvent comes from so you can understand the clumping and propose the correct solution to the problem. There are eight common methods for determining the solvent in a particulate solid. Some are specific to the most common solvent, water. Each one can give a slightly different result because of the technique. The methods are:

1. *Karl Fischer* — for free and bound moisture (note other chemicals can be used to titrate solid for presence of other solvents);
2. *Loss on drying* (LOD) — for free or surface solvent and a major portion of bound solvent (for moisture, the test usually takes place at 90°C for 6 hours);
3. *Infrared* — for surface moisture, which is a close approximation to free moisture;
4. *Radio frequency* — for inherent (sometimes), bound and free moisture;
5. *Microwave* — for total and interstitial moisture (using different wavelengths);
6. *Loss on ignition* — for total solvent (can be done following LOD to get solvent of crystallization). Often this test is carried out in temperature steps to observe crystallization solvent as well as other volatile components or decomposition;
7. *Thermo-gravimetric analysis* — for total solvent loss with time (differential thermal analysis is more precise for solvent flux and can be combined with gas chromatography/mass spectroscopy to identify chemicals in a multi-component solvent system); and
8. *DSC* — for heat flow with time (which is especially useful in multi-component solvent systems).

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but use non-standard equipment (large balls are placed on the upper screen of a sieve stack and vibrated; amount of solids that pass to the pan is compared to the amount that goes to the pan without the balls).

In general, these tests will give an indication of the potential for clumping to occur upon storage when starting particulate solids are cohesive or flow poorly, i.e., Carr flowability index of less than 50 (see Table 1). However, initial good flowability doesn't predict lack of clumping upon storage. So, when facing a problem, it's important to look at the 10 most common sources of agglomeration to ascertain the likely culprit(s). In that respect the following techniques are useful in determining potential for phase change and can identify chemical components that are the cause of the problem:

- *Raman spectroscopy* — can find polymorphs;
- *Differential scanning calorimetry* (DSC) — can identify changes in structure and presence of unstable chemical forms or polymorphs;
- *Scanning electron microscopy* — can see formation of bridges (this can be combined with a probe to look at specific elements);
- *Transmission electron microscopy* — can detect bridge composition and impurities (but has limited application unless sample is very strong because it must be thin); and
- *Atomic force microscopy* — can observe real-time agglomeration (this technique is evolving rapidly and eventually may be able to see atomic-level changes).

CLUMPING SOLUTIONS

Is it possible to predict cohesion? Probably not. No one test is appropriate but many qualitative tests can help define the potential. So, what do you do to minimize clumping?

The solution to many prospective causes of clumping centers on minimizing solvent contact with solids, controlling particle size, limiting attrition sources and avoiding putting excess energy into solids, especially prior to packaging. Table 2 gives some specific suggestions for the ten common causes of clumping. For each there's often a first line of defense. However, many of the solutions can treat other related causes. For example, filling a drum from an excessive height can increase attrition, mechanical deformation and solvent vapor trapped (by boosting the amount of voids). Also, packaging a hot material can promote chemical reaction, phase change, recrystallization and formation of viscous films. As a last resort you can modify solids through agglomeration or addition of flow aids such as silica. But that's another subject.

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- Wall Friction
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- Arching Dimension
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What You Don't Know About Powder Flow

Shear cells can quickly reveal information about flow behavior

By Robert McGregor, Brookfield Engineering

THE NEW formulation is ready for scale up. The ingredients have been finalized. The blend comes out of the mixer and goes into a silo prior to final packaging. Part way through the bagging operation, outflow from the silo seems erratic. The blend appears to be changing in make up. There is no way to fix the problem without shutting down and investigating what is happening in the silo. Material appears to be hanging up around the outside while there is a clear open vertical path down through the center of the vessel. This is the sure sign of "funnel flow." The vertical path is referred to as a "rat hole" and is shown in Figure 1. This type of flow behavior can be responsible for desegregation of blended materials.

Should your process engineer have known that this was going to happen? Experienced plant personnel may be able to predict this type of occurrence based on similar startup situations in the past. But is there a flow test of some kind that gives you better information? A standard tool, like the flow cup, or a simple measurement, such as angle of repose, may give an idea that smooth continuous flow behavior is not likely, but cannot quantify the degree of difficulty that may be involved in getting powder out of the silo.

FLOW BEHAVIOR

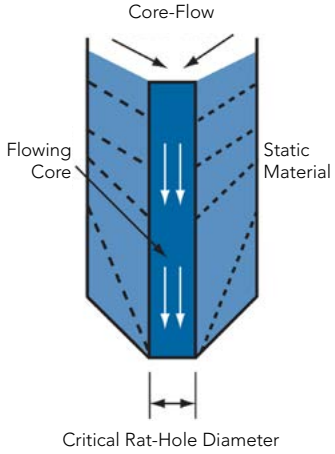

Figure 1. A "rat hole" formation in a silo can be responsible for desegregation of blended materials.

ANNULAR SHEAR CELL

Figure 2. Shear cells can help make predictive assessments because they simulate the forces that affect powder flow in a silo.

FLOW BEHAVIOR INSTRUMENT

Shear cells are becoming the tool of choice to make predictive assessments because they simulate the forces that affect powder flow in the silo. Figure 2 shows an "annular shear cell" that holds the powder sample. The "trough" contains the material and is weighed prior to the test so that density can be calculated during the test as the powder undergoes compaction. The "lid" is one of two types: one with pockets separated by vanes used to measure flow (Figure 3), the other with a smooth surface similar to the material of construction in the hopper wall (Figure 4).

The instrument applies pressure to the powder sample by bringing the lid into contact with the material in the Trough and pressing downward. The amount of pressure is controlled so that discreet

pressure levels are applied as the test progresses. The amount of pressure equates with increasing fill levels of powder in the silo. At each pressure level, the lid shears against the powder in the trough and the instrument measures the resistance to flow. The data gives a clear picture of how powder flow behavior can change as the fill level in the silo goes down during processing operation.

FLOW BEHAVIOR DATA

Graphs/tables of flow behavior data include flow function and wall friction information, which is used to mathematically calculate values for potential arching of powder over the hopper opening and the rat hole diameter in the silo described above. The software used to control the test with the shear cell automatically performs these calculations, so the test technician has only to load the sample in the trough and start the test.

One final piece of information that comes from the two tests is to calculate the hopper half angle that would be needed to achieve "mass flow" behavior in the silo. This would avoid the funnel-flow problem described at the beginning of the article. Mass flow allows the powder to flow uniformly down through the silo and discharge out the opening without formation of the rat hole.

This may sound simple and straightforward. It is. Current instrumentation for annular shear cells is fully automated so that operators worry only about sample prep. The data from each test is quickly analyzed for Arching Dimension, Rat Hole Diameter, and Hopper Half Angle (Figure 5). These numerical values allow the processor to decide whether to proceed with the equipment as is or make some modifications that will facilitate improved flow behavior during operation. The final point to note is that shear cells have become less expensive and can easily pay for the initial investment by preventing one or two stoppages during processing.

Perhaps it's time to investigate the shear cell and understand why it may be a better choice for predicting powder flow behavior.

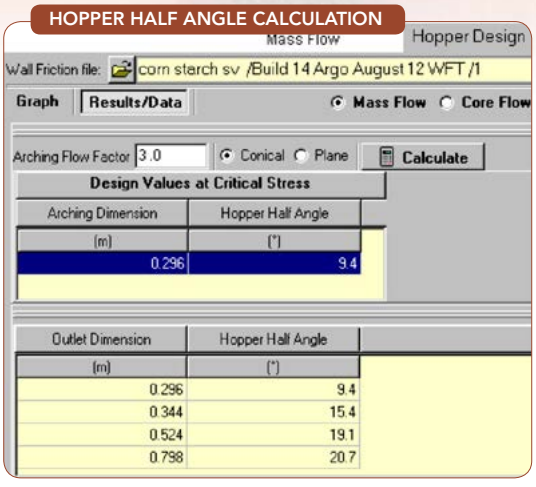

Figure 5. The annular shear cells are fully automated and data from each test can help quickly detect the hopper half angle and other flow measurements.

Figure 3. This shear cell's lid has pockets separated by vanes used to measure flow.

Figure 4. This lid in an annular shear cell has a smooth surface similar to the material of construction in the hopper wall.

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Know Your Vacuum Pumps

Understand the differences between liquid ring and rotary screw dry pumps for distillation

By Phil Vibert, Tuthill Vacuum & Blower Systems

DISTILLATION IS one of the many separation processes such as degassing, drying, filtration, membrane separation, adsorption, crystallization, etc. that rely upon the differences in the physical properties of substances in a mixture. Distillation relies upon the differences in the boiling point or the vapor pressure versus temperature characteristics of substances to provide a mechanism for separation. The different boiling points for of solvents can be seen in Figure 1.

Heating, evaporation and condensing then become the tools for separation of the liquid constituents in a liquid mixture. For separating substances with differences in boiling points of less than 30°C, a fractionating column with plates or packing is normally recommended to provide repeated condensing and re-evaporation of the reflux liquid as it progresses up the column for better separation of the constituents. The more volatile liquid will have a lower boiling point or higher vapor pressure versus temperature curve, and will be more readily evaporated. The vapor phase mixture will be richer in the more volatile compounds and then can be condensed, contained and returned for further separation and purification, if necessary. The greater in difference in the volatility of a component from the mixture the more easily it is separated.

Volatility of substance i is defined as Ki = yi/xi where Ki is the volatility of the i component and

BOILING POINTS OF SOLVENTS

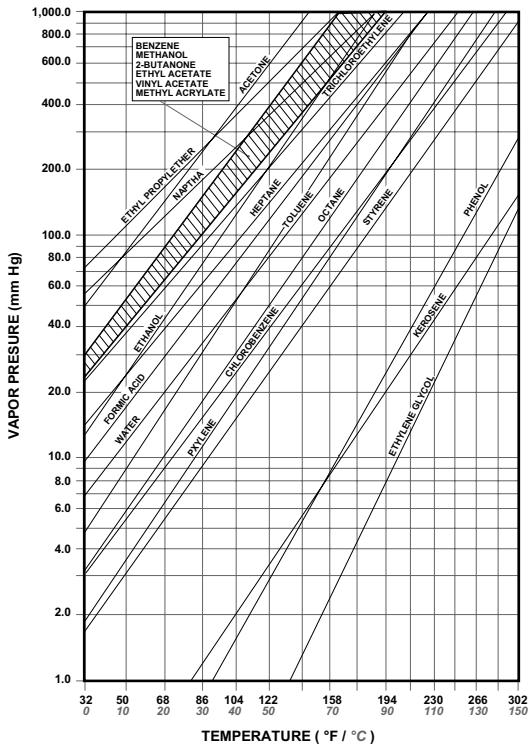

Figure 1. Distillation relies upon the differences in the boiling point or the vapor pressure versus temperature characteristics of substances to provide a mechanism for separation.

yi is the mole fraction of the i component in the vapor phase compared to the mole fraction of the i component in the liquid phase, xi. Because yiP = xiPvi, where P is the total pressure, yi and xi are the mole fraction in the vapor phase and liquid phase, respectively, and Pvi the pure component vapor pressure, then yi/xi = Pvi/P and for two substances the relative volatility a = K1/K2 = (y1/x1)/(y2/x2) = Pv1/Pv2 which is just the ratio of their pure component vapor pressures. For a simplified binary mixture that behaves as an ideal liquid, a phase diagram at constant pressure can be drawn with the mole fraction of the more volatile component on the horizontal axis and the temperature on the vertical axis. Vacuum distillation provides a convenient and efficient format for this separation at lower temperatures without harmful reactions with other gases such as oxygen.

For a simplified binary mixture that behaves as an ideal liquid, a phase diagram at constant pressure can be drawn with the mole fraction of the more volatile component on the horizontal axis and the temperature on the vertical axis. The lower curve is normally referred to as the bubble point where for a given mole fraction of liquid mixture, the liquid begins to boil at a given temperature. The higher curve is normally referred to as the dew point, which is the different temperatures where the different mole fractions of the vapor would start to condense. As an example, the phase diagram at constant pressure for a well-behaved binary mixture shows that the mixture will boil at 0.59T when the more volatile component 1 represents 0.3 mole fraction of the liquid mixture and will have a saturated vapor component y1 that represents almost 0.75 mole fraction of the entire vapor. This large difference between the vapor and liquid contribution of component 1 makes it easier to distill off (Figure 2).

In some cases, a mixture of two or more liquids at a given mole fraction of constituents will behave as a pure liquid where the vapor that boils off at a constant temperature has the same mole fraction in the vapor phase as in the liquid phase and no further separation of the constituents occurs. This is known as an azeotrope. For example, a mixture of ethanol and water will separate through simple distillation until the mole fraction of ethanol reaches 0.895 and no further change in concentration will occur. Some

PHASE DIAGRAM FOR BINARY DISTILLATION AT CONSTANT PRESSURE

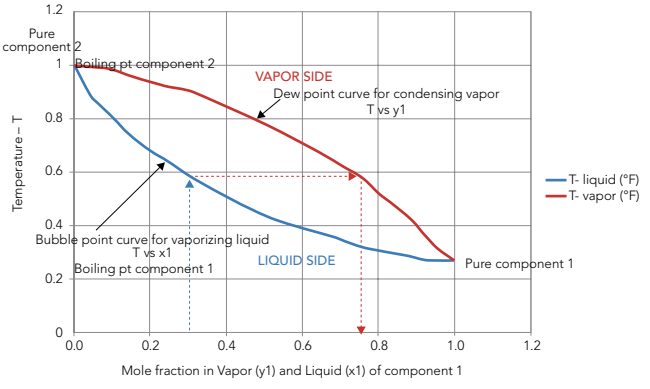

Figure 2. The large difference between the vapor and liquid contribution of component 1 makes it easier to distill off.

azeotropes can be separated by changing the pressure at which distillation occurs.

Vacuum distillation can help in some of these cases by providing a pressure variation for shifting the azeotrope to allow for further separation. The ethanol/water azeotrope disappears at distillation pressures below 70 mm Hg A. As in all processes the cost of further separation dictates its feasibility.

THE MOLECULAR DISTILLATION PROCESS

Molecular distillation is a similar process but occurring at much lower pressures (normally from 0.1 to 0.0005 mm Hg A) such that collision of the distillate molecules with the condenser predominate, compared to intermolecular collisions. The use of thin film distillation process using Wiped Film Stills (WFS) and Evaporators (WFE) provides a convenient method for separating out compounds for the chemical, food or pharmaceutical sectors, that have high boiling points, or high viscosity, or are sensitive to thermal degradation but are readily evaporated at modest temperatures at low pressures.

LIQUID RING VS. DRY VACUUM PUMPS

Condensers are used for knocking out most of the condensable vapors. But, for removing the permanent gases including air leakage along with the saturated vapors at the exhaust temperature of the vent condenser, for simple or fractional vacuum distillation, the most common and preferred pumps are the liquid ring and dry vacuum pumps. For lower pressure operation, a rotary lobe booster can be connected in series with either of these to provide higher pumping capacity at a lower pressure.

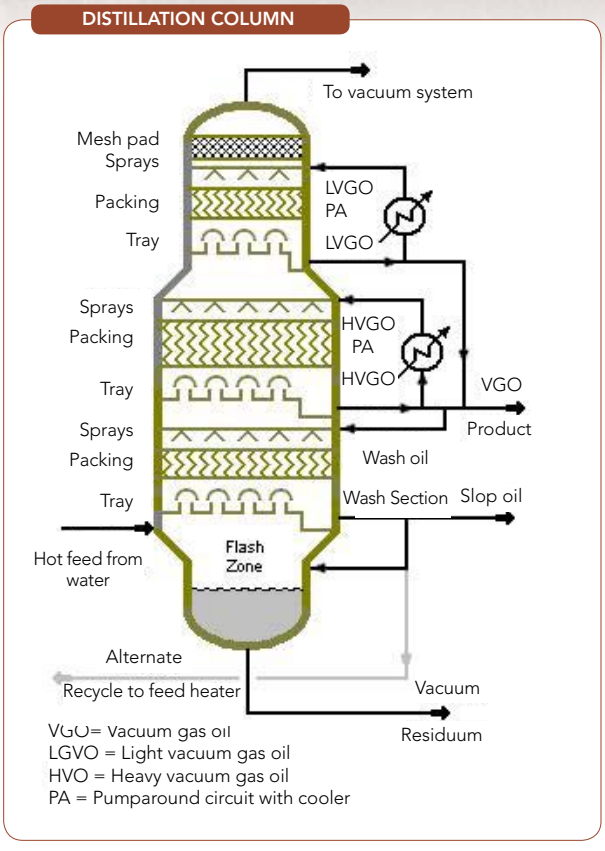

Figure 3. Vacuum distillation can help provide a pressure variation that allows for further separation.

The liquid ring pump (Figure 4) does not require internal lubrication and can run on most liquids such as water or low viscosity oil or many solvents that are compatible with its materials and the process in terms of vapor pressure and viscosity. It can handle liquid slugs from process upsets or a continuous flow of

LIQUID RING PUMP

Advantages	Disadvantages
Can perform as both vacuum pump and direct contact condenser	Normally higher operating cost than dry
Lower purchase price	Higher power and cooling water consumption
Simplicity of rotating parts improves reliability	Larger footprint
Low maintenance	Pump performance is limited by vapor pressure of sealant
Because of pump simplicity, can be readily disassembled and reassembled on site by end user	Requires a supply of liquid sealant for makeup or change out
Lower operating temperature for thermal sensitive or polymerizable process material	Operation normally results in larger amount of hazardous waste
Liquid sealant allows for handling higher temperature inlet gases/vapors	
Can ingest liquid from process or condensate from upstream condenser	
Less sensitive to process particulate due to larger clearances	
Liquid within pump may act as quench to reduce chance of ignition from sparking	

DRY PUMP

Advantages	Disadvantages
Lower ultimate pressure and higher capacity at low pressure end for single-stage pump	Higher purchase price
Lower power consumption	Higher complexity effects reliability
Lower cooling water usage	More difficult to disassemble and reassemble on site by end user
More compact footprint	Solvent handling limited by auto-ignition temperature of solvent
Can pump high vapor pressure solvents	Limited liquid ingestion
Environmentally friendly with less pollution	

liquid condensate from a pre-condenser. In some cases it can perform as both a vacuum pump for non-condensables and a direct contact condenser for vapors increasing its overall pumping capacity. It is one of the most reliable and durable mechanical pumps because of its simplistic design with one rotating shaft assemblage. It is also available in 316 stainless steel for greater corrosion resistance to process effluents.

The rotary screw dry pump (Figure 5) also does not require internal lubrication and can handle some liquid carryover, but as the name implies, it is preferred to keep the pump dry for optimum performance. Knockout pots would normally be recommended to trap out liquid slugs. Since the dry pump contains no liquid within its pumping chamber, it is not limited by the vapor pressure of the liquid and can achieve lower pressures without producing process contaminated waste products. The dry pump handles condensable vapors by keeping them in the vapor phase at an elevated temperature

DRY PUMP

Figure 5. The rotary screw dry pump doesn't require internal lubrication and can handle some liquid carryover, it's preferred to keep the pump dry for optimum performance.

while traveling from suction to discharge so that they can be condensed out in an after-condenser. The rotary screw dry pump and rotary lobe boosters are also available with optional protective coatings.

Because of the low pressure requirements for molecular distillation and reduced carryover, multi-stage booster packages utilizing either liquid ring, dry rotary screw, or oil sealed rotary piston vacuum pumps as the atmospheric stage can be provided.

Tuthill has been providing vacuum pumps and systems for vacuum distillation for decades and can work with the customer to provide recommendations and system designs to optimize performance.

The liquid ring and dry pump each have their advantages and disadvantages (See Tables on page 23).

LIQUID RING PUMP

Figure 4. This pump can handle liquid slugs from process upsets or a continuous flow of liquid condensate from a pre-condenser.

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