Designation: D 6941 – 03

Standard Practice for Measuring Fluidization Segregation Tendencies of Powders[1]

This standard is issued under the fixed designation D 6941; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers an apparatus and procedure for simulating the segregation tendencies of powders by means of the fluidization mechanism.

1.2 Powders must be capable of being fluidized in order to be tested by this practice.

1.3 Temperature- and humidity-sensitive powders may need to be tested at different temperatures and moisture contents, as would happen in an industrial environment. Further, the gas supply (type, temperature, and humidity) should also match the industrial conditions.

1.4 This standard is not applicable to all bulk solids and segregation mechanisms: while fluidization is a common segregation mechanism experienced by many fine powders, other segregation mechanisms not evaluated by this standard might induce segregation in practice.

1.5 The extent to which segregation will occur in an industrial situation is not only a function of the powder and its tendency to segregate, but also the handling equipment (for example, bin design), process (for example, transfer rates), and environment.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

3. Terminology

3.1 *Definitions*—Definitions of terms used in this test method shall be in accordance with Terminology D 653.

3.1.1 *fluidization*, *n*—the state in which a powder exhibits fluid-like properties.

3.1.2 *fluidization segregation*, *n*—a mechanism that causes vertical segregation, that is, horizontal layering of fine and coarse particles, as resulting from fluidization of the bulk solid.

3.1.3 *segregation*, *n*—a process through which blended or uniform powders or bulk solids become non-uniform, with regions of varying composition, for example, particle size.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *high flow-rate*, *n*—the first stage flow-rate used to initiate fluidization.

3.2.2 *hold time*, *n*—the time for which the Low Flow-rate is held.

3.2.3 *low flow-rate*, *n*—the second stage flow-rate used to maintain fluidization.

3.2.4 *ramp time*, *n*—the time during which the airflow is reduced from the Low Flow-rate to zero.

3.2.5 *representative sample*, *n*—a quantity of the bulk solid to be tested that is representative of that solid in an industrial application being studied. Parameters of interest that may affect whether or not a sample is representative include: moisture, particle size distribution, raw material variation, method of production, aging, chemical composition.

4. Summary of Practice

4.1 A representative sample of a powder is placed in the apparatus.

4.2 Pressurized gas (usually air) is blown from the bottom at a series of flow-rates for specified times, creating a state of fluidization of the powder.

4.3 Once the airflow is stopped, the powder in the test chamber is divided into three samples from the bottom, center, and top of the column.

4.4 The samples are then available to be tested for differences relevant to the application, for example, particle size or chemical assay.

5. Significance and Use

5.1 Fluidization segregation can cause vertical segregation within bins used to hold and transport powders. This can affect final product quality in industrial applications.

5.2 By measuring a powder's segregation tendency, one can compare results to other powders with known history, or determine if the given powder may have a tendency to segregate in a given process.

---

[1] This practice is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.24 on Characterization and Handling of Powders and Bulk Solids.

Current edition approved July 10, 2003. Published August 2003.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

5.3 Fine powders generally have a lower permeability than coarse bulk solids and therefore tend to retain air longer. Thus, when a bin is being filled with a fluidizable powder, the coarser particles settle or are driven into the bed while the finer particles remain fluidized near the surface.

5.4 Fluidization, which serves as a driving force for this mechanism of segregation, is likely to occur when fine powders are pneumatically conveyed into a bin, the bin is filled or discharged at high rates, or if sufficient air flow counter to the flow of powder is present within the bin.

6. Apparatus

6.1 The apparatus including critical dimensions is shown in Fig. 1. It consists of the following:

6.2 *Gas Supply with Flow Meter*—A gas supply capable of fluidizing the powder is required (15 to 30 psig [100 to 200 kPa] range, 25 psig [170 kPa] recommended, maximum flow rate 10 000 cm^3/min). The gas flow rate must be adjustable during the test-an automated controller may be used for this purpose.

NOTE 1—Generally, clean, dry air is used. If air is not suitable (that is, it reacts with or adversely affects the powder being tested) another gas, such as nitrogen, may be used.

6.3 *Cylinders*—Three transparent cylinders are stacked, identified (from the bottom) as the bottom, center, and top cylinders. The bottom cylinder sits against the diffuser in the air supply plenum. The top cylinder mates to the expansion chamber. When the cylinders are stacked together, they make up the test chamber, where the powder is placed. The assembled test chamber dimensions are 24 mm I.D. by at least 185 mm tall. The test chamber should have at least 25 mm additional height to allow expansion of the powder bed. The cylinders must be held together so they do not separate during the tests and so leakage does not occur, while still able to be separated at the end of the test in a way to allow for sample recovery. This can be done a number of ways, including taping the sections together.

6.4 *Expansion Chamber*—The expansion chamber allows the powder to disengage from the air stream.

6.5 *Filter*—The filter prevents powder from being blown out of the apparatus. The filter material should be appropriate for the application and should not contaminate the powder (which may affect the analysis of the samples), and should provide sufficient containment of the powder (from both a safety perspective and a loss of powder perspective).

6.6 *Diffuser*—The diffuser distributes the air uniformly into the test chamber; therefore, a sufficient pressure drop across the diffuser is required.

NOTE 2—A sintered metal disk, such as 5 µm filtration grade porous stainless steel sheet available from Mott Industrial, Farmington CT, may

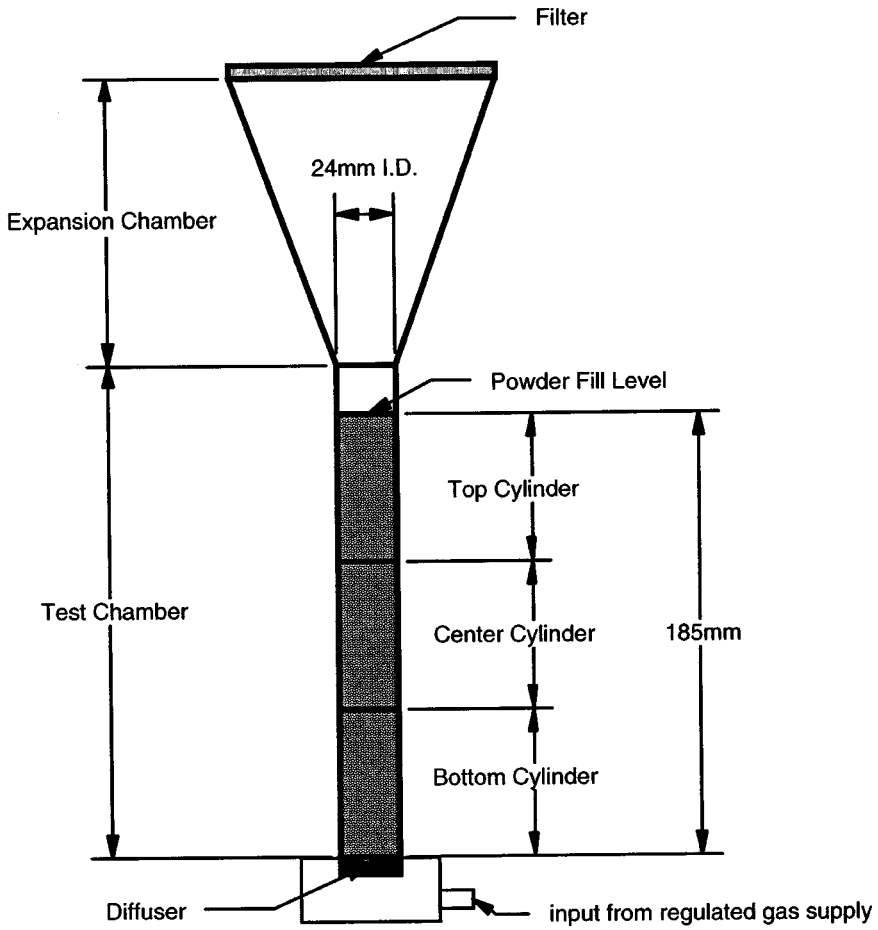

FIG. 1 Apparatus

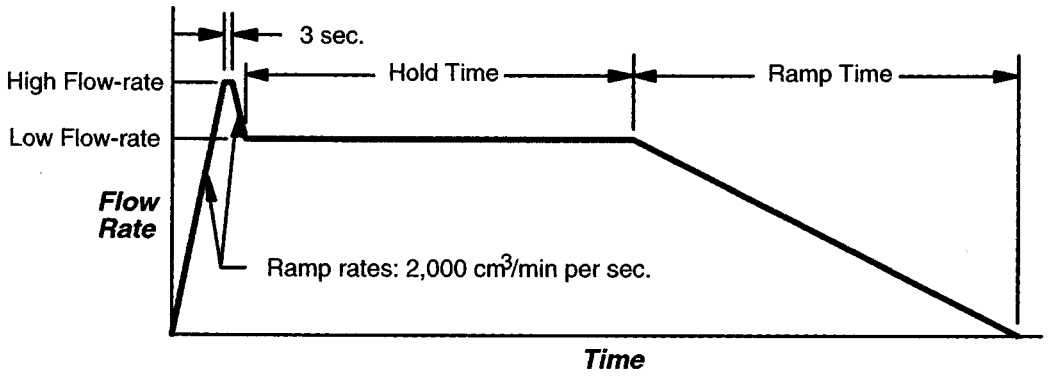

FIG. 2 Timing Profile

be an appropriate material for the diffuser. Some cohesive powders do not fluidize well, and simply form an air channel through the test bed, allowing the air to flow past stationary powder. In this case additional airflow will not serve to fluidize the powder. If this occurs, this test is not valid. However, a diffuser with a lower permeability may serve to distribute the air better thereby reducing channeling.

7. Procedure

7.1 Clean the apparatus and allow all parts to dry.

7.2 Stack the cylinders one above the other, secure them together, and align the bottom cylinder on the diffuser.

7.3 Place the apparatus on a table or bench that is free from vibration, in a suitable laboratory environment to approximate the industrial environment.

7.4 Obtain a representative, 100 mL sample of the powder to be tested.

7.5 Carefully spoon or scoop the powder into the test chamber. Fill the cylinder to a height of 185 mm.

7.6 Attach the expansion chamber and filter.

7.7 If the requesting agency has not specified values of High Flow-rate, Low Flow-rate, Hold Time, and Ramp Time for the powder to be tested, an initial test of the powder is required prior to running this test; See Annex A1, "Determining Inputs."

7.8 Start the airflow using the flow meter, and increase the airflow by 2000 cm^3/min per second to the High Flow-rate.

7.9 Hold the airflow at the High Flow-rate for 3 s.

7.10 Reduce the airflow by 2000 cm^3/min per second to the Low Flow-rate. Keep the airflow at this rate for the Hold Time to allow the powder to reach a steady state. For most applications, 120 s is appropriate to achieve this goal.

NOTE 3—To investigate the effect of Hold Time for particular powders, this test can be repeated with different Hold Times to determine the effect of Hold Time on segregation behavior.

7.11 Reduce the flow-rate to zero through the duration of the Ramp Time.

NOTE 4—The Ramp Time simulates the deaeration behavior in an actual industrial application. 30 s is appropriate for most applications. Varying the Hold or Ramp Times is not recommended without experimental evidence showing that the new times are more appropriate for the applications at hand.

7.12 At the end of the Ramp Time, gently tap the side of the expansion chamber to allow residual dust to fall back into the test chamber.

7.13 Allow additional time after stopping the airflow for the powder to settle completely.

NOTE 5—Settlement is considered complete when the top powder surface no longer appears to move (typically several minutes for fine powders).

7.14 Carefully separate the cylinders and place the entire contents of each cylinder into its own appropriate sample container.

8. Analysis of Samples

8.1 If needed, use appropriate sample splitting methods to reduce the size of the samples in each of the three cylinders to a suitable size for analysis. Use proper subdivision techniques, such as the use of a rotary riffler.

NOTE 6—Collecting sub-samples from the sample jars by scooping or thieving may be prone to errors. Analysis of multiple samples from a single location yields further confidence in the results.

8.2 Analyze the samples with respect to the parameters of interest: for example, particle size, particle shape, chemical assay, bulk density, color, solubility, or any other differences that may affect the suitability of the powder.

8.3 The trend from the top to the bottom of the tester is an indication of segregation potential. Typically, if fluidization segregation has occurred, the top cylinder is fines-rich, while the bottom is coarse-rich.

8.4 The difference between the top and bottom samples can be used as an indicator of segregation potential when a single-valued result is needed for comparison of different samples.

8.5 Segregation test results for a new powder should be compared to prior tests on other powders, whose segregation properties are well known and understood.

9. Report

9.1 Report the following information:

9.1.1 Date test was run,

9.1.2 Operator,

9.1.3 Name of project or client including project number if used,

9.1.4 Generic name of powder tested,

9.1.5 Temperature and relative humidity of room where tests performed,

9.1.6 High Flow-rate,
9.1.7 Low Flow-rate,
9.1.8 Hold Time,
9.1.9 Ramp Time, and
9.1.10 Any observations of interest during running of tests, including indications of poor fluidization, such as channeling or lifting of a solid plug of the powder, and the need for tapping to break up a mass of powder to promote fluidization.

10. Keywords

10.1 fluidization; powder; segregation

ANNEX

(Mandatory Information)

A1. DETERMINING INPUTS

A1.1 Determining Flow Rates and Times

A1.1.1 The goal of the fluidization segregation test is to bring the powder to a completely fluidized state, then allow slow deaeration (settlement) of the powder. For this to occur a specific flow-rate/time profile is used. See Fig. 2 for the general flow-rate/time profile.

A1.1.2 To determine the proper High Flow-rate, slowly increase the flow-rate until all of the powder is in a state of fluidization.

NOTE A1.1—This state is characterized by observing bubbles or turbulence within the bottom cylinder. When starting with a deaerated bed of powder in the test chamber, as the air begins to flow, initially the bed will remain stationary. As the airflow increases, the bed will begin to expand. This expansion behavior is highly powder dependent-in some cases the top surface may start to "bubble," while with cohesive powders the entire bed may lift as a plug. As the flow-rate increases further, eventually, all of the powder within the test chamber should become fluidized. In many cases, once the powder becomes fluidized, the flow-rate can be decreased while keeping the powder fluidized.

A1.1.3 To determine the proper Low Flow-rate, immediately after determining the High Flow-rate, slowly reduce the flow-rate until powder movement in the tester stops, then increase the rate again just to the point where motion is initiated within the top cylinder. This point is the Low Flow-rate.

NOTE A1.2—The second stage of the fluidization segregation test is to reduce and hold the airflow at a Low Flow-rate corresponding to the minimum fluidization velocity. This stage is characterized by observing some turbulence or bubbling in the top cylinder. Under these conditions, powder movement might not occur in the center or bottom test chambers.

A1.1.4 Upon completion of A1.1.2 and A1.1.3, empty the tester of the powder, and refill the tester with fresh powder for the actual test.

A1.2 Determining Inputs for Multiple Powders

A1.2.1 Often times the goal of these tests is to compare one powder to another. Variations in the powder may result in different fluidization behaviors. However, if the differences in the powders are small, usually the same rates can be used for each powder. Use the same flow-rates and times when comparing slight variations on one powder. In running each test, the test chamber should be observed to ensure that at the High Flow-rate, all the powder has become fluidized, while at the Low Flow-rate the top surface still shows signs of movement.

A1.2.2 Vastly different powders require their own flow-rates. However, the same Hold and Ramp Times should be used for each powder.