Designation: D6941 – 12

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

This standard is issued under the fixed designation D6941; 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 creating several specimens of a powder sample that, if the powder is one that segregates by the fluidization mechanism, should be different from one another.

1.2 A powder sample is fluidized then, after the fluidizing gas is turned off, it is separated into three or more specimens that can be analyzed for parameters of interest. The difference in these parameters between the specimens is an indication of the segregation potential of the powder.

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

1.4 Temperature- and moisture-sensitive powders may need to be tested at different temperatures and moisture contents, as would happen in an industrial environment.

1.5 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. Practice D6940 covers another common mechanism: sifting.

1.6 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.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word Standard in the title of this document means only that the document has been approved through the ASTM consensus process.

1.9 *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:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D6940 Practice for Measuring Sifting Segregation Tendencies of Bulk Solids

3. Terminology

3.1 *Definitions:*

3.1.1 For common definitions of technical terms in this standard, refer to Terminology D653.

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

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

3.1.4 *segregation, n—in powders*, 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:*

[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.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard**

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

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

3.2.3 *representative sample, n—in powders*, 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, but are not limited to: 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 test chamber.

4.2 Pressurized gas (usually air), which is blown from the bottom, is ramped up from zero to a pre-determined High Flow-rate, held there, ramped down to a pre-determined Low Flow-rate, then held there, all for specified times, creating a state of fluidization of the powder.

4.3 The airflow is ramped down to zero over a specified time.

4.4 The powder in the test chamber is divided into N specimens.

4.5 The specimens 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 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.

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 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.

NOTE 1—The quality of the result produced by this practice is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this practice are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

Practice D3740 was developed for agencies engaged in the testing and/or inspection of soil and rock. As such it is not totally applicable to agencies performing this practice. However, users of this practice should recognize that the framework of Practice D3740 is appropriate for evaluating the quality of an agency performing this practice. Currently there is no known qualifying national authority that inspects agencies that perform this practice.

6. Apparatus

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

6.2 *Gas Supply with Flow Meter*—A supply of dry, non-toxic and non-flammable gas capable of fluidizing the powder

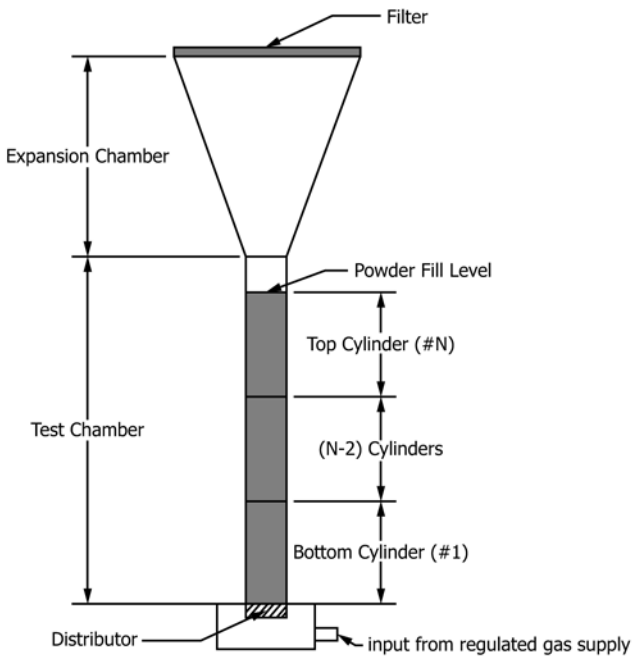

FIG. 1 Apparatus

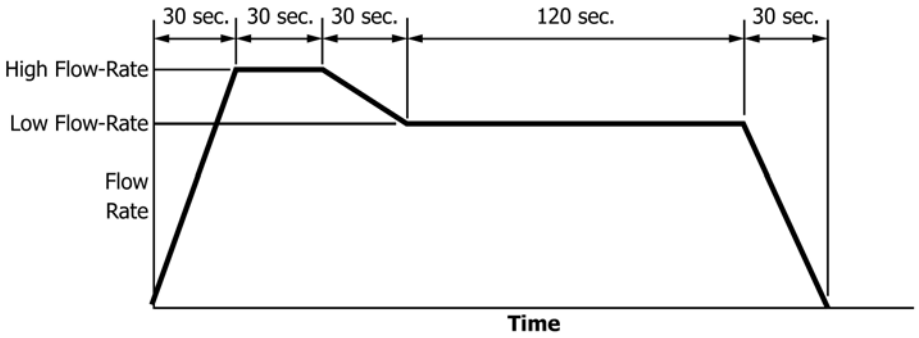

FIG. 2 Timing Profile

is required. The recommended gas supply pressure is 170 kPa [25 psig] with a range from 100 to 200 kPa [15 to 30 psig]. The recommended gas supply flow rate is 10 000 cm^3/min or higher. The gas flow rate shall be adjustable during the test. An automated controller may be used for this purpose.

NOTE 2—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 *Test Chamber*—This consists of three or more cylinders stacked above the distributor, which is at the top of the air supply plenum. These cylinders which, when stacked together have a total height of at least 195 mm, are connected at the top to a conical expansion chamber. The test chamber is where the powder sample is placed.

6.3.1 *Cylinders*—Each circular cylinder is transparent and has a diameter of 24 ± 1 mm I.D. They are stacked, identified as #1 (the bottom) to #N (the top). The bottom cylinder (#1) sits against the distributor in the air supply plenum. The top cylinder (#N) mates to the expansion chamber. The cylinders shall be held together in a manner that prevents separation during the test and prevents leakage. This method must allow separation of the cylinders upon completion of the test to allow specimen recovery.

6.4 *Expansion Chamber*—This conical extension of the test chamber allows the powder to disengage from the air stream. It must be sufficiently tall to prevent the expanded powder bed from contacting the filter at its top and sufficiently steep to cause the powder to slide back into the test chamber when the airflow is turned off.

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

6.6 *Distributor*—The distributor diffuses the air uniformly into the test chamber; therefore, a sufficient pressure drop across the distributor is required. The pore size of the porous media in the distributor does not have to be smaller than the smallest particle size of the test sample, but should be small enough to prevent particle seepage into the plenum.

NOTE 3—A sintered metal disk, such as 5 µm filtration grade porous stainless steel sheet, may be an appropriate material for the distributor. 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 distributor with a lower permeability may serve to distribute the air more uniformly, 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 (#1) on the distributor.

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. Make sure that the apparatus is level.

7.4 Obtain a representative, about 100 mL sample of the powder to be tested. Measure its temperature and, if appropriate, its moisture (water) content using Test Method D2216 or another method specified by the requesting agency.

7.5 Carefully spoon or scoop the powder into the test chamber. Fill it to a height of 170 ± 5 mm.

7.6 Attach the expansion chamber and filter.

7.7 If the requesting agency has not specified values of High Flow-rate and Low Flow-rate 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 uniformly ramp up the airflow over 30 ± 3 s to the High Flow-rate.

7.9 Hold the airflow at the High Flow-rate for 30 ± 3 s.

7.10 Uniformly ramp down the airflow over 30 ± 3 s to the Low Flow-rate. Keep the airflow at this rate for 120 ± 3 s.

7.11 Uniformly ramp down the flow-rate to zero over 30 ± 3 s.

7.12 Once the flow rate is reduced to zero, 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 4—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 specimen container. This is accomplished by separating the top specimen from the others first, and working downwards to the bottom specimen.

8. Analysis of Specimens

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

NOTE 5—Collecting sub-specimens from the specimen containers by scooping or thieving may be prone to errors. Analysis of multiple sub-specimens from a single location yields further confidence in the results.

8.2 Analyze the specimens or sub-specimens with respect to the parameters of interest: 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 specimens divided by the mean composition can be used as an indicator of segregation potential when a single-valued result is needed for comparison of different specimens.

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 Record as a minimum the following general information:

9.1.1 Requesting agency or client and/or identifying number for job or project,

9.1.2 Technician, and

9.1.3 Date test was run.

9.2 Record the following sample information:

9.2.1 Generic name of powder tested,

9.2.2 Chemical name of sample, if known,

9.2.3 Specimen moisture (water) content, if determined. Record value to nearest 0.1 %. Indicate method used to determine moisture if not Test Method D2216, and

9.2.4 Temperature of sample.

9.3 Record the following apparatus information:

9.3.1 Make and model of apparatus,

9.3.2 Material of construction of cylinders, how many, and height of each,

9.3.3 Gas composition, and

9.3.4 Maximum gas supply flow rate.

9.4 Record the following test data:

9.4.1 High Flow-rate,

9.4.2 Low Flow-rate,

9.4.3 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,

9.4.4 Results of analysis of specimens or sub-specimens such as particle size, particle shape, chemical assay, bulk density, color, solubility, or any other differences that may affect the suitability of the powder, and

9.4.5 Segregation trend from the top to the bottom of the test chamber.

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. Tapping the side of the test chamber may be necessary to break up a mass of powder to promote fluidization. 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.

A1.1.5 Record High Flow-rate and Low Flow-rate within ±1% of maximum gas supply flow rate.

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. Therefore, even with slight variations on one powder the flow rates may be different. 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.

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D6941 – 05$^{\varepsilon 1}$) that may impact the use of this standard.

(1) Sections 1.1, 4.2, 4.3, 6.3 and 7.4 revised, and Sections 1.2, 1.7 and 1.8 added.
(2) In most instances, "sample" replaced by "specimen".
(3) Reference to D2216 and D6940 added.
(4) Number of cylinder sections changed from three to three or more.
(5) Cylinder fill height changed from 185 to 170 mm.
(6) "Diffuser" changed to "Distributor".
(7) Example of indicator of segregation potential in 8.4 redefined.
(8) Section 9 text revised.
(9) Dimensions removed from Fig. 1.
(10) Section A1.1.5 added.
(11) Section A1.2.1 text revised.

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