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Standard Guide for Selection of Substitute, Non-hazardous, Particulate Solid Filling Substances for Packagings Subjected to the United Nations Performance Tests¹

This standard is issued under the fixed designation D8135; 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 The purpose of this guide is to clarify the selection, use, and description criteria of non-hazardous particulate solid substitutes used to replace hazardous materials for the purpose of performance testing packagings. This includes identification of the physical parameters of substitute non-hazardous solid test fill materials that may affect packaging performance and test results and should be considered when selecting and describing a test fill material that conforms to the requirements of the Hazardous Materials Regulations (HMR). This guide is intended for use with package testing procedures for particulate solid materials that have flow characteristics. It is not intended for use with hazardous articles.

1.2 This guide provides information to assist packaging users, manufacturers, and performance testing service suppliers regarding the types of physical properties that should be considered when selecting substitute filling substances for the testing, certification and manufacture of packagings under the United Nations packaging protocols (UN Recommendations on the Transport of Dangerous Goods-Model Regulations) as adopted by US DOT in 49 CFR HMR.

1.3 This guide provides the suggested minimum information concerning the physical characteristics of the filling substances that should be documented in the certification test report and notification to users to allow for test repeatability and analysis, and to provide guidance to the user of a packaging of pertinent physical differences between potential hazardous lading and the filling substance with which the packaging was tested.

1.4 This guide does not purport to address regulatory requirements regarding the compatibility of filling substances with transport packagings. Compatibility requirements must be assessed separately, but it should be noted that under certain national and international dangerous goods regulations, the

selection of the filling substances for package performance testing may be prescribed with respect to chemical compatibility requirements.

NOTE 1—Under the US HMR determination of packaging compatibility with a particular hazardous fill material is “the responsibility of the person offering the hazardous material for transportation” as prescribed in 49 CFR § 173.24(e).

1.5 When testing packaging designs intended for hazardous materials (dangerous goods), the user of this guide shall be trained in accordance with 49 CFR §172.700 and other applicable hazardous materials regulations such as the ICAO Technical Instructions, IMDG Code, other applicable national or international dangerous goods regulations that govern the testing, manufacture and use of packagings authorized for the transportation of dangerous goods, and carrier rules such as the IATA Dangerous Goods Regulations.

1.6 The units of measurement are consistent with the HMR.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D4919 Guide for Testing of Hazardous Materials (Dangerous Goods) Packagings

D6393 Test Method for Bulk Solids Characterization by Carr Indices

¹ This guide is under the jurisdiction of ASTM Committee D10 on Packaging and is the direct responsibility of Subcommittee D10.22 on Hazardous Materials.

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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.

D7481 Test Methods for Determining Loose and Tapped Bulk Densities of Powders using a Graduated Cylinder
 E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

2.2 Federal Standard:³

U.S. Department of Transportation Code of Federal Regulations Title 49, Transportation (49 CFR) Parts 100-199

2.3 UN Standard:⁴

United Nations Recommendations on the Transport of Dangerous Goods, Model Regulations (UN Orange Book)

2.4 International Air Transport Association (IATA) Standard:⁵

International Air Transport Association (IATA) Dangerous Goods Regulations

2.5 ICAO Standard:⁶

International Civil Aviation Organization (ICAO) Technical Instructions for the Safe Transport of Dangerous Goods by Air

2.6 IMDG Standard:⁷

International Maritime Dangerous Goods Code (IMDG Code)

2.7 ISO Standards:⁸

ISO 2234 Packaging – Complete, Filled Transport Packages – Stacking Tests using Static Load

ISO 16495 Packaging – Transport Packaging for Dangerous Goods – Test Methods

3. Terminology

3.1 Definitions:

3.1.1 *angle of repose, n*—when particulate solids are poured onto a flat, horizontal surface, they form a conical pile. The angle of repose is the interior angle formed between the base of this cone and its side (see Fig. 1).

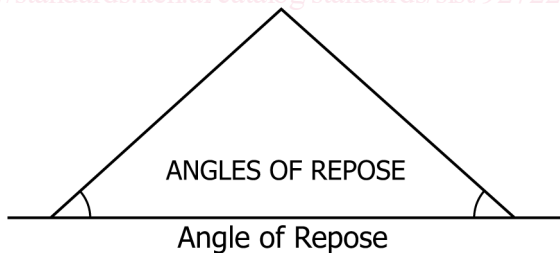


FIG. 1 Angle of Repose

³ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-9371 (website: <http://phmsa.dot.gov/hazmat>).

⁴ Available from the UN Economic Commission for Europe, Information Service, Palais des Nations, CH-1211 Geneva 10 Switzerland (website: <http://www.unecce.org/trans/danger/danger.htm>).

⁵ Available from the International Air Transport Association (IATA), 800 Place Victoria PO Box 113 Montreal - H4Z 1M1 Quebec - Canada (website: <http://www.iata.org>).

⁶ Available from the International Civil Aviation Organization, (ICAO) 999 University Street, Montréal, Quebec H3C 5H7, Canada (website: <http://www.icao.org>).

⁷ Available from the International Marine Organization (IMO), 4 Albert Embankment, London, SE1 7SR United Kingdom (website: <http://www.imo.org/>).

⁸ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

3.1.2 *bulk density, n*—the mass (weight) of particulate solids divided by the total volume of sample; that is, bulk density is the ratio of the mass of large number particles of the material to the total volume of the sample and is an aggregate property that includes the volume(s) of the particles, inter-particle interstices and particle porosity. It is expressed as a ratio of weight/volume, or may be expressed as a unit-less ratio referenced to a standard material, such as water, as a specific gravity.

3.1.2.1 *Discussion*—It is not a characteristic intrinsic to a material. It is affected by how the sample is handled. Commonly expressed as “freely settled” or “poured” density; in distinction to a “tapped” density value that would be determined by a compaction method such as tapping a sample on a table during filling to represent compaction due to various types of vibration. For this guide, poured density would be the most applicable.

3.1.3 *particle size distribution, n*—the physical size of the individual particles in a given sample of a particulate solid. In almost all common solids this will be expressed as a particle size distribution. There are many ways to model or measure this distribution given the wide variety and irregularity of particle geometries and styles.

3.1.3.1 *Discussion*—The most effective and common way to measure this property is by sieve analysis. Where the particle size distribution is expressed as a percentage of the weight of the sample, retained in each step as the sample is passed through progressively finer sieves, to the weight of the whole sample. See Specification E11 and Table X1.2.

3.1.4 *specification packaging, n*—a packaging conforming to one of the specifications or standards for packagings in part 178 or part 179 of this subchapter (49 CFR).

3.1.4.1 *Discussion*—Packaging, as used in this guide, includes packaging constructed and tested to UN performance standards as adopted in the HMR (the focus of this guide) but does not exclude its use for other types of packagings.

NOTE 2—Additional terms and definitions are located in 49 CFR sections 171.8 and the UN Orange Book, Section 1.2.1.

4. Summary of Guide

4.1 The manufacturer, test facility or user of a packaging will apply the procedural considerations, based on the physical characteristics of the particulate solid hazardous material to be transported, outlined in this guide to select an appropriate fill material to use when testing a packaging. In addition, packaging manufacturers and test facilities will use this guide to provide sufficient information concerning the relevant physical characteristics of the test fill material to the user of a packaging to allow for a proper evaluation of suitability of a packaging for a particular particulate solid hazardous material.

5. Significance and Use

5.1 Regulations prescribing the test procedures for hazardous materials packaging allow for the substitution of non-hazardous fill materials for packaging performance tests with certain limitations prescribed and guidance offered (see 49 CFR 178.602(c)). This regulatory guidance has proven to be flexible enough, in common industry practice, to produce

variations in the selection of fill materials for package performance tests sufficient to cause inconsistent and non-repeatable test results. This variation creates significant problems in product liability, packaging selection and regulatory enforcement in this highly regulated industry. Use of this guide should enhance uniformity in test procedures.

5.2 Consistent and repeatable test results coupled with clear test fill product descriptions will enhance transportation safety by simplifying packaging selection. This will also increase the general level of confidence that package testing, manufacture, and use are being guided by sound, generally accepted engineering principles. It also aids in clarifying expectations between the packaging industry and the regulatory authorities.

5.3 The guide will be used by packaging manufacturers and packaging test labs to create packaging test plans that meet customer needs and conform to the HMR. In addition, for the user of a packaging, certain information about the type and physical characteristics of the material used to test the packaging must be available in the test report and/or notification instruction to allow evaluation of whether a particular packaging was tested with a substitute material appropriate for the hazardous material to be shipped.

6. Procedure

6.1 Packagings may be tested when filled with the actual product to be transported to the required minimum fill levels. This option is often completely impracticable. Personnel safety and training, testing facility chemical handling capability, possible site contamination, local regulations or zoning are among the strongest reasons to use non-hazardous substitute fill materials for package testing.

6.2 A non-hazardous particulate solid may be substituted as a fill material for testing purposes as prescribed in 49 CFR § 178.602(c): “If the hazardous material to be transported is replaced for test purposes by a non-hazardous material, the material used must be of the same or higher specific gravity as the material to be carried, and its other physical properties (grain, size, viscosity) which might influence the results of the required tests must correspond as closely as possible to those of the hazardous material to be transported.”

6.2.1 In the case of particulate solids the “specific gravity” mentioned in the regulatory citation above often will be expressed as a bulk density value (g/cm^3 or lb/ft^3). These values may be readily converted into a unit-less ratio as a specific gravity, usually using water as the reference material (water density $1.0 \text{ g}/\text{cm}^3$).

6.2.2 Other characteristics of fill materials that may influence test results and should be evaluated when selecting a substitute material:

6.2.2.1 *Particle (Grain) Size and Type*—Grain affects both flow properties and the ability to detect leakage from the package during testing. Smaller grain, freely flowing particulate solids are more readily detectable from potential leak sites after tests are performed.

6.2.2.2 *Flow Properties*—For particulate solids flow is a complex property affected by particle size, shape, surface texture, etc. As the flow rate of a particulate solid increases,

impact and vibratory energy (effects) are more readily transferred to the packaging.

(1) For package testing purposes, much of the complexity of flow can be effectively reduced to a single and fairly straightforward physical characteristic of a bulk solid known as angle of repose.

(a) When particulate solids are poured onto a surface a conical pile forms. As more material is added the pile becomes taller and increases in diameter at a ratio that keeps the angle between the flat surface and the side of the pile near constant.

(b) There are a variety of methods used to determine the angle of repose for a material, the easiest and most common being the funnel method. The material is poured through a funnel onto a flat surface. The tip of the funnel should be held close to the top of the cone as it grows to minimize the effect of impact energy from the particles on the pile. The pile can be formed to a predetermined height, width of base or a given mass of material. (In Fig. 2, the base diameter is the selected limit by use of a pedestal structure.) The angle of repose can be measured directly from the pile using a protractor, or it can be computed from the relationship between the diameter (or radius) of the pile and its height. The angle of repose would be computed by taking the inverse Tangent of the ratio of the height and the radius ($1/2$ diameter) of the base. There are many relatively simple and inexpensive funnel type testers available in the market.

(c) It is important to note that some external conditions can produce large effects on the flow properties of a particulate solid: humidity, temperature, electro-static charge and flow-rate among them. Control for these variables should be addressed when determining the angle of repose.

(d) For many common materials the AoR is known and may be available as a purchase specification under defined conditions.

(e) ASTM has a document to provide guidance on determining the physical characteristics of solid materials including angle of repose: Test Method D6393. This guidance contains a much more sophisticated method of determining the Carr Angle of Repose in Test Method A.



FIG. 2 Apparatus to Determine Angle of Repose

6.2.2.3 Due to the complexity of matching product densities and required package fill weights with the regulatory requirement in 49 CFR 178.602(b) for the package to be filled to a minimum of 95 % of its maximum capacity, it is often necessary to mix or insert materials with different physical characteristics than the chosen particulate test fill to either raise or lower the net weight of the fill material for the tests. It is important that this be done in a manner that minimizes the effects of a heterogeneous fill on the package tests.

NOTE 3—Chosen filling method should take into consideration the settling of the fill substance or mixture to ensure the volumetric fill continues to meet the minimum 95 % of the maximum capacity.

(1) The augmenting material may be added near the center of mass of the package allowing for the free flowing particulate material to surround it. This will maintain similar impact dynamics by keeping the walls and closure of the package in contact with the fill material.

(a) Examples include shot bags, metal articles, sand bags or other articles intended to add weight to the package. Also, packing peanuts, bubble wrap or air pillows in cases where package weight needs to be reduced while maintaining the minimum volumetric fill.

(2) The augmenting material may be distributed throughout the solid fill material. Two easily separated solids of known but dissimilar densities may be mixed to a known combined density and used to fill the package with a near uniform distribution of both materials.

(a) Examples include the use of packing peanuts, resin pellets or other lower density solid to reduce total package weight and achieve 95 % minimum fill levels.

(b) Shot or other dense fill materials may be added in a distributed manner to augment the weight of the package.

7. Test Methods

7.1 For information on packaging performance for hazardous materials packaging, refer to Specification **D4919**.

7.2 For information on standards and testing of non-bulk specification packagings in conformance with the US Hazardous Materials Regulations (HMR), refer to 49 CFR 178 Subparts L through Subparts Q.

7.3 For information on standards and testing of non-bulk packagings in conformance with international air Dangerous Goods Regulations, refer to International Civil Aviation Organization (ICAO) Technical Instructions for the Safe Transport of Dangerous Goods by Air; Part 6 Packaging Nomenclature, Marking, Requirements and Tests.

7.4 For information on standards and testing of non-bulk packagings in conformance with international maritime dangerous goods regulations, refer to International Maritime Dangerous Goods Code (IMDG); Part 6 Construction and Testing of Packagings, Intermediate Bulk Containers (IBCs) and Large Packagings.

7.5 For relevant test methods in the determination of the physical characteristics of fill materials refer to Test Methods **D7481** and **D6393**.

8. Reporting

8.1 Test reports should contain the following information at a minimum:

8.1.1 Description of fill material (common name).

8.1.2 Density of the fill material.

8.1.3 Particle size of the fill material.

8.1.4 Angle of repose of the particulate fill material.

8.1.5 When using non-uniform (heterogeneous) fill materials in a receptacle:

8.1.5.1 Material used to increase (or decrease) package weight for testing, including physical attributes (shape, size, density), with a description of how it is placed in the receptacle (buried in fill or distributed throughout the fill material), including manufacturer/distributor and stock number, if known and relevant.

8.1.5.2 Combined net fill quantity per inner packaging/receptacle.

9. Keywords

9.1 dangerous good package testing; non-hazardous; package testing; solid; substitute fill material; UN testing