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Standard Guide for Selection of Substitute, Non-hazardous, Liquid Filling Substances for Packagings Subjected to the United Nations Performance Tests[1]

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1. Scope

1.1 The purpose of this guide is to clarify the selection, use and description criteria of non-hazardous liquid substitutes used to replace hazardous materials for the purpose of performance testing packagings. This includes identification of the physical parameters of substitute non-hazardous liquid 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).

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 The units of measurement are consistent with the HMR.

1.6 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.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

D1200 Test Method for Viscosity by Ford Viscosity Cup

D4359 Test Method for Determining Whether a Material Is a Liquid or a Solid

2.2 *Federal Standard:*

U.S. Department of Transportation Code of Federal Regulations Title 49, Transportation (49 CFR) Parts 100-199[3]

[1] This test method is under the jurisdiction of ASTM Committee D10 on Packaging and is the direct responsibility of Subcommittee D10.21 on Shipping Containers and Systems - Application of Performance Test Methods.

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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.

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

2.3 *UN Standard:*

United Nations Recommendations on the Transport of Dangerous Goods, Model Regulations (UN Orange Book)[4]

2.4 *International Air Transport Association (IATA) Standard:*

IATA International Air Transport Association (IATA) Dangerous Goods Regulations[5]

2.5 *ICAO Standard:*

International Civil Aviation Organization (ICAO) Technical Instructions for the Safe Transport of Dangerous Goods by Air[6]

2.6 *IMDG Standard:*

International Maritime Dangerous Goods Code (IMDG Code)[7]

3. Terminology

3.1 *Definitions:*

3.1.1 *kinematic viscosity, n*—the ratio of absolute or dynamic viscosity to density - a quantity in which no force is involved. Kinematic viscosity can be obtained by dividing the absolute viscosity of a fluid with its mass density:

$$\nu = \mu/\rho \quad (2) \qquad (1)$$

where:

ν = kinematic viscosity,
μ = absolute or dynamic viscosity, and
ρ = density.

3.1.1.1 *Discussion*—In the SI-system, the theoretical unit is m^2/s or commonly used Stoke (St) where: 1 St = 10^{-4} m^2/s.

3.1.1.2 *Discussion*—Since the Stoke is an impractically large unit, it is usual divided by 100 to give the unit called Centistokes (cSt) where: 1 St = 100 cSt; 1 cSt = 10^{-6} m^2/s.

3.1.1.3 *Discussion*—Since the specific gravity of water at 68.4°F (20.2°C) is almost one (1), the kinematic viscosity of water at 68.4°F is for all practical purposes 1.0 cSt.

3.1.2 *liquid, n*—a material, other than an elevated temperature material, with a melting point or initial melting point of 20°C (68°F) or lower at a standard pressure of 101.3 kPa (14.7 psia). A viscous material for which a specific melting point cannot be determined must be subjected to the procedures specified in ASTM D4359 "Standard Test Method for Determining Whether a Material is Liquid or Solid" (IBR, see §171.7).

3.1.3 *Newtonian liquid, n*—a liquid that exhibits a constant coefficient of viscosity as represented by a shear rate/shear stress plot that is both linear and passes through the origin of the shear rate/shear stress plot within the ranges of shear rates encountered in testing. A material not meeting this definition would be considered non-Newtonian.

3.1.3.1 *Discussion*—Newtonian liquids: Water, ethanol solutions, un-treated glycol solutions (glycol solutions treated to coat surfaces as a de-icing agent, such as airplane de-icing products, are formulated to be non-Newtonian).

3.1.3.2 *Discussion*—Non-Newtonian liquids: Many paints, toothpaste, peanut butter, ketchup, mayonnaise, blood, drilling mud, many fine particle slurries and pastes.

3.1.4 *shear rate, n*—the relative velocities in the flow of parallel adjacent layers (laminar flow) of a fluid from the application shear force. In aggregate, this is the rate of flow for a liquid in proportion to the amount of force causing the flow.

3.1.5 *shear stress, n*—the resistance (both internal and at the boundary) of a fluid to flow in reaction to a force applied parallel to the direction of motion in the flow channel (shear force).

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

3.1.6.1 *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.

3.1.7 *specific gravity, n*—ratio of the testing substance density to a standard substance (i.e. water) density at a specific temperature and pressure.

3.1.8 *viscous liquid, n*—a liquid material which has a measured viscosity in excess of 2500 centistokes at 25°C (77°F) when determined in accordance with the procedures specified in ASTM Test Method D445-72 "Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)" or ASTM Test Method D1200-70 "Viscosity of Paints, Varnishes, and Lacquers by Ford Viscosity Cup."

NOTE 2—Additional terms and definitions are located in 49 CFR section 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 liquid 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 (density, viscosity, net weight) 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 liquid 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

[4] Available from the UN Economic Commission for Europe, Information Service, Palais des Nations, CH-1211 Geneva 10 Switzerland (website: http://www.unece.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/).

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 under the widest possible situational circumstances. 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 them to evaluate whether a particular packaging was tested with a substitute material appropriate for the hazardous material to be shipped.

6. Procedure

6.1 For packaging intended for shipping liquid hazardous materials, water (or anti-freeze equivalents) is an acceptable and the preferred substitute material for package performance tests. Packagings tested and certified with water (or anti-freeze equivalents) as the fill material may be used to transport the widest variety of liquid hazardous materials without evaluation of viscosity or type of fluid (Newtonian or non-Newtonian). However, shippers must still evaluate fill materials with respect to packaging authorization, density, vapor pressure, compatibility etc., as prescribed in the relevant sections of the HMR.

6.1.1 For drop testing, if water is used as the substitute filling substance, drop heights must be determined using the prescribed methods in 49 CFR §§ 178.603(e). For packagings that must be tested in conformance with 49 CFR §§ 178.603(c), water/antifreeze solutions having a minimum specific gravity of .95 @ –18°C (0°F) may be substituted as water equivalents, provided they remain completely liquid at test temperatures with viscosity approximating water. Examples of commonly used (and recommended) water/anti-freeze solutions:

6.1.1.1 Propylene glycol/water mixture: minimum 50% Propylene Glycol/Water

6.1.1.2 Methanol/water mixture:

(1) By weight: 30%–45% methanol/water (produces freezing point of –25°C; density @ –18°C: 0.97–0.95)

(2) By volume: ≈ 37%–53%

6.1.1.3 Ethylene glycol/water mixture: minimum 40% Ethylene Glycol/Water

NOTE 3—Other anti-freeze mixtures may be used, but they must not be semi-solids (turned to slush) at the required temperature for testing. Salt water solutions may not be used as a water equivalent.

6.2 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.

6.3 If the material to be transported has been determined to be a liquid then a non-hazardous liquid other than water may be substituted as a fill material for testing purposes as prescribed in 49 CFR §§ 178.602(c).

6.3.1 If the 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 (… 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.

6.3.2 Substitute liquid test fill materials must have the same or lower viscosity under test conditions and the same or higher density than the material to be transported.

6.3.3 Characteristics that may influence test results if in variance with the material to be transported, and must be evaluated to ensure that a packaging that passes the required performance tests would be capable of doing so when filled with the actual hazardous product include:

6.3.3.1 Viscosity of the test fill material may prevent the detection of leakage (package failure) that would be apparent with a less viscous substitute fill material.

6.3.3.2 The viscosity of the fill material may affect the impact response of the package. This can result in a variation of package deformation under test conditions that can affect test results.

6.3.3.3 Liquid fill material with solids content (suspended uniformly or not) that may solidify at or clog a leak site and prevent detection of package failure that would be seen with liquid only fill.

6.3.3.4 A test fill material that may rapidly change physical state when in contact with air at a small leak site, turning viscous or solid and preventing the detection of a leak.

6.3.3.5 The use of a non-Newtonian liquid as a substitute fill material would only be indicated when the intended lading for the packaging is a non-Newtonian liquid. In this case, the shear rate/shear stress profile of the substitute material should match as closely as possible the shear rate/shear stress profile of the intended lading.

6.3.4 Given the difficulty of determining and matching the relevant physical characteristics of a hazardous material, as seen in the above list of considerations, when attempting to utilize a non-hazardous substitute fill material other than water, it is obviously more expedient, repeatable and easier to use water as the fill material. But, there are cases where a packaging manufacturer or shipper may select another material for package testing. In that case, test documentation and letters of notification for the subject packagings should indicate what physical characteristics were evaluated and contain a complete description of the substitute material.

6.4 49 CFR § 178.602(c) allows for the use of additives to achieve requisite package weight, with the caveat that the addition of the augmenting material does not affect the test

results. The additional materials added for weight may not act as a cushion material or in any way enhance the performance of the packaging in the required tests. [See: 49 CFR 178.602(c)]

6.4.1 The following are some common recommendations to consider in minimizing the performance effects of solid augmented liquids on the test process:

6.4.1.1 Maintaining the highest ratio of liquid/solid fill as possible under testing conditions. This allows the impact shock profile to be dominated by the liquid component, minimizing any change to drop/impact dynamics.

6.4.1.2 Using a solid material that flows readily and will not significantly alter the viscosity of the liquid in which it is placed under test conditions. Examples include: steel, ceramic, composite or lead shot or other materials with very low levels of cohesion and that do not affect the surface tension of the surrounding liquid.

6.4.1.3 Taking measures to distribute the solid evenly over the area of impact; such as gently shaking the package to distribute the solid material when the package is in the drop orientation on the test platform/fixture. This is particularly important for flat drop orientations, such as side drops.

NOTE 4—Regardless of fill material selected, with or without augmenting material, the minimum per receptacle fill level [98% of maximum capacity for liquids as prescribed in 49 CFR §§ 178.602(b)] for testing must be observed where applicable.

7. Test Methods

7.1 For information on packaging performance tests for hazardous materials packaging, refer to:

ASTM D4919 Standard Specification for Testing of Hazardous Materials Packagings

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 Subpart L-Non-bulk Performance-oriented Packaging Standards

49 CFR 178 Subpart M-Testing of Non-bulk Packagings and Packages

49 CFR 178 Subpart N-IBC Performance-Oriented Standards

49 CFR 178 Subpart O-Testing of IBCs

49 CFR 178 Subpart P-Large Packaging Standards

49 CFR 178 Subpart Q-Testing of Large Packagings

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), Large Packagings, ...

7.5 For relevant test methods in the determination of the physical characteristics of fill materials, refer to:

ASTM D445-72 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)

ASTM D1200-70 Test Method for Viscosity of Paints, Varnishes, and Lacquers by Ford Viscosity Cup

ASTM D891-09 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

ASTM D4359 Test Method for Determining Whether a Material is Liquid or Solid

8. Report

8.1 Test reports for packagings containing liquids should contain the following information at a minimum:

8.1.1 Relative density (Specific Gravity), if different from water or anti-freeze solutions that are considered water equivalents.

8.1.2 Viscosity, if different from water or anti-freeze solutions that are considered water equivalents.

8.1.2.1 If a non-Newtonian liquid was used as a substitute material, the description in the test report should include the type (Kelvin material, Thixotropic, Dilatant, pseudoplastic) and the relevant shear rate/shear stress data appropriate to the application. In addition, the compounded components with mix ratios should be included.

8.1.3 When using non-uniform, weight augmented liquids in a receptacle:

8.1.3.1 Liquid fill component: Specific gravity and viscosity if different from water or anti-freeze solutions that are considered water equivalents.

8.1.3.2 Solid fill component: Material of composition and physical attributes (shape, size, density), with a description of how it is placed in the receptacle (bagged, free flowing etc.), including manufacturer/distributor and stock number, if known.

8.1.3.3 Combined net fill quantity per inner packaging/receptacle.

9. Keywords

9.1 dangerous goods package testing; liquid; Newtonian liquid; non-hazardous; non-Newtonian liquid; package testing; substitute fill material; UN testing