INTERNATIONAL

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Standard Test Method for ~~Thermal Insulation Quality of Packages~~Thermal Insulation Performance of Distribution Packages[1]

This standard is issued under the fixed designation D 3103; 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.

ε[1] NOTE—Removed research report footnote from Section 11.2 editorially in September 2008.

1. Scope

~~1.1This test method covers the determination of the thermal insulation quality of a package and its enclosed packaging from temperature differentials between the packaged item and the outside environment. It is suitable for testing packages with and without various internal refrigerants and with or without interior packaging. Representative test conditions covered are indicated by Fig. 1 and Fig. 2. Depending upon the type of insulation material used, a water-vaporproof barrier might be used just inside the exterior packages of Fig. 1.~~

1.1 This test method covers the determination of the thermal insulation quality of a package and the thermal stability of its contents when exposed to variable ambient temperature conditions. It is suitable for testing packages with various internal energy sources with or without product payloads.

1.2 *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.* Specific precautionary statements are given in ~~5.2.3~~5.3 ~~and 9.2.2~~.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 996 Terminology of Packaging and Distribution Environments

D 4332~~Practice for Conditioning Containers, Packages, or Packaging Components for Testing~~[2] Practice for Conditioning Containers, Packages, or Packaging Components for Testing

2.2 *Other Standards:*

ISTA 5B Focused Simulation Guide for Thermal Performance Testing of Temperature Controlled Transport Packaging[3]

3. Terminology

3.1 *Definitions*—General definitions for packaging and distribution environments are found in Terminology D 996.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 ~~*draft-free atmosphere*—a relatively stationary atmosphere where the test specimens are remote from air currents.~~ data acquisition unit and associated system—single- or multi-channel recorder and its associated software and hardware utilizing thermocouples and thermistor sensors traceable to NIST (National Institute for Standards and Technology) that collects and date stamps time and temperature.

3.2.2 *draft-free atmosphere*—an atmosphere where the test specimens are isolated from direct air currents while surrounding air temperature is maintained uniformly throughout the chamber.

3.2.3 *eutectic system*, *n*—a mixture or compound in which pure solid phases changes occur at a well-defined specific temperature.

~~3.2.3*exterior atmosphere*—the atmosphere in contact or near the exterior surface of a package.~~

3.2.4 ~~*exterior package*—the outermost container of a package.~~ exterior atmosphere—the atmosphere surrounding the exterior surface of a package.

[1] This test method is under the jurisdiction of ASTM Committee ~~D-10~~D10 on Packaging and is the direct responsibility of Subcommittee D10.23 on Natural Environment 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* ~~, Vol 15.09.~~volume information, refer to the standard's Document Summary page on the ASTM website.

~~[3] Supporting data have been filed at ASTM Headquarters and may be obtained by requesting Research Report RR: D10–1012.~~

[3] Available from International Safe Transit Association (ISTA), 1400 Abbott Rd., Suite 160, East Lansing, MI 48823–1900, http://www.ista.org.

3.2.5 *interior atmosphere*—the atmosphere in contact or near the packaged item.

3.2.6 ~~*interior package*—a package (often of corrugated fiberboard) located within another.~~ mapping—collecting temperature data at a wide range of locations inside a package or chamber to determine the variability of temperature range in the environment.

3.2.7 *package system*—the combination of exterior package, interior packaging, refrigerants, and product payload.

3.2.8 *product payload*—the product and any associated secondary packaging that is to be temperature controlled within the insulated test package.

3.2.9 *refrigerants*—eutectic materials, gel packs, ice, or other material that serves as an energy source or buffer medium within the package system.

3.2.10 *secondary package*—the package that contains the primary container/closure system(s).

3.2.11 *thermal conductivity, homogeneous material*— the rate of heat flow, under steady conditions through unit area, per unit temperature gradient in the direction perpendicular to the area.

4. Significance and Use

~~4.1Certain items, such as biological materials, pharmaceuticals, industrial adhesives, gyroscopes, blood, and some foods, must be shipped inside temperature-controlled packages. Factors affecting the rate of heat transfer of the package include the moisture content of the different package components and the thickness, continuity, density, position, and uniformity of the insulation.~~

~~4.2Because of the variety of factors affecting the performance of a thermally insulated package, testing should be conducted with the actual package whenever possible. When simulated packages are used, special care must be exercised so that the simulated payload and coolant of the model will be as close as possible to the actual materials in temperature and other relevant physical properties.~~

4.1 This test method is intended for use for evaluating the performance of thermal insulated packaging used for high-value, high-risk materials. This test method may also be used for any product that requires accurate internal package temperature readings while being exposed to a range of external air temperatures.

4.2 Certain items, such as biological materials, pharmaceuticals, diagnostics, and blood products, must be shipped inside temperature-controlled packages. Factors affecting the rate of heat transfer of the package include the insulation of the exterior package, the energy source, and the product payload.

4.3 Because of the variety of factors affecting the performance of a thermally insulated package, testing should be conducted with the actual package whenever possible. When simulated packages are used, special care must be exercised so that the simulated payload and coolant will be as close as possible to the actual packages in temperature and other relevant physical properties.

5. ~~Test Conditions and Apparatus~~

~~5.1*Temperature of Exterior Atmosphere*— Draft-free environments large enough to accommodate the packages, but not necessarily the recorder, are required. Usual evaluation of the packaging materials will involve simple refrigerated or ambient exterior atmospheric conditions. These may be established as 4 ± 2°C (39 ± 4°F), 37.8 ± 2°C (100 ± 4°F), and 23 ± 2°C (73± 4°F). Additionally, other temperature extremes or cycles between various temperatures expected during shipment might be used as an exterior atmosphere.~~ Test Conditions and Apparatus

5.1 *Temperature Profile of Exterior Atmosphere*—The time-temperature test profile should be established prior to testing based on actual field data, compendial or regulatory requirements, or contractual requirements.

5.1.1 *Field Data Time-Temperature Profile*—It is recommended that the test profile represent actual worst case distribution conditions as closely as possible. The test profile of exterior package temperatures should be based on actual ambient air data accumulated during package handling and transit whenever possible. Any published test cycle or cycle developed using ISTA 5B may also be used as applicable. When using a method based on actual data or developed in accordance with ISTA 5B, the rate of temperature change between trip segments should reflect, as closely as possible, actual transit conditions. Should other than worst case conditions be used, indicated the percentile of the data pool that the profile represents.

5.1.2 *Regulatory Requirements*—When using a time-temperature test profile from a regulatory or compendial source, such as the WHO, cite the source and its application.

5.1.3 *Contractual Requirements*—Should the time-temperature test profile be stipulated by contract, cite the source and, where available, the rationale for the profile.

5.1.4 *Constant Temperature*—A constant temperature profile may be used, especially to determine relative performance of insulating materials. Constant temperatures do not reflect actual transit conditions but may be useful for comparative testing or for research.

5.2 ~~*Temperature of Interior Atmosphere of Package* —When no refrigerant is used, interior package temperature may serve as a measure of the thermal insulation quality of the package. If it is desired to control the interior temperature of the atmosphere, use one of the following:~~

~~5.2.1*Refrigerant Gel, Water Ice, or Other Refrigerant Source*, for internal temperatures above 0°C (32°F),~~

~~5.2.2*Solid Carbon Dioxide CO*~~ Test Chamber—Tests must be performed in one or more rooms or cabinets (chambers) for which test samples can be individually placed with adequate space around all surfaces for air circulation at the desired temperature. An access port should be available for leading thermocouple wires out of the chamber for hook-up to the data acquisition unit (DAU). A temperature indicator should be placed 10 in. from the test package to record the temperature of the exterior atmosphere during the entire test duration.

5.3 *Test Chamber Controller*—The room or cabinet must maintain a uniform temperature around the test specimen. The test chamber control apparatus must be capable of maintaining the desired temperature to within ±3°C. It may be desirable to incorporate a programmable controller with the capability of performing temperature profiles (for example, multiple temperature changes over time). However, the temperature cabinet heating and cooling mechanisms must have the capability to change temperature at the desired ramp rates of the profile. (**Warning**—Gaseous CO_2 (*Dry Ice or Euctectic Systems*), wrapped as used in shipments for maintaining internal temperatures down to−73°C (−108.4°F), or

5.2.3*Liquid Nitrogen*, for temperatures to−195°C (−319°F).

NOTE1—**Caution:**Gaseous CO is colorless, odorless, and noncombustible. In well-ventilated uses they present few problems, but evaporation or sublimation in airtight enclosures for prolonged periods (for example, 12 h) can produce sprung doors and asphyxiation of operating personnel. Usually these CO_2 and nitrogen are colorless, odorless, and noncombustible. In well-ventilated uses they present few problems, but evaporation or sublimation in airtight enclosures for prolonged periods (for example, 12 h) can produce sprung doors and asphyxiation of operating personnel. Usually these refrigerants can be used if provisions are made to evacuate the built-up gas periodically.

5.3*Temperature Indicators*can be used if provisions are made to evacuate the built-up gas periodically.)

5.3.1 *Single or Multi-Channel Data Acquisition Unit (DAU)*:

5.3.1*Multi-Channel Recorder with Thermocouples*—The recording capability should be as a datalog by thermocouple number with date and time of reading, that can be presented in a continuous graph form as a secondary presentation. Resolution of the device shall be 0.1°C or greater. Accuracy over the range tested should be ± 1°C. The printer or associated computer datafile shall be activated by a voltage from an insulated pair of copper-constantan or other suitable wires 30 AWG B&S gage (0.255 mm) or less in diameter (not including the thickness of insulation). The recorder and chart shall have a capability that extends beyond the temperature values encountered in the test.

5.3.1.1 The recording capability should be as an electronic datalogger by sensor number with date and time of reading that can be presented in a continuous graph form as a secondary presentation. Resolution of the device shall be 0.1°C or greater. Accuracy over the range tested should be ±0.5°C. The printer or associated computer datafile shall be activated by a voltage from an insulated copper-constantan wires, Type T, or other suitable sensor for the temperature range to be measured that are specified by the manufacturer to be accurate to 0.1°C. The wires may be single or multi-strand and should be flexible enough to be run through repeated bends in the package. Any tips or probes added to the wire should be noted and should not change the accuracy or response time of the thermocouple.

5.3.2 *Thermistor-Recorder*—A thermistor sensor may be used, instead of a thermocouple, for sensing interior temperatures of the package. The thermistor may be attached to recording equipment, as described in 5.3.1, with supplementary electrical circuitry as needed, or it may be a wireless, battery operated, computer programmable unit that stores digital temperature readings at specified time intervals. Programming and data downloading of the units is done through a suitable computer interface with appropriate software. Accuracy over the range tested should be ± 1°C with resolution to 0.1°C. Response time over range should be determined prior to use and suitable for the reading interval of the test. , with supplementary electrical circuitry as needed, or it may be a wireless, battery operated, computer programmable unit that stores digital temperature readings at specified time intervals. Programming and data downloading of the units is done through a suitable computer interface with appropriate software. System accuracy over the range tested should be ±0.5°C with minimum resolution to 0.1°C. Response time over range should be determined prior to use and suitable for the reading interval of the test.

5.4 *Calibration Reference Standard*—A NIST-traceable device used in conjunction with a constant temperature bath when calibrating and verifying accuracy pre- and post-test. The resolution and accuracy must be equal to or better than the sensors used in testing.

5.5 *Constant Temperature Bath*—A device or method that produces a stable and consistent reference temperature within ±1°C of a desired set point used in the calibration and verification of temperature sensors. The bath may produce the temperature reference point by means of an electronic signal or temperature controlled liquid bath (the type of liquid may vary depending of system and temperature). A bath in which the temperature is both stable and consistent within ±1°C of the desired set-point and is used in the calibration and verification of thermocouples. The bath solution may vary depending on the desired set-point.

6. Sampling

6.1Experimental package designs shall be made in accordance with the specifications and methods that will be used during actual production. When possible, choose the test packages by random sampling.

6.1 Experimental package designs (prototypes) shall be made as close to the specifications and methods as possible that will be used during actual production.

6.2 A minimum of three samples must be tested to ensure reproduceability.

7. Test Specimens

7.1A single test specimen shall consist of a package enclosing the actual item or a dummy load simulating the item. The package shall be closed, taped, or sealed in the same manner as will be used for actual shipment.

7.2The mass, configuration, and location of refrigerant, if used, must be the same in each pack.

7.3For development or screening evaluation of the overall insulation effectiveness of the container and insulation material, the interior cavity and package wall thickness shall be kept constant (for example, a 12 by 12 by 12-in. (305 by 305 by 305-mm)

~~interior cavity surrounded by a 1-in. (25-mm) thickness of insulation and a 14 by 14 by 14-in. (356 by 356 by 356-mm) container. Test a minimum of three such packages at each exposure listed in 5.1 to obtain an average result and range of performance.~~

~~7.4When testing packages having known or previously established performance data, a minimum of three identical samples shall be tested to determine reproducibility and repeatability.~~ Test Specimens

7.1 In designing a package system it is suggested that the payload contents within the insulated container is mapped to determine hot and cold locations throughout.

7.2 The packaging system and its components, mass, configuration, and locations shall have identical assemblies. The package system shall be closed, taped, or sealed in the same manner as will be used for actual shipment.

8. Conditioning

~~8.1Condition materials in accordance with Practice D4332 or for 24 h at the conditions expected during actual production packing.~~

8.1 Condition all materials in accordance with Practice D 4332 or in accordance with the instructions provided in the test protocol for a minimum of 24 h or until stable at the conditions expected during actual production packing.

8.2 *Pre-conditioning*—Specific test procedures may require certain pre-conditioning of components just prior to assembling the test packages. Pre-conditioning, if any, should be done in accordance with individual test requirements.

9. ~~Procedures~~

~~9.1*Packages Enclosing Solid Refrigerants*~~ Procedures

9.1 The desired test packaging configuration will be documented in such a way to identify all components, their locations, thermocouple locations, test equipment, test conditions, and test start time.

9.2 Calibrate the sensors using the NIST-traceable reference standard. Place sensors in stable temperature bath along with the reference standard. Record temperatures from the sensors and reference standard independently for a minimum of three temperature points. The overall system difference between the reference standard and each sensor should be within ±0.5°C. If using a thermistor, ensure the equipment is certified as calibrated to a NIST-traceable source.

9.3 *Test Package Assembly*:

~~9.1.1Verify the calibration of the temperature-monitoring equipment per manufacturer's directions against an NIS traceable standard suitable for the ranges being measured. Battery-operated thermistors recorders with or without external probes, should be calibrated by the manufacturer and a certificate of NIS traceability of accuracy provided.~~

~~9.1.2Place the contents in the conditioned package according to the specific configuration to be tested.~~

~~9.1.3Insert the sensors in the package so that they are in contact with the item(s) as follows:~~

~~9.1.3.1For packages having an inside volume of 1 ft ^{3} or less and a single open cavity for both ice and product, place one sensor at or near the diagonally opposite corners of the item as shown in Fig. 1*a*. Additionally, the sensors and at least ½ in. (13 mm) of wire immediately behind them should be in contact with the item. Wrapping the wires around the item, if practical, is advantageous to minimize heat flow from the outside.~~

~~9.1.3.2For packages designed with interior walls separating product and refrigerant, more thermocouples may be needed to adequately monitor temperatures. Again, porting through a wall directly into the product cavity is recommended. The port should be kept as small as possible and sealed after insertion of thermocouples to prevent leaking air.~~

9.3.1 Pre-condition all packaging materials, refrigerants, and products at specified pre-test temperatures for the package assembly to be tested. Pre-conditioning should be for at least 24 h, or as specified. Be sure to allow sufficient time for all materials to reach desired temperature, that is, frozen solid. Record all pre-conditioning temperatures.

9.3.1.1 If feasible, attach probes directly to the wall of the primary product package and/or inside the package in direct contact with the product, prior to pre-conditioning. When attaching probes, avoid covering the sensor area with tape or other material that could inhibit the sensor's ability to read temperatures at the specified speed and accuracy.

9.3.1.2 The number of probes per package will be dependent on the size and number of the primary containers in the payload. In general, when first evaluating a thermal package, a minimum of 5 to 10 probes should be used on payloads of up to 25 primary packages. Record the probe placements in the package assembly.

NOTE 2—~~It is desirable to use wireless data recorders, if possible, on smaller units to minimize temperature leaks at the thermocouple insertion site. If thermocouples are used, a small port can be opened through the wall of the carton as far away from the ice as possible, the thermocouples inserted into the carton, and the port sealed to prevent leaking of air. Do not place battery operated recorders in packages containing dry ice as the units will be damaged and nonfunctional at these temperatures. Units with remote probes may be placed outside the carton where the battery and microchip will not be exposed to dry ice conditions.~~

~~9.1.3.3For packages having usable inside volume of more than 1 ft^3, a screening test using a minimum of ten thermocouples to determine the locations of the warmest and coldest spots is recommended. The larger the usable capacity, the more thermocouples may be needed to adequately screen and test the package. Packages with interior walls separating product and refrigerant may require testing at extreme temperatures above or below those specified in 5.1 to fix locations of hot and cold spots. Every interior configuration, including quantity and placement of both ice and product should be screened, as changes to either element will alter the interior temperature profile of the package. Once warmest and coldest locations are identified, the number of thermocouples can be adjusted upward or downward, depending on the reproducibility of the warm and cold locations.~~