Designation: D3103 – 14

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

This standard is issued under the fixed designation D3103; 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 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 5.3.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D996 Terminology of Packaging and Distribution Environments

D4332 Practice for Conditioning Containers, Packages, or Packaging Components for Testing

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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 D996.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *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.

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

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

3.2.6 *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

4.1 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.

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

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 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 *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 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 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):*

5.3.1.1 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. 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

6.1 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

7.1 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

8.1 Condition all materials in accordance with Practice D4332 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 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:*

9.3.1 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 1—It may be desirable to use wireless data recorders, if possible, on smaller units to minimize air exchange caused by 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.

9.3.2 When a cooling or heat source is used, place a weighed and measured quantity of refrigerant or phase change material in the package as specified in the test protocol. When water, ice, or refrigerant bottles, gel, or bricks are used, measure and record the temperature of the conditioning chamber before removal. Any pre-conditioning prior to package the test assemblies should be monitored during pre-conditioning by ambient air probes.

9.3.3 The quantity of energy source or eutectic system may be varied. However, when materials and designs of similar inside dimensions are being evaluated, the quantity by mass, configuration, and total surface area must be constant for the series for accurate evaluation of performance. When crushed or chopped dry ice is used, the fragments should be of the same general size for each package tested and should be free of dry ice dust.

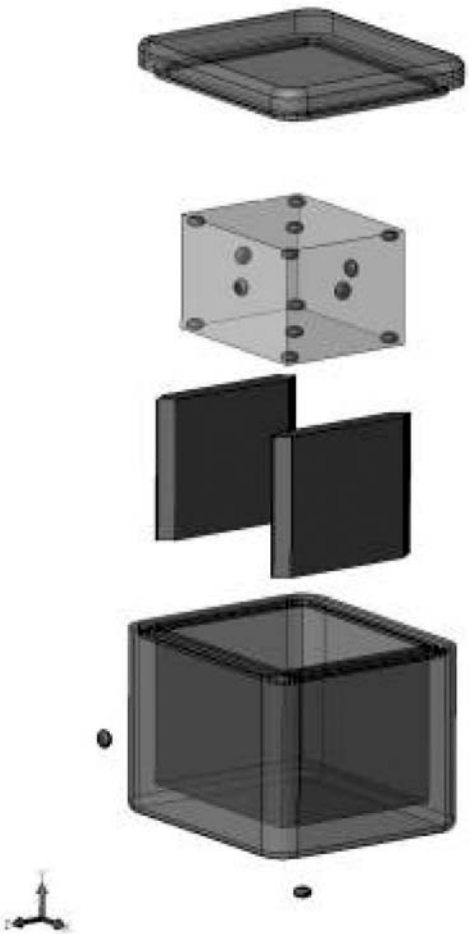
9.3.4 If wires are not ported through the wall of the insulated carton, place the cover on the package with the sensor wires arranged so that the least possible heat transfer occurs through the joint.

9.3.5 Tape or otherwise secure the cover to the package so the air transfer around the wires is minimized. Do not tape around the entire top/base seal unless this is to be done in normal use. If dry ice is used, precautions must be taken to allow the package to vent off CO_2 vapors as the dry ice sublimates.

9.3.6 Place the package in the test environment on a non-conducting platform or shelf in such a manner that no part of the package contacts the chamber walls or floor. If more than one package is tested at one time, each package shall be separated from each other by at least 6 in. (152 mm). Mark the chart with date and time and start the recorder.

9.3.7 The orientation of the carton should be the same as that experienced in shipping. For small cartons where various orientations occur during shipping, all orientations may be screened and tested, as the orientation may affect the interior temperature distribution. If more than one carton is shipped at a time, duplicate the usual configuration of the units during shipping in the test, as the added insulation value of adjacent cartons affects the time/temperature profile of the carton as well as location of coldest and warmest spots. See Fig. 1.

9.3.8 If data is collected in digital format, the recorder should be set to record temperatures at a desired time interval not to exceed 30 min. A recording interval of 10 min is recommended for most tests, but can be shorter if needed. Continue to collect data as instructed by the product acceptance criteria until the interior temperatures are above the maximum



NOTE 1—Payload about 1 ft^2.

FIG. 1 Example of Sensor Placement for Payload with Multiple Small Inner Packages

or below the minimum temperatures dictated by the product, or until the total test time has elapsed. If the data can be reviewed on an exterior monitor, end the test when the desired limit is reached. If using microchip based battery operated recorders without exterior monitoring, end the test when the total elapsed time limit is reached.

9.3.9 Verify the calibration accuracy of the temperature-monitoring equipment at the end of the test. Place thermocouples used in a stable constant temperature bath with a NIST-traceable calibration reference standard. Record temperatures from the thermocouples and reference standard independently for a minimum of three temperature points. The overall system difference in temperatures between the calibration reference standard and each thermocouple should be within ±0.5°C.

9.3.10 Mark the date and time of the start and end of the testing on the chart or digital data record.

10. Report

10.1 Report the following information:

10.1.1 Dimensions of the package under test, its structural specifications, the materials of construction, the name of manufacturer, and the method of closing or securing a lid.

10.1.2 Description of item(s) and secondary packaging materials.

10.1.3 A diagram of the internal thermal component assembly.

10.1.4 A layout diagram of test product placement within the thermal package assembly and location of thermocouples or other sensors.

10.1.5 Type, quantity, and location of refrigerant and devices within the package (in the case of water, ice, and refrigerant gel, record the initial ice and gel temperatures and its pre-test storage temperature and pre-conditioning, if any).

10.1.6 A complete description and identification of temperature data acquisition unit system used.

10.1.7 Method of conditioning package materials and product payload prior to test.

10.1.8 Temperature and relative humidity of the test environment.

10.1.9 Number of hours recorded on the chart or digital data record by each sensor from the start of the test to the point where the temperature starts to exceed the maximum or minimum temperature established for the test.

10.1.10 Quantity, temperature, and state (if pertinent) of refrigerant remaining at the end of the test, or the test duration has been reached.

10.1.11 Description of the physical state of the contents at the end of the test.

10.1.12 Evaluation of the test with respect to the effective storage time in comparison to tests of other packaging materials or other designs.

10.1.13 Thermocouple pre-test calibration and post-test verification data or manufacturer's certification.

10.1.14 A statement that all tests were made in accordance with this test method.

11. Precision and Bias

11.1 The precision of this test method is based on an interlaboratory study conducted in 2013. As many as seven laboratories tested two different packaging materials for temperature failure, at three different positions, and at two discrete temperature levels. Every test result represents an individual determination, and all participants were asked to report triplicate test results. Practice E691 was followed for the design and analysis of the data; the details are given in RR:D10-1016.[4]

11.1.1 *Repeatability, r*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

11.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D10-1016. Contact ASTM Customer Service at service@astm.org.

TABLE 1 8°C Failure at Position 1 (hours)

Packaging Material	Average,[A] $\bar{x}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
SAMPLE A- EPS	1.29	0.28	1.11	0.77	3.09
SAMPLE B- PUR	2.01	0.51	2.18	1.44	6.10

[A]The average of the laboratories' calculated averages.

TABLE 2 12°C Failure at Position 1 (hours)

Packaging Material	Average,[A] $\bar{x}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
SAMPLE A- EPS	3.98	0.67	1.93	1.87	5.40
SAMPLE B- PUR	8.80	2.81	4.32	7.87	12.10

[A]The average of the laboratories' calculated averages.

TABLE 3 8°C Failure at Position 2 (hours)

Packaging Material	Average,[A] $\bar{x}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
SAMPLE A- EPS	1.94	0.38	1.52	1.07	4.25
SAMPLE B- PUR	2.59	0.97	2.01	2.72	5.64

[A]The average of the laboratories' calculated averages.

TABLE 4 12°C Failure at Position 2 (hours)

Packaging Material	Average,[A] $\bar{x}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
SAMPLE A- EPS	5.36	0.66	2.54	1.84	7.11
SAMPLE B- PUR	9.65	2.69	4.57	7.52	12.80

[A]The average of the laboratories' calculated averages.

TABLE 5 8°C Failure at Position 3 (hours)

Packaging Material	Average,[A] $\bar{x}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
SAMPLE A- EPS	2.36	0.48	2.26	1.36	6.32
SAMPLE B- PUR	2.97	0.23	3.23	0.64	9.06

[A]The average of the laboratories' calculated averages.

TABLE 6 12°C Failure at Position 3 (hours)

Packaging Material	Average,[A] $\bar{x}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
SAMPLE A- EPS	6.85	0.46	3.93	1.29	10.99
SAMPLE B- PUR	11.70	1.64	4.21	4.59	11.77

[A]The average of the laboratories' calculated averages.

11.1.1.2 Repeatability limits are listed in Tables 1-6.

11.1.2 *Reproducibility, R*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

11.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

11.1.2.2 Reproducibility limits are listed in Tables 1-6.

11.1.3 The above terms (*repeatability limit* and *reproducibility limit*) are used as specified in Practice E177.

11.1.4 Any judgment in accordance with statements 11.1.1 and 11.1.2 would have an approximate 95 % probability of being correct.

11.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

11.3 The precision statement was determined through statistical examination of 216 test results, reported by 7 laboratories, on 2 materials, at 2 temperatures.

11.4 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

12. Keywords

12.1 data acquisition unit; insulated package; thermal profile; thermal package