Designation: D 3103 – 99

Standard Test Method for Thermal Insulation Quality of 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.

1. Scope

1.1 This 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.

1.2 *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 and 9.2.2.

2. Referenced Documents

2.1 *ASTM Standards:*

D 996 Terminology of Packaging and Distribution Environments[2]

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

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:*

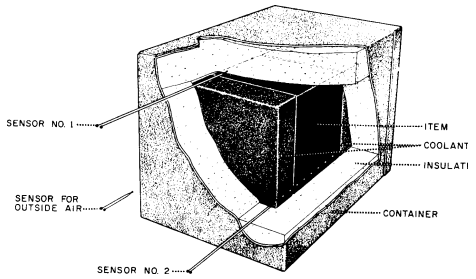
3.2.1 *draft-free atmosphere*—a relatively stationary atmosphere where the test specimens are remote from air currents.

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

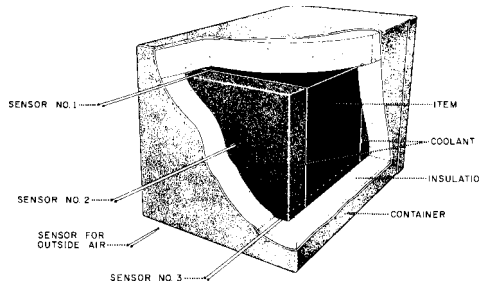
3.2.3 *exterior atmosphere*—the atmosphere in contact or near the exterior surface of a package.

3.2.4 *exterior package*—the outermost container of a package.

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



a—Containers Having Usable Inside Volume of 1 ft^3(0.03 m^3) or Less.



b—Containers Having a Usable Volume of More Than 1 ft^3(0.03 m^3)

FIG. 1 Thermally Insulated Packages with Sensors Positioned

3.2.6 *interior package*—a package (often of corrugated fiberboard) located within another.

3.2.7 *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 Certain 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.

4.2 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 of the model will be as close as possible to the actual materials in temperature and other relevant physical properties.

[1] This test method is under the jurisdiction of ASTM Committee D-10 on Packaging and is the direct responsibility of Subcommittee D10.23 on Natural Environment Test Methods.

Current edition approved Oct. 10, 1999. Published December 1999. Originally published as D 3103 – 72. Last previous edition D 3103 – 92.

[2] *Annual Book of ASTM Standards*, Vol 15.09.

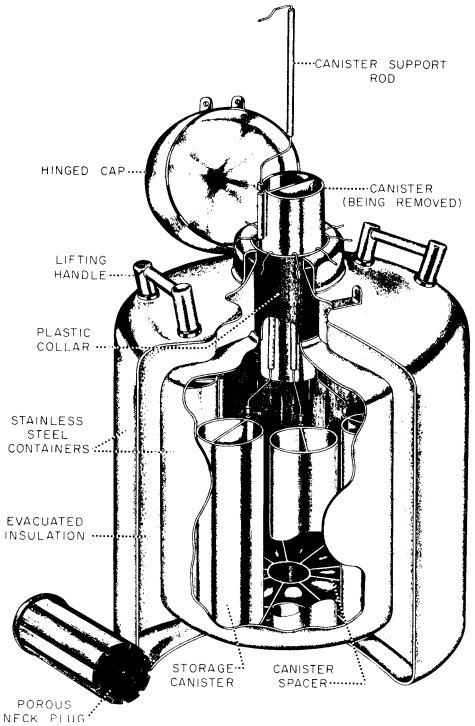


FIG. 2 Liquid Nitrogen Refrigerator

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.

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_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).

NOTE 1—**Caution:** Gaseous 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*:

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.

5.3.2 *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.

6. Sampling

6.1 Experimental 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.

7. Test Specimens

7.1 A 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.

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

7.3 For 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.

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

8. Conditioning

8.1 Condition materials in accordance with Practice D 4332 or for 24 h at the conditions expected during actual production packing.

9. Procedures

9.1 *Packages Enclosing Solid Refrigerants*:

9.1.1 Verify 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.

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

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

9.1.3.1 For 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.

9.1.3.2 For 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.

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.

9.1.3.3 For 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.

9.1.4 Place a weighed and measured quantity of refrigerant in the package. When water ice or refrigerant gel are used, measure and record their temperature at the start of the test. Do this by securely taping a sensor to the outside of the refrigerant container or by inserting a sensor into the water prior to freezing.

9.1.4.1 The quantity of refrigerant 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.

9.1.5 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.

9.1.6 Tape or otherwise secure the cover to the package in the normal manner.

9.1.7 Place the package in the test environment on a wooden 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, they shall be separated from each other by at least 6 in. (152 mm). Mark the chart with date and time and start the recorder.

9.1.7.1 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 should be screened and tested, as the orientation affects the interior temperature profile. 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.

9.1.8 If data is collected in digital format, the recorder should be set to record temperatures not longer than every 30 to 60 min in screening tests. Intervals as short as 5 min may be needed for thin-wall containers exposed to low exterior air temperatures. Once a temperature profile has been established, recording intervals can be extended to every 3 h, depending on the duration of the test and the rate of internal temperature change. Continue to collect data until the interior temperatures are above the maximum 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 micro-chip based battery operated recorders without exterior monitoring, end the test when the total elapsed time limit is reached.

NOTE 3—Larger capacity cartons show a slower rate of temperature change than small cartons. Temperature reading intervals should be adjusted to the rate of change, that is, shorter for small packages with lower insulation values and longer for larger packages with higher insulation values.

9.1.9 Check the calibration of the temperature-monitoring equipment at the end of the test.

9.1.10 Mark the date and hour of the start and end of the testing on the chart or digital data record.

9.2 *Containers with Liquid Nitrogen Refrigerant (*Fig. 2*)*:

9.2.1 Detach the necessary sensor wires from the temperature recorder, calibrate, and place the sensing end(s) within the container adjacent to the contents.

9.2.2 Condition the container and contents by immersing them in liquid nitrogen for the length of time specified by the manufacturer, or by another recommended method.

NOTE 4—**Caution:** The temperature of liquid nitrogen is about − 195°C (−319°F). Therefore, when liquid nitrogen is used as the cooling agent, extreme care must be used during purging to maintain a nitrogen atmosphere adjacent to surfaces having a temperature near that of liquid nitrogen. This is necessary to prevent oxygen condensation and the related explosive fire danger.

9.2.3 With the contents in the conditioned container, add the liquid nitrogen as recommended by the container manufacturer.

9.2.4 Attach the cap or closure to the container with the sensor wire or wires extending between the closure and container in such a manner that the least possible nitrogen leakage occurs.

9.2.5 Attach the ends of the sensor wires to the recorder and proceed as in 9.1.7.

9.2.6 Allow the recorder to run until the indicated temperatures are above the maximum established for the particular test.

9.2.7 Check the calibration of the temperature-monitoring equipment and record the date and hour.

9.3 *Packages Without Refrigerants*:

9.3.1 When packages are tested without refrigerants, the thermal insulation quality is determined by observing the temperature change between the interior atmosphere or product and the controlled atmosphere outside the package.

9.3.2 Calibrate the temperature-monitoring equipment.

9.3.3 If the package (with or without interior packaging), is to be tested empty, place at least one sensor on the inside surface of the package, and at least one additional sensor near the center of the package.

9.3.4 If the package is to be tested with the product, embed at least two sensors in or in contact with the product, or as many sensors as are needed to test the entire interior.

9.3.5 Suspend one sensor in the controlled atmosphere outside and in close proximity of the package(s) to serve as a check against the usual recording controls of the environmental chamber.

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 securing the lid.

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

10.1.3 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).

10.1.4 Type of temperature recording system used, (if a thermocouple system was used, indicate the location of thermocouple leads and junctions within the package).

10.1.5 Method of conditioning package and contents prior to test.

10.1.6 Temperature and relative humidity of the test environment.

10.1.7 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.

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

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

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

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

11. Precision and Bias

11.1 *Precision*—The precision of this test method has been determined by an interlaboratory study.

11.1.1 *Repeatability*—When an analysis is conducted on the times in hours to reach a critical temperature, the within-laboratory standard deviation is typically about 4 % of the mean. When a lognormal analysis is conducted on the natural logarithms of the time in hours, the typical within-laboratory standard deviation is 0.04. These values may vary depending on the package or container being tested and the capabilities of each laboratory.

11.1.2 *Reproducibility*—When an analysis is conducted on the times in hours to reach a critical temperature, the between-laboratory standard deviation is typically about 10 % of the mean. Then a lognormal analysis is conducted on the natural logarithms of the times in hours, the typical between-laboratory standard deviation is about 0.1. These values may vary depending on the package or container being tested and the capabilities of each laboratory.

11.2 *Bias*—It is not possible to specify the bias of this test method as no accepted reference material or method is available.[3]

12. Keywords

12.1 heat transfer of packages; thermal insulation; temperature controlled packages

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