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PQS Independent type-testing protocol

TITLE: Phase-change material containers

Product verification protocol:	E005/PCMC01-VP0.1
Applies to specification ref(s):	E005/PCMC0.1
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Contents

1. Scope

This document describes the procedure for verifying the performance of phase-change material (PCM) containers used in vaccine cold chain equipment (CCE) and includes verifying specifications for both the PCM itself and the container holding the PCM. This verification protocol addresses fixed containers that are not intended for removal from the CCE by end users. Removable containers that are regularly removed from the CCE by the end user are not permitted under this specification. CCE includes equipment prequalified in categories E001 – Cold rooms, freezer rooms, and related equipment; E003 – Refrigerators and freezers; and E004 – Carriers and cold boxes.

2. Normative references

International Organization for Standardization (ISO)/International Electrotechnical Commission (IEC) 17025:2005: General Requirements for the Competence of Testing and Calibration Laboratories.
ISO 12048: 1994: Packaging – Complete, filled transport packages – Compression and stacking tests using a compression tester.

3. Terms and definitions

Cold chain equipment (CCE): Equipment used to maintain the temperature of vaccines or other medical products and samples in an acceptable temperature range. This definition includes refrigerators, refrigerated rooms, carriers and cold boxes.
Container: A closed volume with walls designed to hold a PCM.
Fixed container: A PCM container that is an integral part of the CCE and is not expected to be regularly removed from the CCE by the end user.
In writing: Communication by letter, fax or email.
Legal manufacturer: The natural or legal person with responsibility for the design, manufacture, packaging and labelling of a product or device before it is placed on the market under her/his own name, regardless of whether these operations are carried out by that person or on her/his behalf by a third party.
Member: A geometrical part of a container such as a face, edge, corner, side or chime.
Phase-change material (PCM): A material, other than water, which changes its state between solid and liquid or changes between two different solid crystallization states over a defined temperature range, absorbing or releasing heat during the phase change. This process is reversible and can be useful for thermal control in cold chain devices and products.
Removable container: A PCM container that is expected to be regularly removed from the CCE by the end user.
Reseller: A commercial entity, licensed to act on behalf of a legal manufacturer and which carries product liability and warranty responsibilities no less onerous than those carried by the legal manufacturer.

4. Applicability

Type-testing may be carried out by either the manufacturer or an independent ISO/IEC 17025 testing laboratory accredited by the World Health Organization (WHO). If the testing is carried out by the manufacturer, a WHO PQS representative shall visit the manufacturer prior to testing to verify that the manufacturer has the necessary equipment and has prepared for all proper procedures to complete the tests in this verification protocol. If the manufacturer does not have the necessary equipment or is not prepared to complete the testing, then testing must be performed by an independent testing laboratory as specified above.

If a manufacturer introduces a new container that that uses the same materials, wall thickness, sealing and manufacturing techniques with the same PCM as have been used in a different PCM container from the same manufacturer that has already passed this verification protocol and been approved by PQS, then type-

testing of the new container is not required. Changes to container design that will not trigger the type-testing requirement are limited to changes in container shape.

5. Type-testing procedure

5.1 Number of samples

If the testing is performed by an independent laboratory, the legal manufacturer or reseller must supply the testing laboratory with a full duplicate of the Product Dossier already supplied to WHO in accordance with the requirements of specification Clause 7.

A minimum of 15 samples of fixed containers that have not been integrated or affixed into the applicable CCE must be tested. If the testing is performed by an independent laboratory, the legal manufacturer or reseller must supply the samples to the laboratory. Samples must be tested with the same side upwards as they would have when installed in the CCE during the freeze/thaw and migration tests. If the container is used in multiple orientations with different sides upwards, an additional five samples per orientation per test are required.

5.2 Test procedure

5.2.1 Test 1: Type examination

- **Step 1:** Check all samples for similarities between different models,[1] dissimilarities between samples of one model and any physical or operational defects or damage that could affect form, fit, or function.
- **Step 2:** Record any differences between the samples ordered and those received.
- **Step 3:** Tabulate the following information for each model submitted for examination. Obtain any additional supporting information required in writing from the legal manufacturer or reseller and attach this information to the report.

Identification

i. Code (a unique identifier to be assigned by the testing laboratory).
ii. Model number as applicable.
iii. Legal manufacturer or reseller.
iv. Product type.
v. Country of origin.

Performance characteristics

vi. Nominal volume of container.
vii. Container filling and sealing conforms/does not conform to specification Clause 4.2.1.
viii. Container wall resistance to PCM migration conforms/does not conform to specification Clause 4.2.2 and does/does not pass verification test 5.2.3.

[1] The purpose of this inspection is to establish whether products offered by competing companies are re-badged versions of an otherwise identical device.

ix. Container material compatibility with PCM conforms/does not conform to specification Clause 4.2.3.
x. Container conforms/does not conform to robustness specification in verification tests 5.2.2 and 5.2.4.

Environmental requirements

xi. Ambient temperature range during transport, storage and use conforms/does not conform to specification Clause 4.3.

Physical characteristics – PCM hazards

xii. Physical hazards conforms/does not conform to specifications in Clause 4.4.1.
xiii. Health hazards conforms/does not conform to specifications in Clause 4.4.2.
xiv. Environmental hazards conforms/does not conform to specifications in Clause 4.4.3.

Container labelling

xv. Container conforms/does not conform to specifications in Clause 4.5.

Materials

xvi. Record materials used for container.
xvii. Materials conform/do not conform to specification Clause 4.6.

Warranty

xviii. Warranty conforms/does not conform to specification Clause 4.7.

Servicing provision

xix. Servicing provision conforms/does not conform to specification Clause 4.8.

Instructions

xx. Instructions conform/do not conform to specification Clause 4.9.

Disposal and recycling

xxi. Recycling and disposal information conforms/does not conform to specification Clause 4.11.

- **Step 4:** Take a three-quarter view digital photograph of one sample of each model.

Acceptance criteria: Inspection indicates full conformity with all specification document and verification protocol requirements.

5.2.2 Test 2: Freeze/thaw cycling

- **Samples:** Five new samples.

Note: During testing, samples must be positioned with the same side facing upwards as they would be if installed in the CCE. If the same container is used in multiple orientations in the CCE, this test shall be repeated for each distinct orientation with regards to the side facing upwards.

- **Step 1:** Clean and dry the outside of the samples to remove any PCM from the outside of the samples. Measure the initial mass of each sample (±0.1%).
- **Step 2:** Cool samples in freezer at -10°C until PCM is completely frozen.
- **Step 3:** Completely melt the PCM in the samples by placing samples in a chamber at +43°C.
- **Step 4:** Repeat steps 1 and 2 until 100 freeze-melt cycles have been completed.
- **Step 5:** Squeeze each sample by hand. Inspect samples for visible leaks and note any samples that have visibly leaked.
- **Step 6:** Clean and dry the outside of the samples. Measure the final mass of each sample (±0.1%).

Acceptance criteria: No samples visually exhibit leaking. Mass decrease between initial and final mass of each container is less than 1%.
Rejection criteria: One or more samples visually exhibit leaking. One or more samples decrease in mass by 1% or more.

5.2.3 Test 3: Migration test

Samples: Five new samples.

Note: During testing, samples must be positioned with the same side facing upwards as they would be if installed in the CCE. If the same container is used in multiple orientations in the CCE, this test shall be repeated for each distinct orientation with regards to the side facing upwards.

- **Step 1:** Clean and dry the outside of the samples to remove any PCM from the outside of the samples.
- **Step 2:** Place the samples into a temperature and humidity regulated chamber set at +43°C and 65% relative humidity. Leave samples at these conditions for five days to allow the moisture content of the PCM container to equilibrate with the humidity in the chamber.
- **Step 3:** Remove the samples from the environmental chamber. Clean and dry the outside of the samples to remove any PCM from the outside of the samples.
- **Step 4:** Measure the initial mass of each sample (±0.01%).
- **Step 5:** Return the samples to the environmental chamber and leave at +43°C and 65% relative humidity for 60 days.
- **Step 6:** Remove samples from environmental chamber. Clean and dry the outside of the samples to remove any PCM from the outside of the samples.
- **Step 7:** Measure the final mass of each sample (±0.01%).

- **Step 8:** Calculate the change in mass for each sample by subtracting the initial mass from the final mass. For each sample, calculate the percent change in mass by dividing the change in mass by the initial mass. Calculate the mean percent change in mass of the five samples, take the absolute value, and divide by two months to obtain the mean percent change in mass rate.

Acceptance criteria: The mean percent change in mass rate is less than 0.1% per month.
Rejection criteria: The mean percent change in mass rate is greater than or equal to 0.1% per month.

5.2.4 Test 4: Compression test

Reference: ISO 12048.

Samples: Five new samples.

- **Step 1**: Condition samples to +43°C.
- **Step 2**: For each sample, position a face/side of the sample on a flat, hard surface. Using a compression tester, apply an 800 N load over the entire surface that is facing upwards. Test each of the faces/sides shown in the following table by container geometry. Alternative sequences may have to be agreed upon for products that have different shape characteristics from those described, but three compressions should be carried out in all cases:

Rectangular	Cylindrical	Sack-shaped
1,6,3	Top surface, quadrant 1, quadrant 2	1,6,3

  Maintain the load for 30 seconds, then remove the load.
- **Step 4:** Inspect the sample for damage. Note any leaking.

Acceptance criteria: No visible leakage from any sample.
Rejection criteria: One or more samples leak.

5.3 Test criteria for qualification

A final report must be issued after all testing is complete. The report must contain the following data and analyses.

Summary: Conclusions and recommendations.

- **Test 1:** Provide general comments on the samples received, including comments on the overall standard of construction, tabulated results of the type inspection and photographs of samples.
- **Test 2:** Results of the freeze/thaw cycling test.
- **Test 3:** Results of the PCM migration test.
- **Test 4:** Results of the vacuum leak test.

- **Annexes:** Include a pre-approved test protocol verifying that the procedures set out in this document have been followed, a description of the test apparatus, test chamber temperature records and a copy of the reference thermometer calibration certificate(s). If testing was performed by an independent laboratory, include any additional supporting documentation requested and received from the legal manufacturer or reseller during the course of the type-testing.

6. Quality control checklist

6.1 Quality control standards

All testing and reporting must be carried out in accordance with the requirements of ISO 17025:2005 or later edition.

6.2 Quality control checklist

An on-site inspection of the manufacturing plant is not required.

6.3 Quality control evaluation

Not required.

7. Prequalification evaluation

A product will qualify for inclusion on the register of Performance, Quality and Safety (PQS) – prequalified equipment in accordance with WHO procedures provided the final report indicates full conformity with the requirements of specification **E005 PCMC 0.1**.

8. Modified products

The legal manufacturer or reseller is to advise WHO in writing of any changes to the materials, wall thickness, sealing, manufacturing techniques or PCM of the container after PQS prequalification has taken place. Any change that WHO believes would alter the test results obtained against the PQS verification protocol **E005 PCMC VP0.1** will result in a request for the container to be retested. Changes to container shape do not require notification of WHO or retesting of the container.

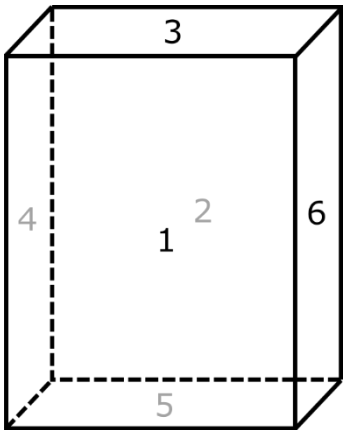
Revision history			
Date	**Change summary**	**Reason for change**	**Approved**
JUNE 2018	Document created	N/a	N/a

Annex 1: Container geometry notation

The following container geometry notation is used for the compression test and assumes that the container is approximately rectangular, cylindrical or sack-shaped. The notation is adapted from ASTM Standard D5276. On all Annex 1 figures, dashed lines and greyed numbers denote hidden edges and faces respectively. Dotted lines indicate quadrant boundaries.

For rectangular containers such as shown in Figure A1, faces are identified by a single number (e.g. the front face is one). If necessary, edges and corners are indicated by the numbers of the shared faces (e.g. 1-6 for the front, right edge; 1-3-6 for the front, top, right corner).

Figure A1: Rectangular-shaped container



The geometry of a cylindrical container is shown in Figure A2. The faces of a cylindrical container consist of two end faces (the top and bottom faces) and the side face. The faces are divided into four quadrants by two planes passing through the dotted lines with the planes perpendicular to the top face. If the side face has one or more seams, at least one of the seams should be centred in one of the quadrants. The chimes (circumferential edges) are the boundaries between the end faces and the side faces.

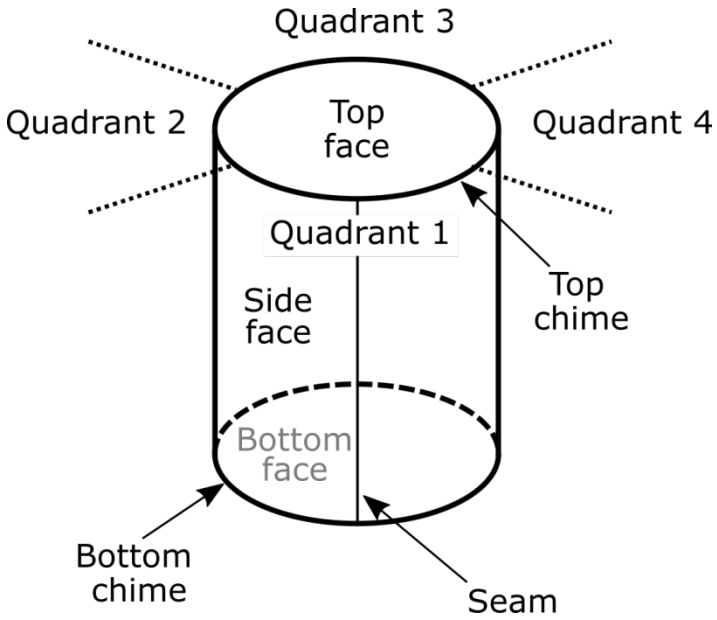


Figure A2: Cylindrical-shaped container

Since sack-shaped containers may not have distinct edges, the members are only referenced by the side number as shown in Figure A3.

Figure A3: Sack-shaped container