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PQS Independent type-testing protocol

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1. Scope

This document describes the procedure for verifying the performance of phase-change material (PCM) containers used in vaccine cold chain equipment (CCE) at ultra-low temperatures (ULTs) and includes verifying specifications for both the PCM itself and the container holding the PCM. This verification protocol addresses removable containers that are regularly removed from the CCE by end

users and contain the PCM generally used as the primary coolant materials for unpowered CCE.

Fixed containers that are not intended for removal from the CCE by the end users are not addressed under this verification protocol. Fixed containers are addressed in two other WHO-PQS documents: specification **PQS/E005/PCMC0.1** and the related verification protocol, **PQS/E005/PCMC01-VP0.1**. CCE includes equipment prequalified in category E004 – Cold boxes and vaccine carriers.

2. Normative references

(Use the most recent version)

DHHS (NIOSH) Publication No. 2019-113: Preventing cold-related illness, injury, and death among workers. By Jacklitsch B, Ceballos D. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health. DHHS (NIOSH) Publication No. 2019-113
ISO/IEC 17025:2017: 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.
OSHA 3408 – 2011: Laboratory Safety: Cryogens and Dry Ice Quick Facts
WHO/PQS/E005/PCMC02.1: Phase-change material containers – ultra-low temperature

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.
Nominal phase-change temperature: The nominal temperature at which the PCM changes phase as defined by the manufacturer.
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.
Ultra-low temperature (ULT): A temperature range attributed to materials that will be used at or lower than -25°C.

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 may 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 shall be performed by an independent testing laboratory as specified above.

If a manufacturer introduces a new container 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 Evidence of conformity assessment

Products shall carry the CE mark, UL, and/or equivalent internationally accepted evidence of conformity assessment.

5.2 Number of samples

If the testing is performed by an independent laboratory, the legal manufacturer or reseller shall 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 30 samples of removable containers shall be tested. If the testing is performed by an independent laboratory, the legal manufacturer or reseller shall supply the samples to the laboratory. During testing, samples shall be positioned such that any filling port, sealing weld or opening, or other structural weakness area is internally submerged within the PCM.

5.3 Test procedure

WARNING: Extreme Temperatures & Frostbite Risk[1]
Always handle ULT PCM containers with dry, thick gloves to protect hands and fingers from frostbite damage due to extremely low temperatures.

[1] Frostbite information paraphrased and quoted from OSHA 3408-2011 and DHHS (NIOSH) Publication No. 2019-113

- *Frostbite is a type of injury caused by freezing. It leads to a loss of feeling and colour in the areas it affects, usually extremities such as the nose, ears, cheeks, chin, fingers, and toes. Frostbite can permanently damage the body, and severe cases can lead to amputation.*
- *If you notice redness or pain in any skin area, get out of the cold or protect any exposed skin—frostbite may be beginning. Any of the following signs may point to frostbite:*
    - *A white or grayish-yellow skin area*
    - *Skin that feels unusually firm or waxy*
    - *Numbness*
- *A person who has frostbite may not know they have it until someone else points it out because the frozen parts of their body are numb. If you notice signs of frostbite on yourself or someone else, seek medical care.*

5.3.1 Test 1: Type examination

- **Step 1:** Check all samples for similarities between different models,[2] 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 or dimensions.
v. Country of origin.
vi. Conformity of assessment markings (e.g. CE mark or UL mark)

Performance characteristics

vii. Container filling and sealing conforms/does not conform to specification clause 4.2.2.
viii. Container material compatibility with PCM conforms/does not conform to specification clause 4.2.5.

Environmental requirements

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

Phase-change material hazards

x. Physical hazards conforms/does not conform to specifications in clause 4.6.1.

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

xi. Health hazards conforms/does not conform to specifications in clause 4.6.2.
xii. Environmental hazards conforms/does not conform to specifications in clause 4.6.3.

Container labelling

xiii. Container conforms/does not conform to specifications in clause 4.7.

Materials

xiv. Record materials used for container.
xv. Materials conform/do not conform to specification clause 4.8.

Warranty

xvi. Warranty conforms/does not conform to specification clause 4.9.

Servicing provision

xvii. Servicing provision conforms/does not conform to specification clause 4.10.

Instructions

xviii. Instructions conform/do not conform to specification clause 4.11.

Training

xix. Training conforms/does not conform to specification clause 4.12.

Disposal and recycling

xx. Recycling and disposal information conforms/does not conform to specification clause 4.13.

- **Step 4:** Photograph all sides of one sample from each model.

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

5.3.2 Test 2: Dimensions and weights

Samples: Ten new samples

Test conditions: Testing room at +21°C (±3°C). Record and report conditions at the time of the test.

- **Step 1:** Label each sample and record its weight in grams, ± 1.0 gram.
- **Step 2:** Record external dimensions of each sample (length, width and height for rectangular packs or maximum thicknesses in the x, y, and z directions if different geometry is used) in mm, ± 0.5 mm.

5.3.3 Test 3: Freeze/thaw cycling

WARNING: Extreme Temperatures & Frostbite Risk

Samples: Ten new samples.

Test Conditions: If using two testing chambers, one test chamber (or freezer) should be set below the nominal phase-change temperature (as specified as necessary for freezing the PCM). Record and report conditions at the time of the test.

Note: During testing, samples shall be positioned such that any filling port, sealing weld, or other structural weakness area is internally submerged within the PCM.

- **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 (±0.1g).
- **Step 2:** Freeze samples in freezer set below the nominal phase-change temperature (as specified as necessary for freezing the PCM by the manufacturer) until PCM is completely frozen.
- **Step 3:** Completely thaw 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:** After final thaw, at laboratory room temperature, 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 g).

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.

5.3.4 Test 4: Migration test

Samples: Ten new samples.

Note: During testing, samples shall be positioned such that any filling port, sealing weld, or other structural weakness area is internally submerged within the PCM.

Test conditions: Test chamber at +43°C ± 5°C and ambient humidity. Record and report conditions at the time of the test including, at a minimum, temperature and humidity.

- **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 the test chamber. 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 test 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 test chamber and leave for 60 days.
- **Step 6:** Remove samples from test 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 60 days to obtain the mean percent change in mass rate.

Acceptance criteria: The mean percent change in mass rate is less than 0.1% per 30 days.

Rejection criteria: The mean percent change in mass rate is greater than or equal to 0.1% per 30 days.

5.3.5 Test 5: Frozen container thickness and adhesion test

WARNING: Extreme Temperatures & Frostbite Risk

Samples: Ten new samples

Test conditions: Freezer set below the nominal phase-change temperature (as specified as necessary for freezing the PCM by the manufacturer). Testing room or chamber at +21°C (±3°C), and ambient humidity. Record and report conditions at the time of the test including, at a minimum, temperature and humidity.

- **Step 1**: Label each sample with a unique identifier (e.g. letter them A-E or other identification) that will remain readable through and after freezing and thawing.
- **Step 2**: **Prep for frozen container drop test -** Mark each container's faces, corners, edges, or any combination thereof with test numbers such that specific members on each sample can be identified after the container's orientation has been changed. See recommended labeling pattern in **Annex 2**.
- **Step 3**: Stack PCM containers on top of one another in the freezer set below the nominal phase-change temperature (as specified as necessary for

freezing the PCM by the manufacturer) and freeze until PCM is completely frozen.

- **Step 4**: Remove frozen PCM containers from the freezer. Record whether they adhere to one another and the extent to which they must be pulled apart.
- **Step 5**: Measure and record the thickness of the frozen PCM containers (±1.0mm).
- **Step 6**: Thaw the PCM containers at room temperature until they reach +5°C ± 5°C or warmer. Measure and record the thickness of the thawed PCM containers (±1.0mm).
- **Step 7**: Return the PCM containers to the freezer until completely frozen in preparation for the subsequent, frozen container drop test.

Acceptance criteria: Increase in sample thickness due to swelling does not exceed the measured dimensions from dimensions and weights test by more than 25% for any of the samples. Thickness of each of the thawed samples equals the measured thickness of the same sample from dimensions and weights test (±1.0mm). Containers do not adhere to one another when frozen to the extent that they cannot be pulled apart by gloved hands without supplemental tools.

Rejection criteria: One or more frozen samples exceed the permitted increase in thickness and/or one or more thawed samples fail to return to the pre-frozen thickness. Containers adhere strongly to one another when frozen such that supplemental tools are required to separate them.

5.3.6 Test 6: Frozen container drop test

WARNING: Extreme Temperatures & Frostbite Risk

Test conditions: Testing room at +21°C (±3°C). Record and report conditions at the time of the test.

Samples: Ten samples, labelled and frozen PCM containers from frozen PCM container thickness and adhesion test.

- **Step 1**: Remove each PCM container one at a time from the freezer immediately before testing and proceed to drop testing within 5 minutes so that the container remains as cold as realistically possible during the drop test.
- **Step 2**: If not already accomplished, mark each container's faces, corners, edges, or any combination thereof with test numbers such that specific members on each sample can be identified after the container's orientation has been changed. See recommended labeling pattern in **Annex 2**.
- **Step 3**: Using a free fall drop tester, drop each container from a height of 1.0 metres (measured from the lowest part of the container at the start of each test) onto a smooth, dense concrete floor in the following numerical order:

Face	Edges	Corners
1 Flat face top	2 Top short edge	3 Top left corner
4 Flat face bottom	5 Bottom short edge	6 Top right corner
	7 Left long edge	8 Bottom left corner
	9 Right long edge	10 Bottom right corner

Cancel the relevant test number marking after each drop to avoid inadvertent duplication.

- **Step 4**: Fully thaw all ten containers to room temperature. Take photographs of all sides of one sample from each model.
- **Step 5**: Take separate, detailed photographs of any areas of damage, physical change, or deformation. These photos shall be of high quality, with ample light and nearness such that small details can be made out easily.
- **Step 6**: Set aside all samples for compression test. Check each container for leaks.

Acceptance criteria: No visible leakage from any sample.

Rejection criteria: One or more samples visibly leaks.

5.3.7 Test 7: Compression test

Reference: ISO 12048.

Test conditions: Testing room at +21°C (±3°C). Record and report conditions at the time of the test.

Samples: Samples from frozen container drop test.

- **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 (see **Annex 1** for geometry notation). 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 3:** Inspect the sample for damage. Note any leaking.
- **Step 4:** Take photographs of all sides of one sample from each model.
- **Step 5:** Take separate, detailed photographs of any areas of damage, physical change, or deformation. These photos shall be of high quality, with ample light and nearness such that small details can be made out easily.
- **Step 6:** Check each container for leaks.

Acceptance criteria: No visible leakage from any sample.

Rejection criteria: One or more samples visibly leak.

5.4 Test criteria for qualification

A final report shall be issued after all testing is complete. The report shall 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 dimensions and weights test. Provide note on if PCM containers are compatible with existing WHO PQS prequalified CCE and, if not, provide documentation that PCM containers are not intended or marketed as compatible with such CCE.
- **Test 3:** Results of the freeze/thaw cycling test.
- **Test 4:** Results of the PCM migration test.
- **Test 5:** Results of the frozen container thickness and adhesion test
- **Test 6:** Results of the frozen container drop test. Provide photographs of samples from step 3 of this test.
    - If any sample exhibits visible deformation or physical change from before the drop test, provide specific photos of each area with such deformation or physical changes.
- **Test 7:** Results of the compression test. Provide photographs of samples from step 4 of this 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 shall be carried out in accordance with the requirements of **ISO 17025**.

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/PCMC02.1**.

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 PCMC02 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.

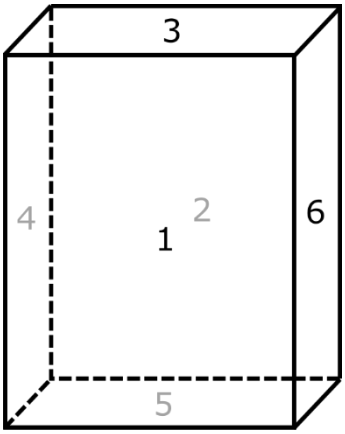
Revision history			
Date	**Change summary**	**Reason for change**	**Approved**
02/04/2021	Document created	N/a	N/a

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.

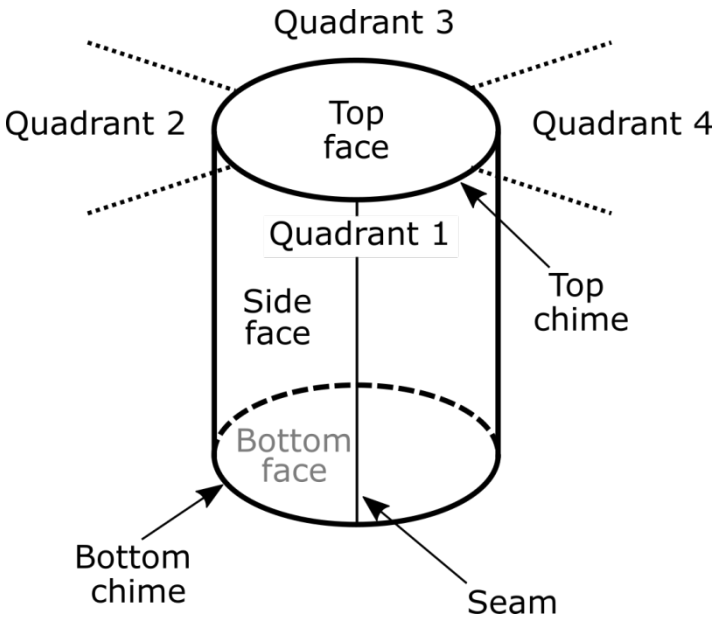
For rectangular containers such as shown in Figure A1.1, faces are identified by a single number (e.g. the front face is one). If necessary, edges and corners are indicated by the numbers of the shared faces (e.g. 1-6 for the front, right edge; 1-3-6 for the front, top, right corner).

Figure A1.1: Rectangular-shaped container



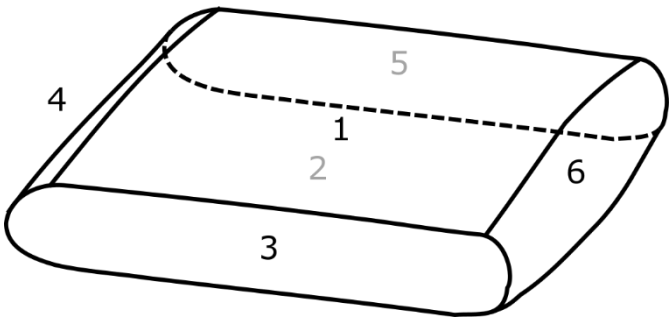
The geometry of a cylindrical container is shown in Figure A1.2. The faces of a cylindrical container consist of two end faces (the top and bottom faces) and the side face. The faces are divided into four quadrants by two planes passing through the dotted lines with the planes perpendicular to the top 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 A1.2: 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 A1.3.

Figure A1.3: Sack-shaped container



Annex 2: Recommended Labeling Pattern for PCM Container Drop Tests

Figure A2.1

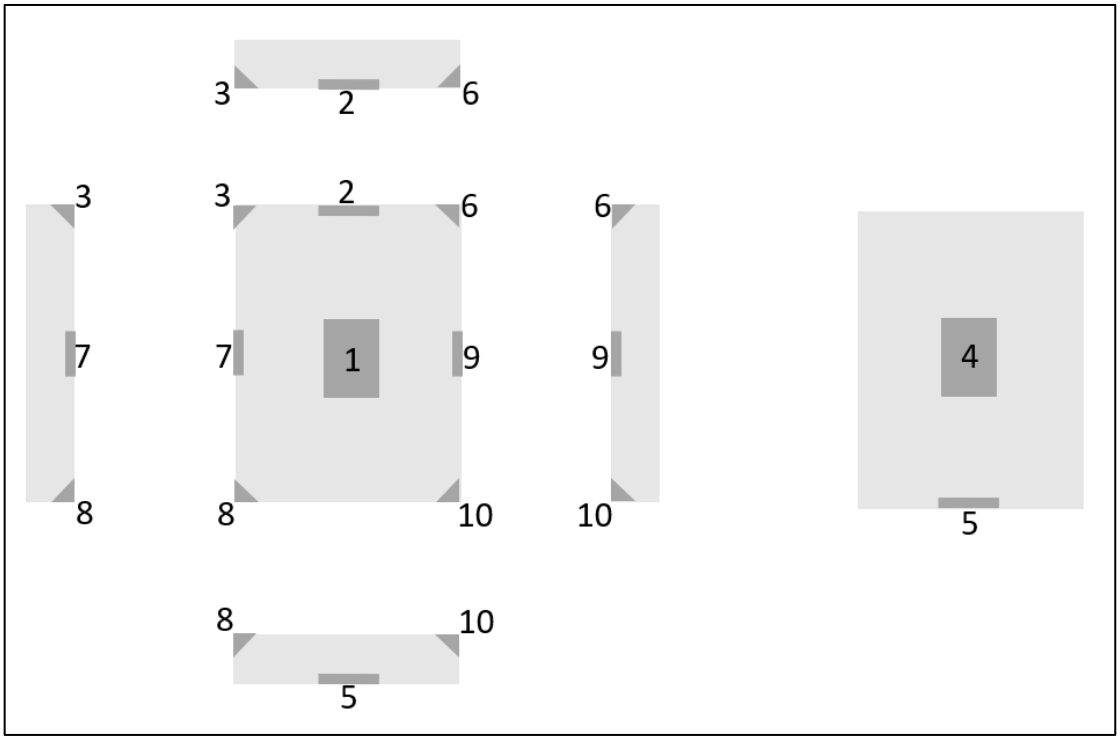


Figure A2.2