



VULCANISED RUBBERS AND THERMOPLASTIC COMPACT OR CELLULAR RUBBERS AGEING IN FLUIDS AND GREASES

THIS NORM REPLACES THE NORMS D47 1118 AND D47 1408

FOREWORD

This document is equivalent to document RENAULT D47 1098. It must not be modified without prior consultation with the Standards Department of this Group.

It is in conformity with the agreement reached between this group and PSA PEUGEOT CITROËN in OCTOBER 1996.

1.OBJECT AND FIELD OF APPLICATION

The purpose of this method is to characterise the influence of the extended contact of a fluid or grease, at a given temperature, on the characteristics of a vulcanized rubber or a thermoplastic, compact or cellular rubber. This method is based on standard NF T 46-013.

2.PRINCIPLE

The following physical characteristics are determined beforehand on new specimens taken from plates or parts:

- volume
- dimensions
- mechanical tensile characteristics
- hardness.

After immersion in a fluid or grease, at a given temperature and for a given time, the variation in the characteristics indicated above is measured in comparison with the initial condition.

3.APPARATUS

3.1.CONTAINERS

That must contain the specimens and the fluids or greases.

- For volatile fluids like fuels, mineral hydraulic liquids, window washing liquids, cooling liquids, etc., use a hermetically closed container defined in Appendix 1.
Note: Choose a container and a closing system capable of withstanding the corresponding internal pressure, depending on the test temperature.
- For greases, choose a container with lid (weighing bottles) with a size such that the specimens are completely covered and all their sides are exposed and can swell or shrink without stress.
- The containers used must be equipped with a specimen holder (3.2) so that the specimens are in contact with the fluid on their entire surface.

3.2.SPECIMEN-HOLDER

This assembly must not have any action either on the rubber or on the fluid and must not hinder the free deformation of the specimens, see Appendix 2.

3.3.DENSITY BALANCE OR HYDROSTATIC BALANCE

Accurate to one centigram.

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3.4.RULER

Graduated in half millimetres.

3.5.COMPARATOR

Accurate to one-tenth of a millimetre, with a load pressure less than 20 kPa.

3.6.SHORE AND D.I.D.C. DUROMETER

Conforming to those described in test methods D45 1290 and D45 1291.

3.7.TENSILE TESTING MACHINE

conforming to the one mentioned in the Test method D45 1099.

3.8.CONDITIONED CHAMBER

at 23 °C ± 2 °C and 50 % ± 5 % relative humidity.

3.9.VENTILATED OVEN

That can be regulated, to the nearest degree, up to 300 °C.

3.10.TEMPERATURE RECORDER

Equipped with thermocouples for controlling the temperature.

3.11.LINT-FREE ABSORBENT PAPER

3.12.LINT-FREE FABRIC

3.13.VACUUM OVEN

4.REAGENTS

Depending on whether plates or parts are used, choose one or more of the products indicated below for the ageing. For approval of a rubber, the tests must be performed with approved products and with a reference product, if any. For normal verifications, the reference product may alone be used.

4.1.BRAKE FLUIDS

Approved liquids whose exact references must be indicated in the test report.

4.2.MINERAL HYDRAULIC LIQUIDS

Approved liquids whose exact references must be indicated in the test report.

4.3.OILS

Conforming to ISO standard 1817.

4.4.COOLING LIQUIDS

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Approved liquids whose exact references must be indicated in the test report.

Note: *The product must be composed of 50 parts in volume of pure ethylene glycol and 50 parts in volume of distilled water.*

4.5.WINDOW-WASHING FLUID

Products	Composition
Reference liquid no. 1	Distilled water 27 parts in volume Ethanol 73 parts in volume Wetting agent * 0.2 g for 100 g of fluid
Reference liquid no. 2	Distilled water 85 parts in volume Ethanol 15 parts in volume Wetting agent * 0.2 g for 100 g of fluid
Approved liquid	Exact references, with concentration in water in case of dilution, to be indicated in the test report

* *The wetting agent is sodium dodecylbenzenesulfonate.*

4.6.FUELS

Products	Composition
Reference diesel fuel	Oil no. 3 or IRM903 oil 90 parts in volume Paraxylene 10 parts in volume
Fluid C *	Trimethyl-2,2,4 pentane (isooctane) 50 parts in volume Toluene 50 parts in volume
N category fluid *	Fluid C 90 parts in volume Ethanol 7 parts in volume Methanol 3 parts in volume
O category fluid *	Fluid C 85 parts in volume Methanol 15 parts in volume

* *Respectively liquid C, liquid no. 3 and liquid no. 4 according to norm NF T 46-013.*

4.7.DISTILLED WATER

4.8.SURFACE ACTIVE AGENT

Wetting agent

4.9.SOLVENT

Acetone for instance.

4.10.APPROVED GREASES

The exact conditions must be indicated in the test report.

5. PREPARING THE SPECIMENS

In all cases, work on at least 3 specimens.

5.1. FORM OF THE SPECIMENS

5.1.1. DETERMINATION OF THE VARIATION IN VOLUME OR LINEAR VARIATIONS (COMPACT RUBBERS)

- The specimen must have a volume of 1 cm³ to 3 cm³. Its thickness must be uniform and range between 1.5 mm and 2.5 mm, measure using the comparator (3.5).
- When the specimen is taken from a plate, it must be rectangular and the largest of its dimensions must not exceed 50 mm, measured using the ruler (3.4).
- Often, if the test is performed on specimens taken from parts, the dimensions required above cannot be respected. Note down in the test report the dimensions of the specimen used, and, where applicable, the place on the part from which it is taken.

5.1.2. DETERMINATION OF THE VARIATION IN MECHANICAL TENSILE CHARACTERISTICS

Work on dumb-bell shaped tensile specimens, according to D41 1099.

5.1.3. DETERMINATION OF THE HARDNESS AFTER IMMERSION

Work on the specimens according to paragraphs 5.1.1 and 5.1.2 by superposing several plates to achieve the required minimum thickness indicated in test method D45 1290 for D.I.D.C. hardness and in test method D45 1291 for Shore hardness.

5.2. CONDITIONING OF THE TEST SPECIMENS

Condition the specimens for three hours in the chamber (3.8.).

6. OPERATING PROCEDURE

Perform the test in the chamber (3.8).

6.1. PRELIMINARY OPERATIONS

6.1.1. DETERMINATION OF THE VARIATION IN VOLUME

- Carefully wipe the specimens using paper (3.11) to eliminate impurities and small rubber fragments.
- Weigh each specimen in air using the balance (3.2); let M1 be their weight.
- Add a small quantity of surface active agent (4.8) to the water (4.7).
- Weigh each specimen in water, ensuring that there is no air bubble, using the balance (3.2); let M2 be their weight.
- Wipe the specimens using the paper (3.11).

6.1.2. DETERMINATION OF THE VARIATION IN TENSILE CHARACTERISTICS

Proceed according to the operating procedure prescribed by the test method D41 1099 and determine the tensile characteristics, like rupture strength, rupture elongation and stress for a given elongation in the initial condition as well as the transversal section of the three specimens to be subjected to ageing.

6.1.3. DETERMINATION OF THE VARIATION IN HARDNESS

Measure the specimen's hardness according to one of the test methods D45 1290 for D.I.D.C. hardness or D45 1291 for Shore hardness, using durometers (3.6).

6.2.AGEING

- Preferably choose the ageing temperature and duration according to norm NF T 46-013 or, otherwise, in accordance with the documents.
- For ageing in a fluid, place the specimens in the specimen-holder (3.2).
- Fully immerse the holder and the specimens in the fluid or grease, ballasting them if necessary and avoiding any contact between the specimens.
The fluid volume must be greater than or equal to 15 times that of the specimens and at the most $\frac{3}{4}$ th of the volume of the container (3.1).
The grease volume must be two to five times the volume of the specimens.
Note: *Never place specimens of different rubbers in the same container. Similarly, a fluid placed in contact with specimens must never be reused for another ageing.*
- Store the specimens away from light, during the immersion.
Plug the container (3.1) after placing the specimens and place it in the oven (3.9) regulated to the temperature required by the documents, by placing the thermocouple, fitted with the temperature recorder (3.10), near the container.
- Remove the container (3.1) from the oven (3.9) after the immersion period and allow them to return to the temperature of the chamber (3.8); then take the measurements.

6.3.MEASUREMENTS

Remove the specimens from the container (3.1) within 4 to 5 hours after removing the container (3.1) from the oven (3.9).

6.3.1.CASE OF GREASE

Remove the specimens from the container (3.1); wipe them using paper (3.11) and determine the variations in characteristics immediately or after drying, if this is required in the documents.

6.3.2.CASE OF A VOLATILE FLUID (FUEL)

6.3.2.1.Without drying

The test must be completed 5 minutes after removing the specimen from the test fluid.

6.3.2.2.With drying

Arrange the specimens flat and allow them to remain $24 \text{ h} \pm 1 \text{ h}$ in the chamber (3.8).

Dry the specimens according to one of the following conditions:

- $24 \text{ h} \pm 1 \text{ h}$ at $23 \text{ °C} \pm 2 \text{ °C}$ in vacuum in the oven (3.13),
- $24 \text{ h} \pm 1 \text{ h}$ at $70 \text{ °C} \pm 1 \text{ °C}$ in vacuum in the oven (3.13),
- $48 \text{ h} \pm 1 \text{ h}$ at $70 \text{ °C} \pm 1 \text{ °C}$ in a ventilated oven (3.9).

6.3.3.CASE OF A HARDLY VOLATILE FLUID

Eliminate the excess fluid or grease on each test specimen, directly using the paper (3.11) or fabric (3.12) or after quickly dipping it in the appropriate solvent (4.9) if the fluid is viscous.

After one hour maximum, weigh the specimens in air and in distilled water (4.7), using the balance (3.2), to determine the variation in volume; Let M3 and M4 be their weight respectively.

Determine the tensile characteristics according to Test method D41 1099, D.I.D.C. hardness according to test method D41 1290 and Shore hardness according to Test method D41 1291.

7.EXPRESSION OF RESULTS

7.1.VARIATION IN VOLUME

The variation in volume of each specimen $\frac{\Delta V}{V}$, expressed in percentage (%), is obtained using the following formula:

$$\frac{\Delta V}{V} = \frac{(M3 - M4) - (M1 - M2)}{M1 - M2} \times 100$$

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where:

- M1 = initial weight of the specimen in air, expressed in grams (g)
- M2 = initial weight of the specimen in distilled water, expressed in grams (g)
- M3 = weight of the specimen after processing in air, expressed in grams (g)
- M4 = weight of the specimen after processing in distilled water, expressed in grams (g)

Note down the arithmetic mean of the results obtained on the 3 specimens.

7.2.VARIATION IN TENSILE CHARACTERISTICS

The variations in tensile characteristics $\frac{\Delta X}{X}$, expressed in percentage (%) (rupture strength R_r , rupture elongation A_r and stress for a given elongation C_x), are obtained using the formula below and by respecting the signs:

$$\frac{\Delta X}{X} = \frac{X_0 - X_1}{X_0} \times 100$$

where:

- X_0 = initial characteristic (average of 3 specimens), expressed in megapascals (MPa) or in percentage (%),
- X_1 = characteristic after ageing (average of 3 specimens), expressed in megapascals (MPa) or in percentage (%),

Note:

- Rupture strength and stress for a given elongation are determined with respect to the initial cross-section of the specimen.
- The markers used for determining rupture elongation are drawn after ageing on the calibrated part of the specimen.
- It is often useful to express the absolute values of the characteristics after ageing, i.e. respectively R_r , A_r and C_x for a stress for a given elongation x %.

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7.3.VARIATION IN HARDNESS

For hardness, it is generally more useful to have the absolute value of the hardness after ageing rather than the variation.

As a result, express the hardness measured after ageing or if necessary the algebraic difference between the hardness measured after ageing and the initial hardness.

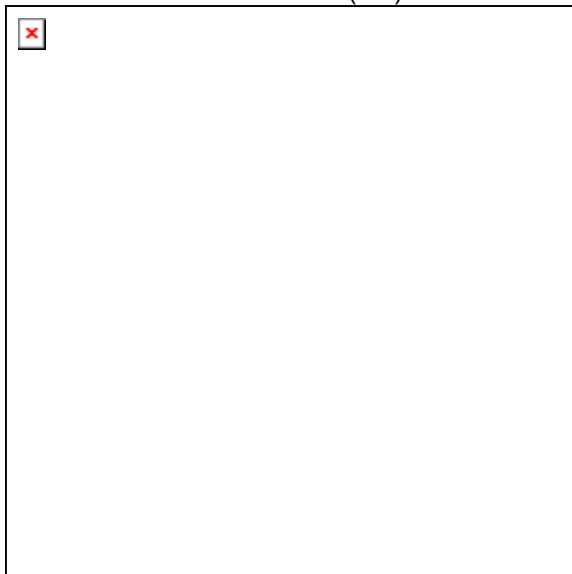
8.TEST REPORT

In addition to the obtained results, the test report must indicate:

- the reference of this method
- the exact reference of the rubber examined and the name of the supplier
- the test temperature
- in the case of a fluid other than one of the reference fluids, indicate its exact commercial reference and the name of its supplier
- test duration
- initial thickness and dimensions of the specimen; in the case of specimens cut from parts, indicate the place from which the sampling is done
- Any abnormal change in appearance of the specimen apart from variations in dimension (cracking, sticky surface, ...),
- any change in appearance of the immersion liquid (fluorescence, sedimentation, etc.)
- if the duration between the return to ambient temperature and the measurement is other than 4 hours (tolerance 0 hours to - 1 hour), indicate this duration
- procedural details that are not specified in the method, as well as any incidents that might have influenced the results.

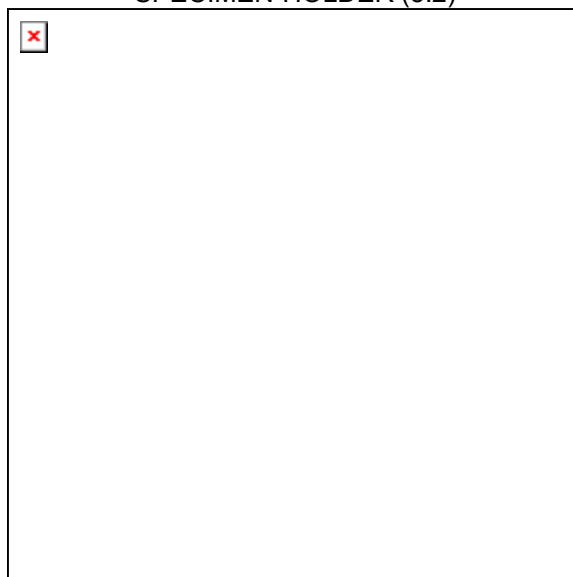
Appendix 1

CONTAINER (3.1)



Appendix 2

SPECIMEN-HOLDER (3.2)



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9.RECORDS AND REFERENCE DOCUMENTS

9.1.RECORDS

9.1.1.CREATION

- OR: 01/06/1978 - CREATION OF THE PSA NORM. REPLACES THE ASSOCIATION NORM No.1098

9.1.2.SUBJECT OF THE MODIFICATION

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9.2.REFERENCE DOCUMENTS

9.2.1.PSA DOCUMENTS

9.2.1.1.Norms

D411099, D451290, D451291.

9.2.1.2.Other

9.2.2.EXTERNAL DOCUMENTS

NF T 46-013 (12/1985) ISO 1817 (03/1985)

9.3.EQUIVALENT TO:

9.4.CONFORMS TO:

9.5.KEYWORDS