

**PAINTS
CLEANLINESS
(FILTRATION)**

Page 1/6

NO USE RESTRICTION*This is a translation, the French original shall be used in all cases of litigation**Date of translation : 14/10/1996***1. OBJECT AND FIELD OF APPLICATION**

The object of this méthode is to describe a method of operation for determining the cleanliness characteristics of paint before being connected to a distribution circuit.
This applies to solvent based, concentrated or diluted paints with the exception of pearlescent paints.

2. PRINCIPLE

Filtration of a known volume of product through a membrane of known porosity and quantification of impurities collected on the membrane.

3. EQUIPMENT AND REAGENTS**3.1. VERTICAL LAMINAR FLOW HOOD**

With filtration threshold lower than 0,3 μm and 99,9% efficiency.

3.2. CALIBRATED GLASS VACUUM FLASK

Of 5 litre capacity.

3.3. VENTURI

Equipped with a graduated manometer from 0 - 1 bar.

3.4. FILTER SUPPORT

Two possibilities :

3.4.1. PALL SUPPLIER

- Disc holder according to PALL drawings : E61041, D72083 and D72084.
- Support membrane of 450 μm with a control valve.
- Two O-rings 39,34 T 2,62, in fluorocarbonated rubber.

3.4.2. BIOCLOCK SUPPLIER

- BIOBLOCK filter support, reference PFA 47 mm.
- PFA Teflon connectors for 8 mm diameter tube, reference VOR 44730 PT6-147-6.

Note. When seals are required, they must be used for a single purpose. Note that the use of seals may generate rubber particles which must not be attributed to the tested product.

3.5. RIGIMESH FILTER MEMBRANES

Supplier PALL, of 45, 70 and 105 µm.

3.6. FILTER MEMBRANES

Multipore N66 of 0,8µm, supplier PALL or MILLIPORE of 1 µm.

3.7. SAMPLING ROD

With a height reference-mark for drums or sampling system adapted to the container valves.

3.8. PIPE

In Teflon or Rilsan, 8 mm diameter for the link between the rod (3.7) or the sampling system and the filter support (3.4).

This pipe must be used for a single purpose.

3.9. TUBE

In stainless steel or Teflon, 8 mm external diameter, to be fitted at the filtration system output.

3.10. TWEEZERS**3.11. PETRI DISHES**

In glass, 50 mm diameter with a lid.

3.12. WASH BOTTLES

For each solvent used.

3.13. CHRONOMETER

To one tenth of a second.

3.14. BINOCULAR MICROSCOPE

With a zoom lens for obtaining a 25 and 50 x magnification, equipped with a micrometer eyepiece and a displacement table 2D.

3.15. DILUTION SOLVENT RESERVE

Of a 5 litre minimum capacity, with a cover, to be connected to the filter support (3.4).

3.16. ULTRASONIC TANK**3.17. OVEN AT 100°C ± 1°C.****3.18. RINSING SOLVENTS**

Compatible with the products to be tested.

Note. *In all cases, the rinsing solvent must be defined by the Quality Department with the agreement of the supplier concerned.*

4. METHOD OF OPERATION

4.1. SAMPLE

- One drum or product container per batch, in relation to conditioning.
- For drums, a prior planetary stirring of 30 minutes is required. By derogation, a 30 minute stirring on a roller may be allowed.
- For containers, the prior stirring is 60 minutes.

Note. *When no stirring system is available within the production plant, sampling without stirring may be carried out. This procedure is however considered as a retrograde procedure.*

4.2. CLEANING OF THE EQUIPMENT

- Prepare, under the hood (3.1), a sufficient quantity of solvent (3.18) compatible with the product to be tested.
- Filter this solvent through the filter support (3.4) fitted with a membrane of 0,8 or 1 µm (3.6).
- Clean successively the vacuum flask (3.2), the filter support (3.4), the pipe (3.8), the tube (3.9), the tweezers (3.10), the Petri dish (3.11), a pipette (3.12) and the reserve (3.15) with the filtered solvent [use, if required the ultrasonic tank (3.16)].
- Fill the wash bottle (3.12) and the reserve (3.15) with the solvent previously filtered.

4.3. CHOICE OF MEMBRANE (3.5)

This choice is made according to the product to be tested.

- Membrane of 105 µm (3.5) : metallic paints.
- Membrane of 45 µm (3.5) : opaque paints, finish paints, intermediate paints and varnishes.

Note. *In the case of filtering problems, by derogation and after agreement with the supplier, a membrane of 70 µm (3.5) may be used for intermediate paints.*

4.4. CHECKING THE MEMBRANE

- Never handle the membrane.
- Store membranes (3.5) and (3.6) in a clean Petri dish (3.11).
- Check the cleanliness of the membrane to be used with the binocular microscope (3.14), if required, humidify the membrane with a filtered dilution solvent before checking.
- Only use thoroughly clean membranes for the test.

4.5. POSITIONING THE MEMBRANE

- With the tweezers (3.10), place the membrane in the filter support (3.4).
- Close the filter support.

4.6. CONNECTION

4.6.1. DRUMS

- Connect the pipe (3.8) to the connector upstream of the filter support (3.4).
- Place the sampling rod (3.7) into the drum in a vertical position :
 - half way into the quantity contained in the drum if the latter has previously been stirred,
 - at 15 centimetres from the bottom of the drum (by agreement if the latter could not be stirred).
- Fit the pipe (3.8) to the sampling rod (3.7).

4.6.2. CONTAINERS

- If the container is equipped with a stirring device, purge approximately 2 litres of product before carrying out the sampling.
- If the container is not equipped with a stirring device, purge approximately 10% of its volume before carrying out the sampling.
- Connect the filter support (3.4) to the container using the pipe with the specific coupling.

4.7. SAMPLING

- Create a vacuum which must be adjusted between 0,5 and 0,8 bar.
- Open the valve progressively so as to obtain a flow between 200 and 500 ml/min.
- Close the valve after filtering and collecting 2,5 litres of product in the flask (3.2).
- Stop the vacuum.
- Connect the pipe (3.8) to the supply system of clean rinsing solvent obtained in § 4.2.

4.8. RINSING

- Create a vacuum.
- Open the valve progressively so as to obtain a flow between 800 and 1000 ml/min.
- Rinse the membrane with a minimum of 2,5 litres of solvent or if required the quantity necessary to obtain a thorough wash of the filter (visual check of the membrane), 6 to 7 litres may be required.
- Ensure that the solvent runs out perfectly clear at the end of the rinsing operation.
- Close the solvent inlet.
- Disconnect the inlet and outlet pipes.

4.9. INSPECTION OF THE MEMBRANE

- Under the hood (3.1) disconnect the filtering system and retrieve the membrane with the tweezers (3.10).
- Place the membrane in the Petri dish (3.11) closed with its lid.
- Dry the membrane, in the Petri dish, in the oven (3.17) for 30 minutes.
- Remove the Petri dish from the oven and leave to cool without removing the lid.
- The inspection must be made with the binocular microscope (3.14) under a x 25 magnification by default or x 50 magnification.
- Observe the membrane in the Petri dish by sweeping over the whole surface.
- Observe, count and measure all the particles and assess the developed length of actual fibres.
- If grading becomes difficult, it is recommended to take a photograph in order to be able to discuss this with the supplier.
- The product reference, the batch number of the container or drum as well as the scale of the photograph must be stated on the back and enclosed in the test report.

5. EXPRESSION OF RESULTS

The cleanliness of a paint is characterised by the absence of foreign bodies (fibres, particles, etc.) and agglomerates.

The result must be expressed by the number of foreign bodies or agglomerates corresponding to the various requirement categories in the documents.

6. ACCURACY

6.1. REPEATABILITY

The relative difference between 2 individual results obtained in conditions of repeatability and with a probability defined at 95% must not exceed 100%.

6.2. REPRODUCIBILITY

The relative difference between 2 individual results obtained in conditions of reproducibility and with a probability defined at 95% must not exceed 200%.

7. TEST REPORT

As well as the results obtained, the test result must indicate :

- the reference to this méthode,
- the complete identification of the paint and the name of the supplier,
- the complete identification of the membrane and the filter support used,
- the complete identification of the rinsing solvent,
- the operating details not specified in the method as well as any possible incidents likely to have affected the results.

8. RECORDS AND REFERENCE DOCUMENTS

8.1. RECORDS

8.1.1. CREATION

- OR : 16/10/1996 – CREATION OF THE NORME

8.1.2. SUBJECT OF THE MODIFICATION

-
-

8.2. REFERENCE DOCUMENTS

8.2.1. PSA DOCUMENTS

8.2.1.1 Normes

8.2.1.2. Others

8.2.2. EXTERNAL DOCUMENTS

8.3. EQUIVALENT TO :

8.4. CONFORMS TO :

8.5. KEY-WORDS