PET-P0 BOTTLE TEST PROTOCOL

Regeneration by mechanical recycling of PET household bottle and dispenser bottle packaging

COTREP

The mission of the Technical Committee for the Recycling of Plastic Packaging (COTREP) is to help designers and decision-makers develop recyclable plastic packaging while also providing scope for innovation. The committee includes various stakeholders in the plastic household packaging chain (Valorplast, Elipso, Citeo and SRP) and works on all types of plastic packaging (bottles, dispenser bottles, pots and trays, films and flexible packaging). Protocols for tests performed by COTREP are devised based on work with stakeholders in plastic household packaging end-of-life.

VERSION NO.	DATE	DESCRIPTION
1	September 2007	*Initial version*
2	December 2025	*General protocol update*

1. CONTEXT

This protocol is an update of the protocol drawn up by COTREP for the regeneration of PET bottles.

It is representative of industrial practices applied by regeneration plants processing French streams. Its purpose is to specify tests to be performed to assess the suitability of a PET bottle packaging for mechanical regeneration in the industrial stream. This step forms an essential part of the overall recyclability assessment for packaging. If the results of this step are conclusive, the assessment should be continued by implementing a PET-P4 (bottle application), PET-P1 (fibre application), PET-P2 (sheet application) or PET-P3 (strapping application) protocol depending on the target application.

Results obtained from tests described below may be submitted to COTREP for analysis and potential inclusion in French eco-design recommendations aimed at improving recyclability.

The tests performed according to the EPBP protocol are similar to COTREP tests and mainly differ in terms of market penetration rates and the standard material used. COTREP specifically endeavours to translate actual marketing conditions in France and analyse the impact of packaging on recycled material produced by industrial facilities. The results of EPBP protocols may be considered by COTREP with a specific analysis of representativeness on the market.

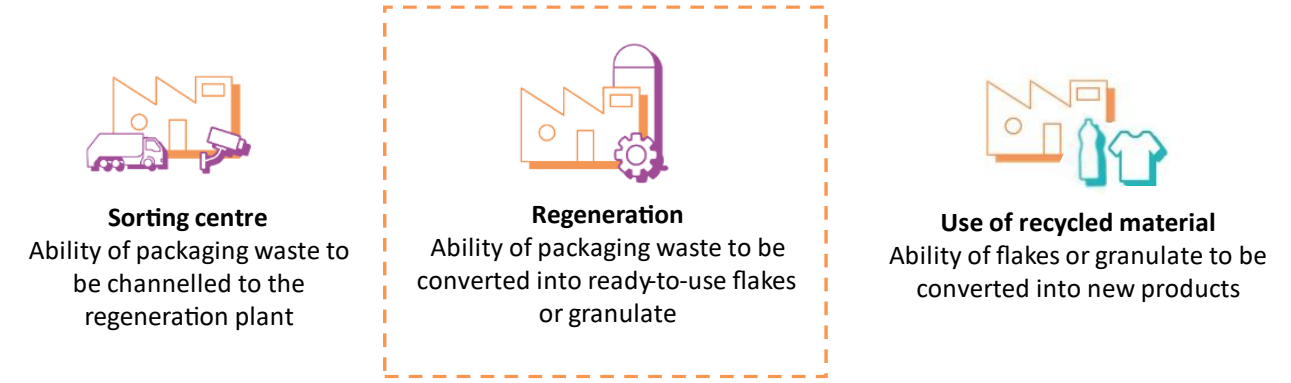

Figure 1: Scope of the PET-P0 bottle protocol

This protocol takes account of current technical knowledge and processes applied by mechanical recycling plants processing French PET bottle and dispenser bottle household packaging streams.

Results obtained from tests performed based on this protocol are insufficient for determining packaging recyclability. This protocol only reflects the process of regenerating packaging as granulate and provides no basis for judging the suitability of packaging for sorting or the feasibility of transforming such regenerated granulate into new products.

2. AIMS

The purpose of this protocol is to assess the impact of new packaging items, barriers, additives or components on the mechanical regeneration process for the (clear, coloured, opaque) PET bottle stream. It allows packaging manufacturers and marketers to test regeneration processes for packaging such as PET bottles or dispenser bottles in pilot conditions. It includes:

- An impact assessment concerning regeneration processes for producing rPET[1] granulate,
- An analysis of the quality of the rPET produced.

The protocol uses information available to COTREP to determine concentrations of packaging or packaging elements to be tested. These concentrations are calculated based on their current or future market penetration using concentration factors representative of plastic bales generated by French selective collection.

The main regeneration processes are shown in the illustration below:

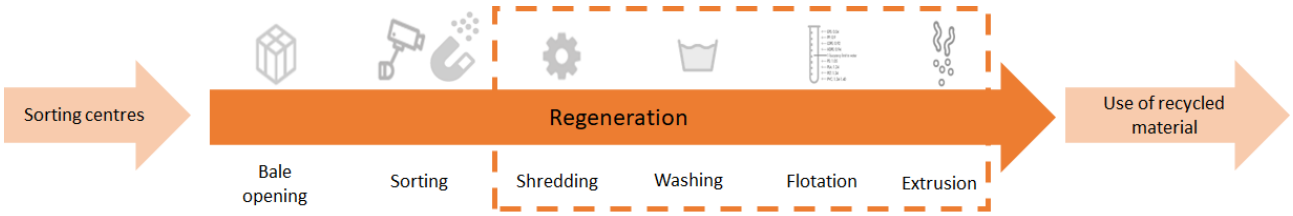

Figure 2: Analytical scope of the regeneration protocol

[1] In this protocol "PET" and "PET bottle/dispenser bottle" are used interchangeably; rPET means recycled PET, i.e. recycled material from the regeneration of household PET bottle and dispenser bottle packaging.

3. TERMS OF REFERENCE

Any company (packaging manufacturer, marketer, resin manufacturer, distributor, etc.) seeking to determine the impact of a specific packaging item on regeneration in the French PET bottle stream can use this protocol to perform testing.

Companies wishing to perform regeneration tests shall be referred to hereafter as "**Requesters**". COTREP-certified test laboratories able to comply with this test protocol shall be referred to hereafter as "**Laboratories**". A list of certified laboratories is provided in the "Practical information" section.

4. PREPARATION FOR TESTS

Step 1: Contacting the Laboratory

The **Requester** should contact the **Laboratory** and describe its request using the document in APPENDIX 1. If the **Requester** wishes to test the regeneration of several rigid packaging types, several copies of APPENDIX 1 should be supplied. Contact details are provided in the "Practical information" section of this document.

Step 2: Preparing test samples

The **Requester** should submit test samples to the **Laboratory**. Only packaging structures listed by COTREP in APPENDIX 2 may be tested to ensure protocol representativeness.

- All types of bottles and dispenser bottles (shape, capacity, applications, etc.) can be tested
- Whole packaging items should be tested (packaging body and associated elements*)
- Depending on their applications, packaging items may be new or emptied of their contents as discarded by the consumer.

Total quantities of packaging to be provided will depend on the capacity of equipment used by the **Laboratory**. A minimum of roughly 10kg of empty packaging is required to ensure significant results, see APPENDIX 2 for more details about quantities. The concentration levels and quantities to provide are determined based on volumes of test packaging marketed and are specified by COTREP in APPENDIX 2. Material quantities should be adjusted to create a minimum of 2 market penetration rates.

* Examples: label, cap, seal lid, handle, etc.

Step 3: Preparing a control sample

The standard material for the protocol is a batch of rigid rPET flakes produced from shredded-washed French selective collection streams. COTREP will provide the **Laboratory** with the standard material. The standard stream will be validated by COTREP according to the colour of the packaging tested and channelling at sorting centres.

The **Laboratory** should visually certify the quality of the control sample received. It should take photographs and ensure the **Requester** has access to these items. All items received should be included in the report.

5. METHODOLOGY

The protocol set out below is intended for COTREP-certified **Laboratories** with equipment representative of regeneration processes applied in existing industrial units.

The steps to be included are described in the figure below. Optical sorting performed on flakes at regeneration plants is not included in this protocol.

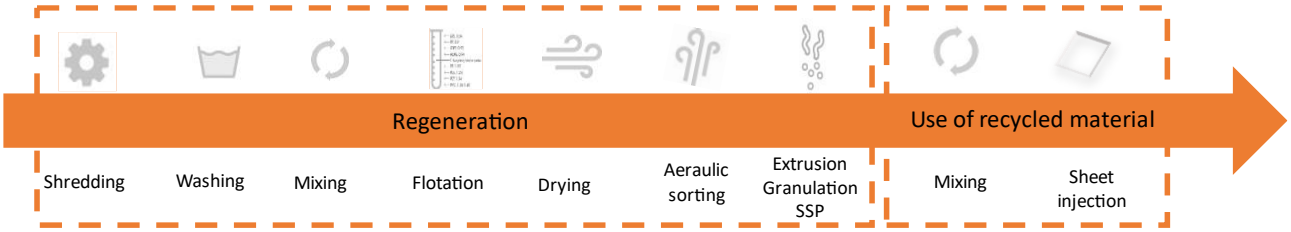

Figure 3: Detailed description of regeneration protocol steps

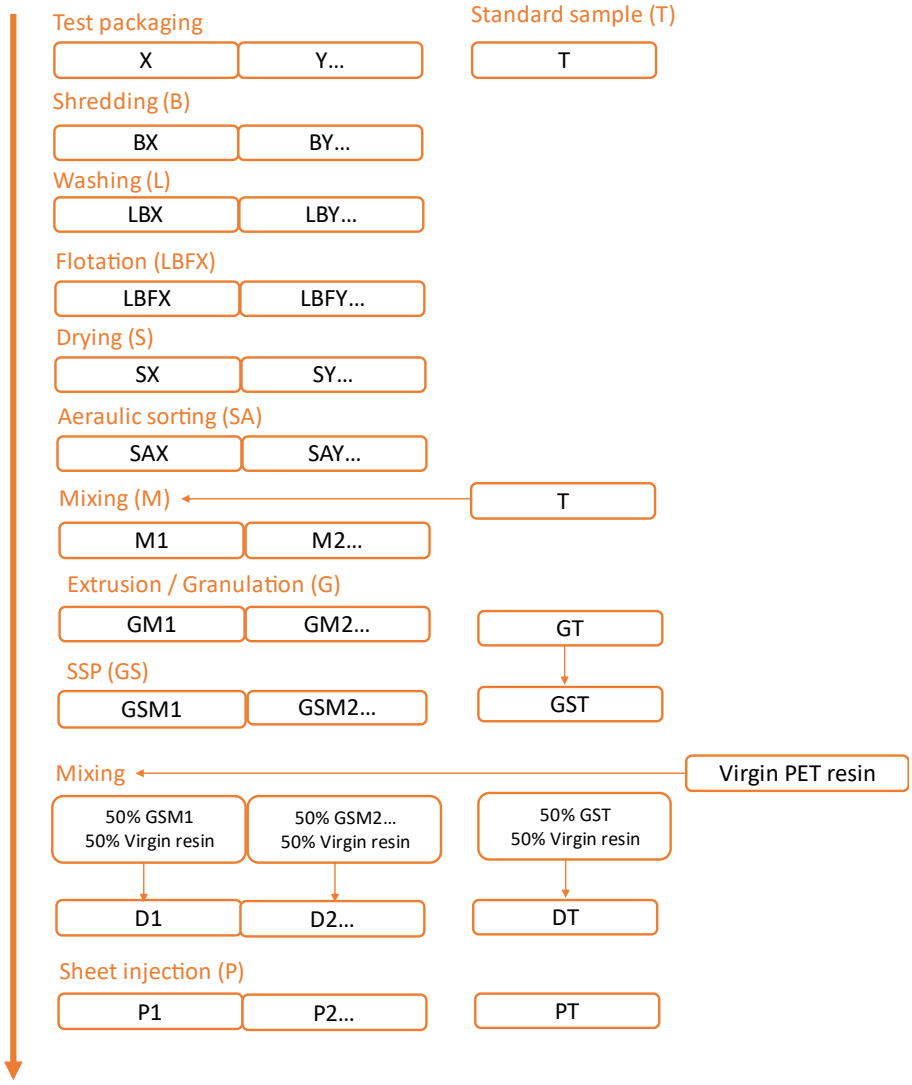

Figure 4: Description of regeneration protocol steps and associated products

The **Laboratory** takes material from the samples and test blends during the various stages of the protocol. These will be kept at least until submission of the test report.

Step 1: Shredding of X samples (BX)

The **Laboratory** shreds the test and standard samples to produce 12mm flakes if needed. The fines (≤ 1mm) are weighed and the result is recorded in a report. The flow rate is also recorded.

The **Laboratory** should indicate any anomalies or difficulties in shredding the test samples in its report. In particular, it should state whether any fines are present and describe the appearance of the shredded material produced (photographs should be included in the report).

Keep a 50g flake sample to compare the visual appearance (colour) before and after washing.

Shredding: success criteria

- No faults or damage to the shredder during testing due to the nature of the sample
- No large clusters in the shredder
- No abnormal quantities of fines

Step 2: Washing BX flakes (LBX)

The BX flakes produced should then be washed under the conditions described below. Washing should be performed in batches weighing at least 5kg which will vary depending on the laboratory, with the number of batches dependent on the quantity to be prepared.

Heat the wash tank to at least 85°C with a 1% mass of sodium and 1% mass of detergent. Place the BX test sample in a tank containing 4L of clear pre-heated water for every minimum 1kg sample. The precise temperature of the water should be recorded in the report. The tank should be sufficiently large to enable rapid agitation. Wash while agitating rapidly (e.g. max. 1,000 rpm for 5 minutes) and record the washing conditions in the test report.

Recover a sample representative of the wash water after filtration with a grille/sieve with a ~1mm mesh for visual inspection. Note down any changes in the colour and transparency of the wash water supported by photographs. Make a note of any suspended particles and their type. The quantity of these suspended particles (paper/fibre, fines, adhesive clusters, etc.) may be determined by standard NF EN 872 if stipulated by the **Requester**. Any observations made subsequent to examination should be recorded in the final report supported by photographs.

Optional stage 1: Visual inspection of LBX flakes

This inspection should be performed if the tested packaging features an affixed label or any other associated element (decoration, banding, etc.), please refer to APPENDIX 2.

Examine 3 x 10g flake samples and make a note of any adhesive, paper, ink or other unwanted substances present on the flakes. Any observations made subsequent to the various operations should be recorded in the final report. Include photographs in the report.

Washed LBX flakes should then be centrifuged and dried.

Washing: success criteria

- No soiling or jamming of equipment
- No residues on the sides of the equipment or on the flakes (adhesive, ink, etc.)
- No change in wash water appearance (no colouring or foam formed, etc.)
- If optional stage 1 is performed: no contaminants on the flakes for the 3 x 10g samples, non-plastic materials (fibre, paper) < 0.01g.

A rinsing step should be performed by the laboratory. This step can be combined with the washing or flotation (sink-float) step. The rinsing parameters (temperature, speed, time, etc.) should be included in the laboratory report.

Step 3: Flotation of LBX flakes (LBFX)

At this stage, the behaviour of the different flakes is tested during flotation.

Optional stage 2: Quick test on LBX flake flotation

This test should be performed if the tested packaging might affect the flotation stage (e.g. density close to 1, surface effects, associated elements with a density <1, etc.), please refer to Appendix 2.

- *Add 150g LBX flakes to a beaker containing 1L of clear water at room temperature*
- *Mix with a magnetic stirrer for 1 minute*
- *Stop the magnetic stirrer then leave to rest for 1 minute*
- *Take a photo of the beaker to examine the sink and float fractions and water quality (cloudy, stained, etc.)*
- *Recover, dry and weigh each fraction to measure the proportion of the sink fraction*

Add the LBX flakes to a tank containing additive-free clear water at room temperature. The tank should be sufficiently large to enable slow agitation and full immersion of the test material.

Collect any floating flakes (LBF). Collect any sunk flakes (LBC). Weigh the float and sink fractions when wet and determine moisture content. Moisture content should be included in the report.

Recover a sample representative of the flotation water after filtration with a grille/sieve with a ~1mm mesh for visual inspection. Note down any changes in the colour and transparency of the flotation water supported by photographs. Specific analyses, for example the nature and quantity of suspended particles (paper/fibre, fines, adhesive clusters, etc.), should be performed in the cases specified in APPENDIX 2. Any observations made subsequent to examination should be recorded in the final report supported by photographs.

Optional stage 3: Visual inspection of LBF flakes

This inspection should be performed if the tested packaging features an affixed label or any other associated element (decoration, banding, etc.), please refer to APPENDIX 2.

Examine the 2 flake fractions (float and sink) and record any adhesive, paper, ink, etc. present on the flakes supported by photographs. The equipment used and the operating conditions implemented should also be recorded in the final report.

Please note: Any observations made subsequent to examinations and included in the final report may be used to identify impacts on regeneration, particularly in terms of treating waste water from washing/rinsing.

Flotation: success criteria

- The innovative packaging is recovered in the sink fraction (no suspended fraction) (except in the specific case of a component or element with a density < 1 which should be recovered in the float fraction).
- No changes in the flotation water.
- *If optional stage 3 is performed: No adhesive, paper or ink on the flakes and sink fraction representing a mass of at least 90%.*

Step 4: Drying LBFX flakes (SX)

Dry the LBFX flakes using a dryer. The drying method and conditions should be adjusted to avoid fusing/deteriorating PET flakes. After drying, measure the moisture content of at least 3 x 10g flake samples. The flakes' moisture content should be no higher than 0.5%.

The conditions applied (temperature, residence time, etc.) and drying type should be specified in the test report.

Examine the flakes and make a note of any significant changes in comparison to the LBX flakes before flotation (changes in the shape/appearance or colour of flakes).

Any observations made subsequent to examination should be recorded in the final report (include photographs in the report). The equipment used and the operating conditions implemented should also be recorded in the final report.

Drying: success criteria

- No changes in the shape or appearance of flakes after drying
- No fines were produced
- Moisture content < 0.5%

Step 5: Aeraulic sorting of SX flakes (SAX)

Perform aeraulic sorting on the SX flakes to remove any remaining fines and light items. The method and conditions should be recorded in the test report. The fraction removed by aeraulic sorting is weighed and the result is recorded in the report.

Aeraulic sorting: no success criterion

Step 6: Analysing SAX flakes

Flake analyses are performed on innovative SAX material and the standard material. The following flake analyses should be performed:

- Oven test on the innovative and standard material at 220°C for 1 hour. Record any yellowing, clusters or blackened particles (indicator of potential PVC content).
- Apparent density according to standard ASTM D1895, Method C. Density must be >0.28 g/cm3.
- Comparison of visual appearance (colour) with the flake sample kept after step 1: shredding.

Flake analysis: success criteria

- Apparent density: higher than 0.28 g/cm3
- No major change in visual appearance (colour) of the flakes between the samples after step 1 and step 5

Step 7: Mixing SAX flakes (M)

Depending on the laboratory, this step can also be performed before step 2: Washing BX flakes.

Mix T flakes produced from the standard material with SAX flakes produced from the test packaging based on market penetration levels defined by COTREP until a consistent mixture is obtained.

Total quantities applied will depend on the capacity of equipment used by the **Laboratory**.

Penetration rates are defined by COTREP and shown in APPENDIX 2 in the following format:

M1 = x% SAXX + y% T
M2 = w% SAX + z% T

Where: x + y = w + z = 100; x and w being the market penetration rates shown in APPENDIX 2.

As many batches as required should be mixed to produce the necessary quantities for implementing the next stages of the test.

Penetration rates have only been identified for scenarios covered by a COTREP General Notice. If your packaging is not shown in APPENDIX 2, you may contact COTREP to notify your wish to have a test. COTREP will then inform you whether it is possible to apply this protocol to your packaging. COTREP regularly updates this list.

Step 8: Extrusion/Granulation (G)

The mixtures and T control are dried until a residual moisture content <100 ppm is obtained.

The mixtures and **T** control are then extruded and granulated to obtain an amorphous PET material. If necessary depending on the equipment, flake re-shredding is allowed and must be recorded in the report.

A filter change should be performed after each test batch.

The equipment used and the granulation conditions implemented should be recorded in the final report.

- Typical extruder: (screw diameter, L/D ratio);
- Filter size: ≤105 µm;
- Granulation type;
- Temperatures: between 280°C and 290°C in the filter;
- Duration: at least 30 minutes;
- Flow rate: 5 to 10 kg/h recommended;
- Quantities;
- Pressures/amperage;
- Vacuum, etc.

The nature and type of filter used should be recorded.

The parameters of the extrusion/granulation process used on each batch should be the same as those used on the standard batch first implemented for the run. Any variations should be recorded in the report.

Extrusion/Granulation: success criteria

- No faults or damage to the extruder during testing due to the nature of the sample (accumulation, clogging, etc.)
- Extrusion process stable during sample transformation (no unusual pressure rise)
- No problems in terms of degassing
- No filter change during granulation

Step 9: SSP (Solid State Polymerisation) (GS)

The amorphous granulate then undergoes a crystallisation and SSP process via a reactor.

The vacuum crystallisation process is necessary to prevent the granulate adhering during subsequent transformation steps. SSP is performed in nitrogen and/or vacuum conditions at a temperature of approximately 200°C until an IV of 0.80 ± 0.02 dL/g is achieved.

The equipment used and the SSP conditions implemented should be recorded in the final report.

- Temperatures;
- Time;
- Vacuum level.

SSP: no success criterion

Step 10: Characterisation of granulate

Granulate should undergo a visual inspection (porosity, gels, colour, etc.) with supporting photographs included in the report. Moreover, all prepared granulate should be characterised:

- Intrinsic viscosity (±0.02 dL/g variation on the standard)
- Colour (L*, a*, b*)
- DSC (crystallinity, melting point: maximum 10% variation on the standard)
- Density of granulate
- Acetaldehyde (AA) concentration: maximum 35% increase on the standard.

Results should be included in the report.

Characterisation of granulate: success criteria

- Intrinsic viscosity: more or less 0.02 dL/g variation on the standard
- DSC: melting point more or less 10% variation on the standard
- Acetaldehyde (AA) concentration at a maximum 35% increase on the standard.

Step 11: Dilution (D)

Granulate GST, GS1, GS2, etc. prepared in advance and characterised should be mixed with a virgin PET resin at a rate of 50% by weight to produce mixtures DT, D1, D2, etc. The mixtures should ideally be produced mechanically.

The virgin PET granulate used for these tests should be PET granulate with an IV of between 0.76 and 0.84. Products identified as appropriate can be any of the following (or equivalent):

- Ramapet N180 (Indorama)
- Ramapet R1 (Indorama)
- PPK FR L (Plastipak)
- PPK FR (Plastipak)

The **Laboratory** should order the virgin materials required for testing and specify the product used in its final report.

A 100g sample of the virgin PET granulate used should be kept by the **Laboratory**.

Step 12: Sheet injection and analysis (P)

Check the moisture content of the diluted crystallised granulate (<100ppm) and dry as required.

Mixtures DT, D1, D2, etc. are injected in the form of 3mm thick sheets.

The following analyses are then performed on the sheets:

- Haze
- Colour (L*, a*, b*).

Sheet analysis: success criteria

- Haze:

- Criterion for clear PET: under development, the criteria will be set out in a subsequent version of the protocol.

- Criterion for coloured and opaque PET: under development, the criteria will be set out in a subsequent version of the protocol.

- Colour (L*, a*, b*):

- Criterion for clear PET: $\Delta b^* = 1.5$ maximum in relation to the standard.

- Criterion for coloured and opaque PET: under development, the criteria will be set out in a subsequent version of the protocol.

6. TEST REPORT

The commissioned **Laboratory** should draw up a test report including the following details:

- A description of samples received including photographs
- APPENDIX 1 completed and appended to the report
- The operating conditions and equipment used for each test
- Results for each step and observations versus the control sample including the required photographs for each step and achievement of success criteria
- Any observations to be made during the tests should be included in the report and are provided in APPENDIX 3
- Sampling performed by the **Laboratory** at the different stages will be available to the **Requester** upon request. All materials relating to the run should be kept by the **Laboratory** for 6 months following publication of the corresponding COTREP Notice unless otherwise instructed by **COTREP**.

Important:

The methodology used for testing all samples submitted for analysis should be strictly identical. The **Laboratory** undertakes to follow the entire protocol and record any deviations in the test report (along with justification of any such deviations).

The report should include the following declaration:

"*Tests were performed according to the COTREP regeneration test protocol for PET bottle and dispenser bottle packaging (Reference/Version/Date). These results do not constitute a full packaging recyclability assessment and are not valid as a recyclability certificate."*

Any deviations should be clarified and will be examined by COTREP to determine whether the results are valid.

7. CONFIDENTIALITY

By signing a confidentiality agreement to be observed with respect to all third parties except COTREP, the **Laboratory** undertakes to maintain the confidentiality of any information concerning the request, the content of the report, and in particular, any results and observations.

8. PRACTICAL INFORMATION

COTREP contacts

Alexana Bellegarde (COTREP)

Tel.: +33 6 81 06 83 24

Email: a.bellegarde@cotrep.org

Laboratory contacts

PTI Europe

Jérôme Larrieu

Tel.: +41 79 351 32 44

Email: j.larrieu@pti-europe.com

Cost of tests

The **Requester** should contact the **Laboratory** for a quotation. It should also budget for the cost of shipping samples to the **Laboratory**.

APPENDIX 1: COTREP test request form

REQUESTER

COMPANY:

FIRST NAME/LAST NAME:

POSITION:

EMAIL:

TELEPHONE:

IMAGE
OF
THE PACKAGING

DESCRIPTION OF THE TEST PACKAGING

PACKAGING TYPE:
E.G. BOTTLE, DISPENSER BOTTLE, POT, TRAY, TUBE, ETC.

MAJORITY RESIN:

PACKAGING STRUCTURE:
IF MULTILAYER, DESCRIBE THE LAYERS. SPECIFY THE % BY MASS OF EACH COMPONENT (BARRIER, ADDITIVES, ADHESIVE, TIE LAYER, ETC.)

FORMING METHOD:

COLOUR/PRINTING:
SPECIFY IF ON SURFACE OR BLENDED

ASSOCIATED ELEMENTS:
LABELS, TAP, ZIP, TIE, ETC. SPECIFY THE COMPOSITION OF EACH ASSOCIATED ELEMENT

VOLUME MARKETED:
TONNES PER YEAR IF NOT YET MARKETED, PROVIDE PROJECTIONS

COMMENTS:
ANY OTHER POTENTIALLY USEFUL INFORMATION FOR THE TEST

Company stamp:	**Date:**	**Last name, first name and signature**

APPENDIX 2: Market penetration rate to be applied

Market penetration rates are estimated by COTREP members based on their expertise and knowledge of the French household packaging market. Market penetration rates change according to packaging type and composition. When conducting tests in accordance with the PET bottle and dispenser bottle regeneration protocol, the penetration rates set out below should be applied to ensure representativeness of quantities marketed in France.

Step 1: Which packaging categories to test

When conducting testing, it is necessary to identify the penetration rates to be applied based on known values. Penetration rates have only been defined for packaging scenarios covered by a COTREP General Notice. The table below lists scenarios and penetration rates to be applied based on the packaging type tested. This appendix is updated regularly to take account of COTREP studies and publications.

Step 2: Identifying applicable penetration rates

If several categories can be identified for your packaging, the highest penetration rates should be applied. Two penetration rates should always be tested to validate the COTREP protocol. Please note that penetration rates should be applied consistently between studies.

Market penetration rates applicable for testing PET bottle and dispenser bottle regeneration

STRUCTURE OF THE TEST PACKAGING	DESCRIPTION	PENETRATION RATE TO BE APPLIED (x and w)	GENERAL NOTICE REFERENCE
SLEEVES ON PET BOTTLES IF CHANNELLED TO CLEAR PET	PET bottles with sleeves, channelled to clear PET	4% and 6%	AG67
SLEEVES ON PET BOTTLES IF CHANNELLED TO COLOURED PET	PET bottles with sleeves, channelled to coloured PET	14% and 21%	AG67
PET AEROSOL DISPENSERS	PET aerosols	1.3%	AG64
OXYGEN SCAVENGERS	Clear PET bottles with oxygen scavengers	8%	*Source: Cotrep (2024)*
PA BARRIER IN CLEAR PET	Clear PET bottles with PA barrier	2.5%	*Source: Cotrep (2024)*
PA BARRIER IN COLOURED PET	Coloured PET bottles with PA barrier	6%	*Source: Cotrep (2024)*

The COTREP roadmap of future studies is provided on the website at www.cotrep.fr.

Penetration rates have only been identified for scenarios covered by a COTREP General Notice. If your packaging is not shown in the list above, you may contact COTREP to notify your wish to have a test. COTREP will then inform you whether it is possible to apply this protocol to your packaging. This list is updated in light of published general notices and is regularly updated by COTREP.

APPENDIX 3: Observations to include in the report

The PET-P0 protocol provides the assessment criteria for the different stages in the protocol.

The observations to include in the report at the different stages are provided below.

Shredding:

- Shredder operation during testing
- Agglomeration in the shredder
- Presence of fines

Washing:

- Soiling or jamming of equipment
- Residues on the sides or on the flakes (adhesive, ink, etc.)
- Wash water appearance
- If optional stage 1 done: visual presence of contaminants on the flakes

Flotation:

- Quantity of sink fraction
- Change in flotation water
- If optional stage 3 done: quantity of sink fraction, change in flotation water

Drying:

- Changes in the shape or appearance of flakes after drying
- Fines produced
- Moisture content

Flake analysis:

- Oven test: presence of yellowing, clusters or blackened particles
- Apparent density
- Visual appearance (colour) of flakes compared with after shredding

Extrusion/Granulation:

- Extruder operation during testing
- Extrusion process stability during sample transformation
- Operation in terms of degassing
- Filter change during granulation

Characterisation of granulate:

- Intrinsic viscosity
- Colour (L*, a*, b*)
- DSC
- Density of granulate
- Acetaldehyde (AA) concentration

Sheet analysis:

- Haze
- Colour (L*, a*, b*)