



TEST PROTOCOL FLEXIBLE PE-1

Regeneration of flexible PE household 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 (Citeo, Elipso, SRP and Valorplast) 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 household plastic packaging end-of-life.

VERSION NO.	DATE	DESCRIPTION
1	September 2021	Initial version
2	December 2021	Penetration rate for PA
3	March 2022	Addition of a penetration rate for PA

1. CONTEXT

COTREP has drawn up this protocol in collaboration with French manufacturers involved in regenerating flexible PE household packaging. It is representative of the most commonly used processes in Europe. Its purpose is to specify tests to be performed to assess the suitability of flexible packaging for mechanical regeneration in the industrial stream for flexible PE packaging. 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 at least implementing protocol PE-2: Blow extrusion.

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

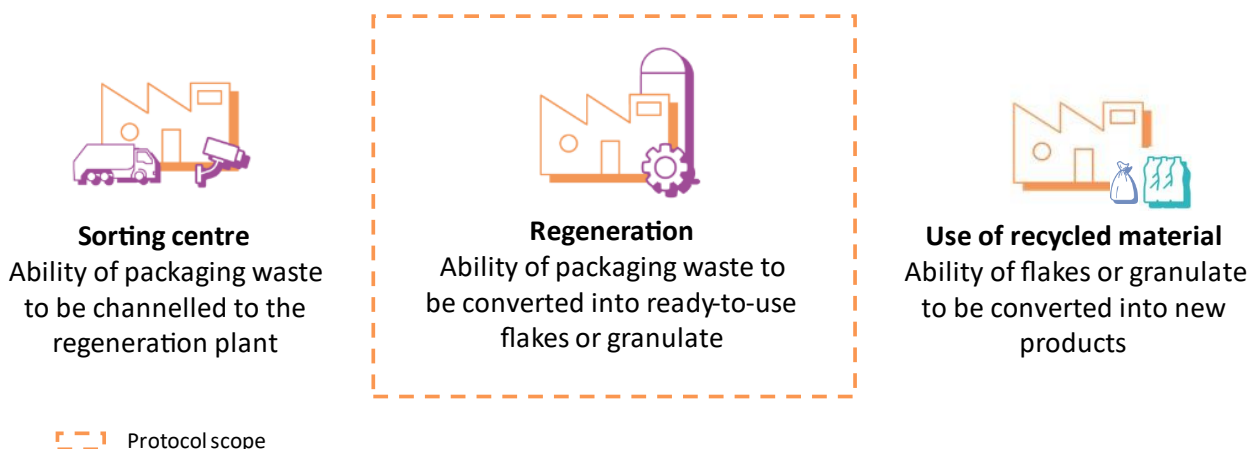


Figure 1: Scope of the COTREP protocol

This protocol takes account of current technical knowledge and processes for regenerating flexible PE household packaging in Europe.

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.

Please note: This protocol is not appropriate for assessing the suitability of packaging for regeneration in a "mixed plastics", "mixed films" or "mixed PO" stream since such a stream does not exist for French packaging waste.

2. AIMS

The purpose of this protocol is to assess the impact of new packaging items or components on the regeneration process for the flexible PE stream. It allows packaging manufacturers and marketers to test packaging regeneration processes in pilot conditions. It includes:

- An impact assessment concerning regeneration processes for producing rPE granulate,
- An analysis of the quality of rPE produced.

The protocol uses information held by COTREP to determine concentrations of packaging or packaging elements to be tested. These levels 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:

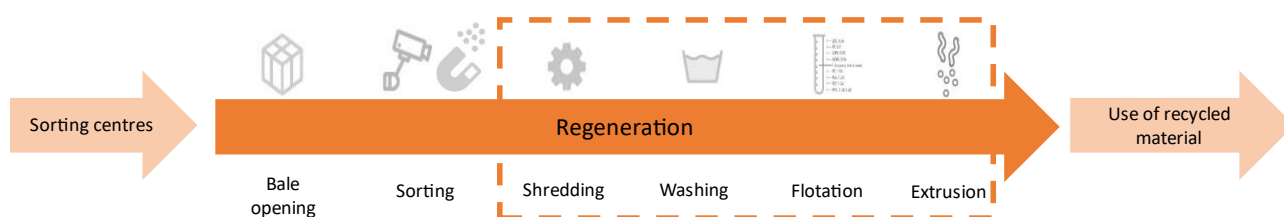


Figure 2: Analytical scope of the regeneration protocol

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 flexible PE 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

Étape 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 flexible packaging types, several copies of [APPENDIX 1](#) should be supplied. Contact details are provided in the "Practical information" section of this document.

Étape 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 flexible packaging can be tested
- Whole packaging items should be tested. Depending on their applications, packaging items may be new or emptied of their contents to reflect consumer habits.

Total quantities of packaging to be provided will depend on the capacity of equipment used by the **Laboratory**. A minimum of 15kg of packaging is required to ensure significant results. The concentration levels tested 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.

Étape 3 : Preparing a control film

The standard material for the protocol is a 50µm film composed of 100% flexible rPE produced by regeneration of French collective selection streams. The **Laboratory** should be in possession of control rPE granulate supplied by COTREP. COTREP will provide the **Laboratory** with the standard material.

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 following steps should be performed:

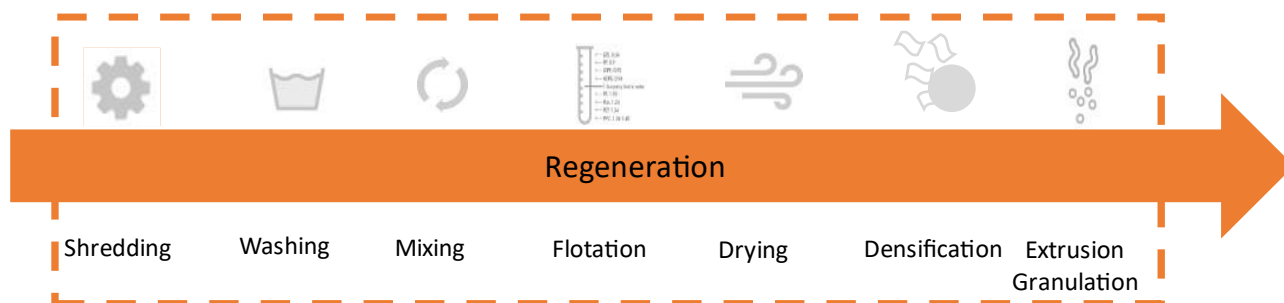


Figure 3: Detailed description of regeneration protocol steps

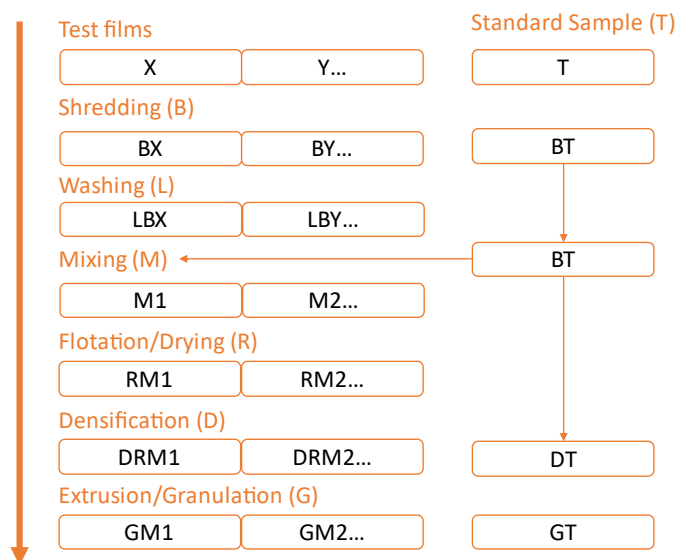


Figure 4: Description of regeneration protocol steps and associated products

Étape 1 : Shredding of X samples (BX)

The **Laboratory** shreds the test and standard samples to produce 20mm flakes. The flakes then undergo a dedusting step to remove any residual elements under 3mm, which are referred to as "fines". The fines 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 clusters are present and describe the appearance of the shredded material produced (photographs should be included in the report).

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

Étape 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 1kg, with the number of batches dependent on the quantity to be prepared.

Place the BX test sample in a tank containing 20L of additive-free clear water at room temperature (the precise temperature should be recorded in the report) per kilogram of test material. The tank should be sufficiently large to enable rapid agitation. Wash while agitating rapidly (max. 1000 rpm) for 5 minutes and record the washing conditions in the test report. Filter with a grille/sieve with a 1mm mesh and recover a representative sample of wash water for visual inspection. Note down any changes in the colour and transparency of the wash water. The nature and quantity of suspended particles (paper/fibre, fines, adhesive clusters, etc.) may be determined if stipulated by the **Requester**.

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 before mixing. Moisture content should be regularly monitored during the drying phase and should not exceed 5%.

Washing: success criteria

- No soiling or jamming of equipment

- No residues on the sides or on the flakes (adhesive, ink, etc.)
- No contaminants on the flakes: for the 3 x 10g samples → less than 0.01g of non-plastic materials (fibre, paper)

Étape 3 : Mixing LBX flakes (M)

Mix clean BT flakes produced from the standard material with shredded BX 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**, with a minimum of 15kg per tested mixture.

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

$$M1 = x\% \text{ BX} + y\% \text{ BT}$$

$$M2 = w\% \text{ BX} + z\% \text{ BT}$$

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**, it is not possible to implement this test protocol. COTREP regularly updates this list.

Étape 4 : Flotation of M mixtures (R)

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

Add the mixed M1 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 (RM1). Collect any sunk flakes (LBC). Weigh the float and sink fractions when wet (FHF and FHC) and determine moisture content.

Retrieve and filter the wash water for examination. Take note of the filtration mesh. Note down any changes in the colour and transparency of the wash water supported by photographs. Determine the nature (paper/fibre, fines, adhesive clusters, etc.) and quantity of suspended particles: measure materials suspended in the water before and after washing (in accordance with NF EN 872). Any observations made subsequent to examination should be recorded in the final report supported by photographs.

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.

Repeat these processes with the other mixtures M2, M3, MX, etc.

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 float fraction should be > 90% of the total weight of the mixture
- No adhesive, paper or ink on the flakes
- No changes in the flotation water

Étape 5 : Drying RM flakes

Dry the RM1 and RM2 flakes using a dryer at a temperature of 130°C. Drying conditions should be adjusted to avoid fusing PE flakes during the drying stage. After drying, measure the moisture content of at least 3 x 1g flake samples. The flakes' moisture content should be no higher than 5%.

Target moisture content may be lower depending on the type of densifier used in step 6.

The conditions applied and the number of drying cycles performed should be specified in the test report.

Examine the flakes and make a note of any significant changes in comparison to the M1 and M2 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

Étape 6 : Densification (D) (optional)

A densification step may be considered for the film flakes produced to enable an even and effective extruder feed. The densification temperature should not exceed 200°C. The temperature used should be recorded in the report.

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.

Please note: At industrial scale, the densification step is generally not performed separately from the extrusion/granulation step.

Étape 7 : Extrusion/Granulation

The mixtures and **LBT** control are extruded and granulated. At least one zone should be 250°C during the extrusion stage.

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

- Typical extruder: (screw diameter, L/D ratio);
- Filter size;
- Granulation type;
- Temperatures of the different zones;
- Duration;
- Flow rate;
- Quantities;
- Pressures/amperage;
- Vacuum, etc.

The nature and type of any filter used should be recorded and representative of standard production, i.e. 150µm.

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 problems in terms of degassing
- No more than 1 filter change during granulation

Étape 8 : 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 based on the tests described below.

PROPERTY EXAMINED	STANDARDS	ANTICIPATED RESULTS
DENSITY	NF EN ISO 1183-1	kg/m ³ value
DSC TESTING	NF EN ISO 11357-3 with a temperature rate of 10°C/min	Values and curves
MELT INDEX	NF EN ISO 1133-1, (2.16 kg, 190°C)	g/10min value + observations of extrudate
ASH CONTENT	NF EN ISO 3451-1 (650°C)	% value
MOISTURE	Internal at 105°C	% value

Results should be included in the report.

The resulting granulate **GM1**, **GM2** and **GT** should be assessed in accordance with protocol Flexible PE-2 Blow extrusion at an appropriately equipped test centre.

Extrusion/Granulation: success criteria

- Under 10% variation between GM samples and the GT standard sample

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.

Important:

The methodology used for testing all samples submitted for analysis should be strictly identical. The **Laboratory** undertakes to follow the entire protocol, record any deviations in the test report, and send the test report to COTREP.

The report should include the following declaration:

"Tests were performed according to the COTREP regeneration test protocol for flexible PE 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 third parties, 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

Benoit Le Dreff (Valorplast)

Tel.: +33 (0)6 31 37 10 77

Email: b.ledreff@valorplast.com

Laboratory contacts

IPC

Jaime Rodrigues

Tel.: +33 (0)6 48 58 74 05

Email: Jaime.RODRIGUES@ct-ipc.com

Jerome Piejak

Tel.: +33 (0)4 26 61 90 48

Email: Jerome.PIEJAK@ct-ipc.com

Cost of tests

For information: the approximate cost of performing tests in accordance with protocol Flexible PE-1 is €10,000 excl. VAT for the standard and two concentrations of a product.

The **Requester** should also budget for the cost of shipping samples to the **Laboratory**.

ANNEXE 1 : COTREP test request form

REQUESTER:

COMPANY: *To be completed*

FIRST NAME/LAST NAME: *To be completed*

POSITION: *To be completed*

EMAIL: *To be completed*

TELEPHONE: *To be completed*

IMAGE
OF
THE PACKAGING

DESCRIPTION OF THE TEST PACKAGING

PACKAGING TYPE: *E.g. bottle, dispenser bottle, pot, tray, tube, etc.*

MAJORITY RESIN: *To be completed*

PACKAGING STRUCTURE: *If multilayer, describe the layers.
Specify the % by mass of each component (barrier, additives, adhesive, tie layer, etc.)*

FORMING PROCESS:

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
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ANNEXE 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 flexible PE packaging regeneration protocol, the penetration rates set out below should be applied to ensure representativeness of quantities marketed in France.

Étape 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.

Étape 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 flexible PE regeneration

CATEGORY	DESCRIPTION	PENETRATION RATE TO BE APPLIED	GENERAL NOTICE REFERENCE
EVOH	Flexible PE packaging with EVOH barrier	1% and 2%	AG68
PP	PP (flexible or rigid) that is not separable from a flexible PE packaging item	1% and 2%	AG69
PA	Flexible PE packaging with polyamide barrier	1% and 4%	AG70

Examples:

→ Mono-PE stand-up pouch and EVOH barrier:

The penetration rates for the EVOH barrier study should be applied for testing. Rates of 1% and 2% of EVOH in the mix should be tested.

→ Mono-PE stand-up pouch with EVOH barrier and a PP tap:

The EVOH and PP categories may be applied to the packaging item. Consequently, the highest market penetration rates should be applied, i.e. those for EVOH: 1% and 2%.

→ Flexible PE packaging with other components:

There is currently no COTREP general notice for metallised elements. Consequently, market penetration rates are unknown. It is currently not possible to perform flexible PE packaging regeneration testing.

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, it is not possible to implement this test protocol. This list is updated in light of published general notices and is regularly updated by COTREP.