TEST PROTOCOL FOR FLEXIBLE PE-1

Mechanical 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 (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 household plastic packaging end-of-life.

VERSION NO.	DATE	DESCRIPTION
1	September 2021	*Initial version*
2	December 2021	*Addition of penetration rates for PA*
3	March 2022	*Addition of an additional rate for PA*
4	February 2025	*Addition of penetration rates for PVOH type water-based coating, SiOx, metallisation, oriented PE, EVA and compostable flexibles* *Addition of details on protocol implementation: addition of moisture content measurement*

1. CONTEXT

COTREP has drawn up this protocol in collaboration with French manufacturers involved in regenerating flexible PE household packaging. It is representative of industrial practices applied by regeneration plants processing streams in France. 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 assessing recyclability.

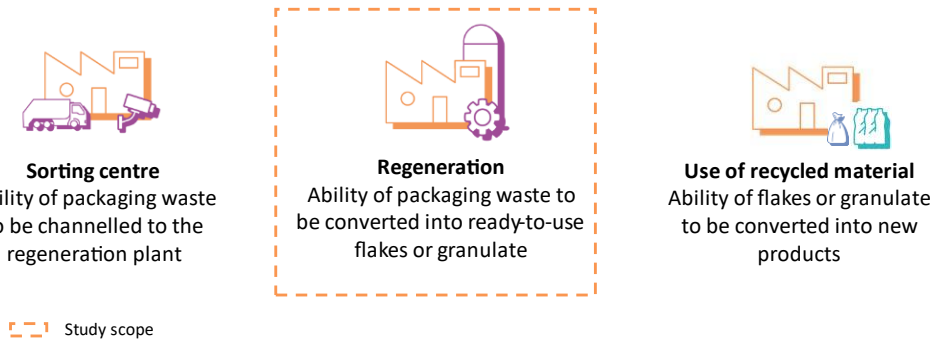

Figure 1: Scope of the Flexible PE-1 protocol

This protocol takes account of current technical knowledge and processes applied by mechanical regeneration plants processing French flexible PE 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.

Comment: This protocol is not appropriate for assessing the suitability of packaging for regeneration in a "mixed plastics", "mixed films" or "mixed PO" stream. A flexible PP and PP/PE stream is under development and not yet fully operational for French household packaging waste.

2. AIMS

The purpose of this protocol is to assess the impact of new packaging items or components on the mechanical 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[1] granulate,
- An analysis of the quality of rPE 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:

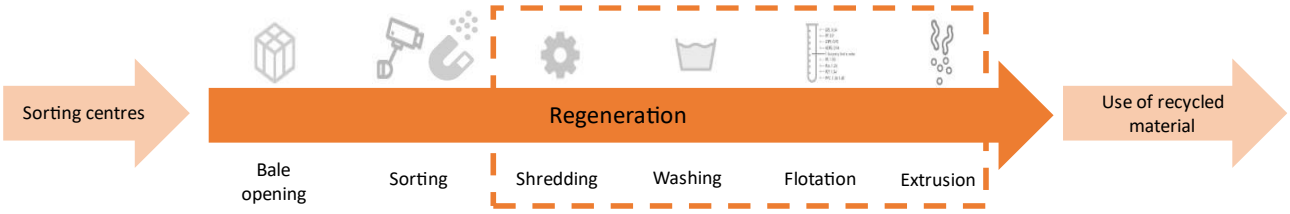

Figure 2: Analytical scope of the regeneration protocol

3. TERMS OF REFERENCE

Any company (packaging manufacturer, marketer, resin manufacturer, distributor, etc.) seeking to determine the impact of a specific packaging item on mechanical 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 "**Requester**". COTREP-certified test laboratories able to comply with this test protocol shall be referred to hereafter as "**Laboratory**". 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 flexible packaging types, several copies of APPENDIX 1 should be supplied. Contact details are provided in the "Practical information" section of this document.

[1] In this protocol "LDPE" and "flexible PE" are used interchangeably; rPE means recycled PE, i.e. recycled material from the regeneration of flexible PE household packaging.

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 flexible packaging 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 15kg of packaging per rate tested 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.

A copy of each sample to be tested should be kept by the **Laboratory**.

Step 3: Preparing a standard sample

The standard sample will be produced by the **Laboratory** using the blow extrusion method from standard material which will be supplied by COTREP in granule form. This material is composed of 100% rPE made from flexible PE packaging produced by regeneration of French selective collection streams.

The moisture content of 3 different granulate samples prior to conversion should be measured by the **Laboratory**. The measurement should be recorded in the report together with the date on which the granulate supplied by COTREP was received and the date of standard film manufacture. 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, in particular details concerning the granulate received (MFI, moisture, density) sent by COTREP.

The **Laboratory** will ensure a uniform batch of product before launching a film production run. The film should be 50µm thick (thickness tolerance: +/- 5% on average and occasionally +/- 20%). The parameters of the blow extrusion process to meet these specifications will be recorded in the report.

A 100g sample of the granulate supplied and a 2 linear meter sample of the standard film produced will be kept by the **Laboratory** for the purposes of inspection (visual or other type) following the run.

Once the standard film has been produced, a batch composed of 100% of such film will undergo the same regeneration protocol stages as the batches containing the test samples, except for the washing and flotation stages. This batch will be used as a control for comparing with batches containing test samples at each step of the protocol.

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:

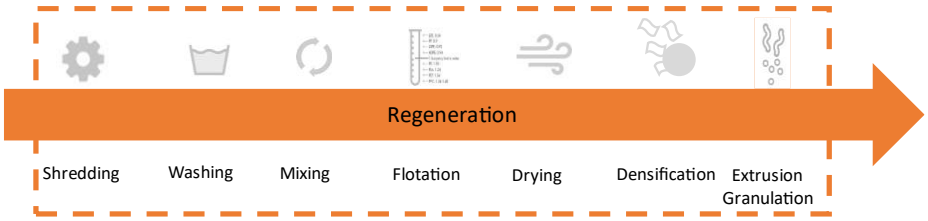

Figure 3: Detailed description of regeneration protocol steps

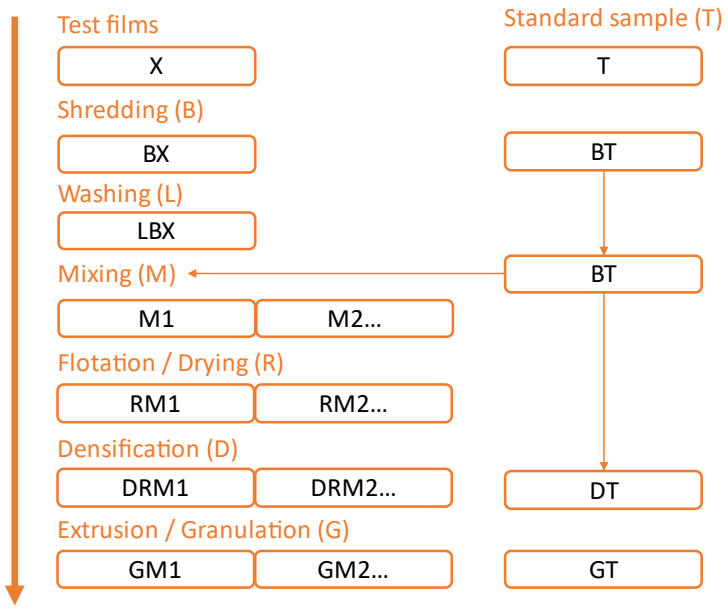

Figure 4: Description of regeneration protocol steps and associated products

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 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 the 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).

Samples of approximately 20g of shredded film and 20g of fines will be kept by the **Laboratory** for each test sample and standard sample.

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 (fines < 15%)

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 1kg, with the number of batches dependent on the quantity to be prepared. Flakes made of the standard material BT are not concerned by this stage.

Place 1kg of BX test sample in a tank containing 20L of additive-free clear water at room temperature (20:1 ratio of water to flakes). 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 at around 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. The nature and quantity of suspended particles (paper/fibre, fines, adhesive clusters, etc.) may be determined, for example,

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.).

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 20%.

A container of wash water should be kept by the **Laboratory** for each test sample.

Washing: success criteria

- No soiling or jamming of equipment
- No residues on the sides or on the flakes (adhesive, ink, etc.)
- No change in wash water appearance (no colouring or foam formed)
- Flake moisture content after the washing stage < 20%
- If optional stage 1 is performed: no contaminants on the flakes for the 3 x 10g samples, non-plastic materials (fibre, paper) < 0.01g

Step 3: Mixing LBX flakes (M)

Mix BT flakes produced from the standard material with washed, shredded LBX 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% LBX + y% BT
M2 = w% LBX + z% BT

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

A sample of approximately 20g of each mixture should be kept by the **Laboratory**.

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 4: Flotation of M mixtures (R)

At this stage, the behaviour of the different flakes is tested during flotation. The batch containing 100% standard material is not concerned by this stage.

Quick test on LBX flake flotation:

- Add 50g LBX flakes to a beaker of at least 5L capacity containing 2L of clear water at room temperature, i.e. a 40:1 ratio of water to flakes
- Mix with a magnetic stirrer for 3 minutes
- Stop the magnetic stirrer then leave to rest for 5 minutes
- Take a photo of the beaker to examine the sink and float fractions and water quality (cloudy, stained, etc.)
- Recover, dry to achieve a moisture content of < 1% and weigh each fraction to measure the proportion of the sink fraction

Add the mixed M flakes to a tank containing additive-free clear water at room temperature. The tank should be sufficiently large to enable slow agitation, full immersion of the test material and a good assessment of the different fractions (float, suspended, sink). For information, a minimum ratio of 27:1 water to flakes (1kg of flakes to 27L water) may be used.

Collect any floating flakes (RM). Collect any sunk flakes. Weigh the float and sink fractions when wet and measure the moisture content of each fraction. 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 2: Visual inspection of RM 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 for 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 the flotation step.

Flotation: success criteria

- The test packaging is recovered in the float fraction (no suspended fraction) (save in the specific case of a component or element with a density > 1 which should be recovered in the sink fraction).
- No changes in the flotation water.
- If optional stage 2 is performed: No adhesive, paper or ink on the flakes and a minimum of 90% of the test packaging is recovered in the float fraction (save in the specific case of a component or element with a density > 1 which should be recovered in the sink fraction).

Step 5: Drying RM flakes

Dry the RM (RM1, RM2, etc.) flakes using a dryer at a temperature of 40°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 0.5%.

The target moisture content may be updated depending on the type of densifier or extrusion/granulation equipment available in the **Laboratory** used in step 6 or 7.

The conditions applied, particularly the drying time needed to reach the target moisture content, and the number of drying cycles performed for each batch should be recorded in the test report.

Examine the flakes and make a note of any significant changes in comparison to the M (M1, M2, etc.) 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.

A sample of approximately 20g of each RM batch should be kept by the **Laboratory**.

Drying: success criteria

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

Step 6: *Optional densification (D)*

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 140°C. The temperature used should be recorded in the report.

Any observations made subsequent to this stage should be recorded in the final report (include photographs in the report). The equipment used for this step and the operating conditions implemented (especially strand or granulate behaviour) should also be recorded in the final report.

A drying step after densification may be added to prepare granulation based on the characteristics of the granulation equipment. The temperature and drying time and method should be recorded in the report. The moisture content of each batch should be measured after this drying stage following densification. The densified material's moisture content after drying should be no higher than 0.2%.

NB: Generally at industrial scale the densification and drying stage at this step 6 is not separate from the Extrusion/Granulation step and so does not exist as such.

A sample of approximately 20g of each batch post densification should be kept by the ***Laboratory****.*

Step 7: Extrusion/Granulation

The mixtures and BT control are extruded and granulated. At least one zone should be 250°C during the extrusion stage and degassing should be performed in a vacuum. Filtration should be representative of standard production, i.e. 150µm. A filter change should be performed after each test batch.

The moisture content of each batch of granulate should be measured after the extrusion/granulation 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
- Nature and type of filter, etc.

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

A sample of 100g of each batch should be kept by the **Laboratory**.

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 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/m³ 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 (2,16kg, 190°C)	*g/10min value + observations of extrudate*
ASH CONTENT	NF EN ISO 3451-1 (650°C)	*% value*
MOISTURE*	Internal at 105°C	*% value*

**3 measurements per property examined will be taken from a sample once a uniform product batch has been achieved.*

The results should be included in the report.

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

Characterisation of granulate: 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 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. For tests performed at COTREP's 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, 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 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 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 contact

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Laboratory contact

IPC
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Cost of tests

For information: the approximate cost of performing tests in accordance with protocol Flexible PE-1 is €15,000 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**.

APPENDIX 1: COTREP test request form

REQUESTER

COMPANY:	*Please complete*
FIRST NAME/LAST NAME:	*Please complete*
POSITION:	*Please complete*
EMAIL:	*Please complete*
TELEPHONE:	*Please complete*

IMAGE OF THE PACKAGING

DESCRIPTION OF THE TEST PACKAGING

PACKAGING TYPE:	*E.g. bottle, dispenser bottle, pot, tray, tube, etc.*
MAJORITY RESIN:	*Please complete*
PACKAGING STRUCTURE:	*If multilayer, describe the layers.* *Specify the % by mass of each component (barrier, additives, adhesive, tie layer, etc.)*
FORMING METHOD:	*Please complete*
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 flexible PE packaging 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 flexible PE regeneration

STRUCTURE OF THE TEST PACKAGING	DESCRIPTION	PENETRATION RATE TO BE APPLIED (x and w)	GENERAL NOTICE REFERENCE
EVOH	Flexible PE packaging with EVOH barrier	2% and 5%	*AG68*
PP	PP (flexible or rigid) that is not separable from a flexible PE packaging item	2% and 5%	*AG69*
PA	Flexible PE packaging with polyamide (PA) barrier	1% and 4%	*AG70*
SIOX	Flexible PE packaging with SiOx barrier	1% and 5%	*AG72*
WATER-BASED COATING	Flexible PE packaging with PVOH type water-based coating	4% and 8%	*AG73*
METALLISATION	Flexible PE packaging with deposition of a thin aluminium layer	10% and 15%	*AG74*
COMPOSTABLE	Compostable flexibles	1% and 5%	*AG58*
ORIENTED PE	Flexible PE with one or more oriented PE layers	5% and 10%	*AG75*
EVA	Flexible PE with EVA	1% and 5%	*AG76*

Examples:

➔ **Mono-PE stand-up pouch and EVOH barrier:**

The penetration rates for the EVOH barrier study should be applied for testing. Incorporation of 2% and 5% EVOH should be tested in the blend.

➔ **Mono-PE stand-up pouch with water-based coating and a PP tap:**

The water-based coating and PP categories may be applied to the packaging item. Consequently, the highest market penetration rates should be applied, i.e. those for water-based coating: 4% and 8%.

➔ **Flexible PE packaging with other components:**

There is currently no COTREP general notice for AlOx elements. Consequently, market penetration rates are unknown. It is currently not possible to ensure regeneration testing on flexible PE packaging with an AlOx barrier.

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 Flexible PE-1 protocol provides the success 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.)
- Change in wash water appearance (staining or foam formed, etc.)
- Moisture content after washing
- If optional stage 1 done: contaminants on the flakes

Flotation:

- Position of the test packaging in the bath (float, sink, suspended fraction)
- Quantity of float fraction
- Change in flotation water
- If optional stage 2 done: adhesive, paper or ink on the flakes, position of the test packaging (float, sink, suspended fraction)

Drying:

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

Extrusion/Granulation:

- Extruder operation during testing
- Extrusion process stability during sample transformation
- Material stability (expansion, strand breakage, etc.)
- Degassing operation
- Filter change during granulation

Characterisation of granulate:

- Variation between GM samples and the GT standard sample