



# TEST PROTOCOL FOR FLEXIBLE PE-2

## PRODUCTION OF RECYCLED PE FILMS BY BLOW EXTRUSION

### COTREP

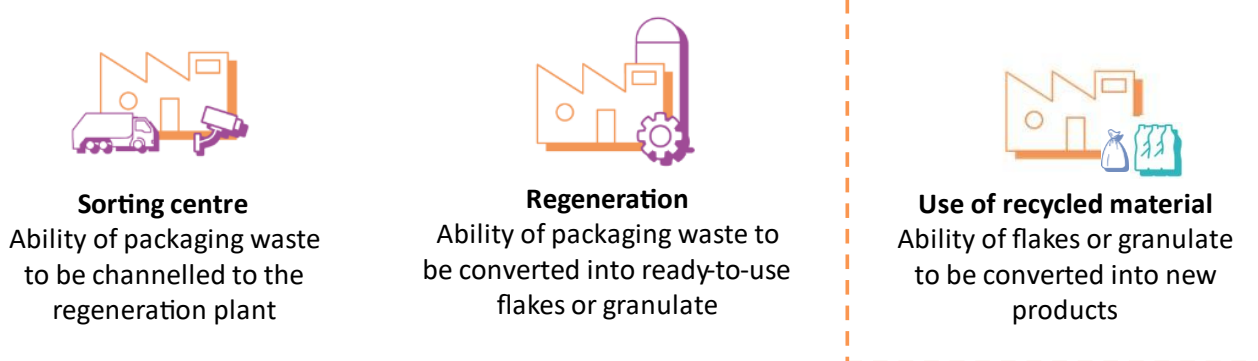
The mission of the Technical Committee for the Recycling of Plastic Packaging (COTREP) is to help designers and decision-makers develop recyclable plastic packaging while also providing scope for innovation. The committee includes various stakeholders in the plastic household packaging chain (Valorplast, 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	February 2025	Addition of details concerning moisture monitoring

## 1. CONTEXT

COTREP has drawn up this protocol in collaboration with French manufacturers of PE film through the blow extrusion process. It is representative of practices adopted by producers receiving rPE<sup>1</sup> granulate from streams of flexible PE packaging sourced from French selective collection. Its purpose is to specify tests to be performed for assessing the suitability of recycled flexible PE granulate produced during regeneration tests in accordance with protocol Flexible PE-1 for transformation into films by blow extrusion. This is currently the major outlet for recycled flexible PE.

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.



Study scope

Figure 1: Scope of the Flexible PE-2 protocol

<sup>1</sup> 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.

This protocol takes account of current technical knowledge and processes for transforming rPE granulate into film. COTREP may review this protocol with regard to developments in the use of granulate from recycled flexible PE household packaging and related manufacturing processes if needed.

Results obtained from tests performed based on this protocol are insufficient for determining packaging recyclability. This protocol merely reflects the process of transforming regenerated granulate into film and provides no basis for judging the suitability of packaging for sorting or regeneration.

## 2. AIMS

This test protocol should be implemented after and in addition to the flexible PE packaging regeneration protocol (Flexible PE-1). Its aim is to allow companies to test the production of recycled PE films including regenerated granulate produced from test packaging in semi-industrial conditions. It covers:

- Technical feasibility of transforming granulate into new film,
- An analysis of the quality of the film produced.

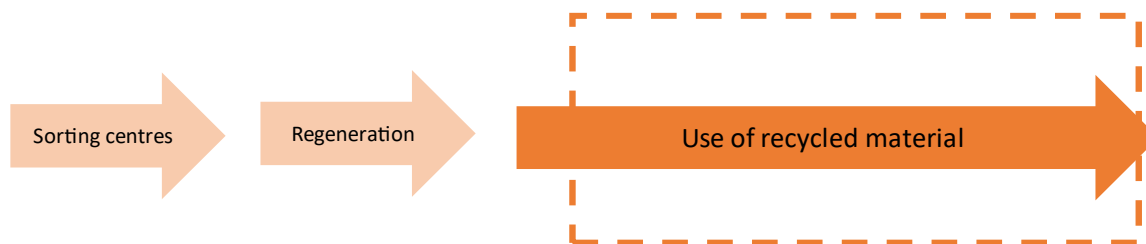


Figure 2: Analytical scope of the blow extrusion protocol

## 3. TERMS OF REFERENCE

Any company (packaging manufacturer, marketer, resin manufacturer, distributor, etc.) seeking to determine how granulate produced from a specific packaging item according to protocol Flexible PE-1 impacts the process of forming by blow extrusion can use this protocol to perform testing.

Companies wishing to perform 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: After validating the success criteria for protocol Flexible PE-1

Once success criteria for protocol Flexible PE-1 have been validated, the **Requester** should confirm that it wishes the **Laboratory** to perform tests in accordance with protocol Flexible PE-2.

### Step 2: Prepare for application of protocol Flexible PE-2

The **Requester** should provide the selected **Laboratory** with GM1, GM2 and GT granulate prepared in accordance with the COTREP test protocol on flexible PE regeneration (Flexible PE-1). The test comprises the following steps:

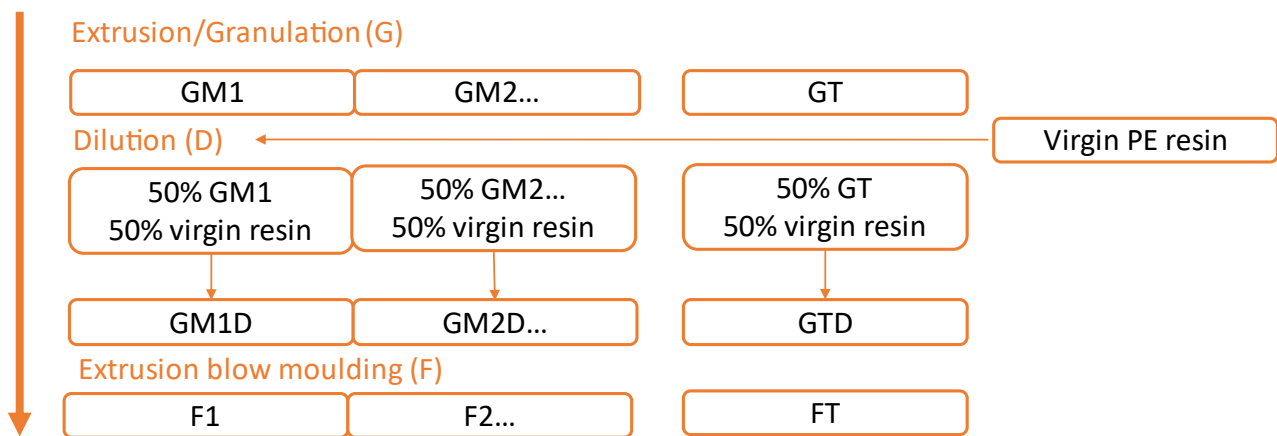


Figure 3: Description of blow extrusion protocol steps

A sufficient quantity of granulate should be supplied to manufacture **25kg** of each blend (GTD, GM1D, GM2D, etc.).

## 5. METHODOLOGY

The protocol set out below is intended for COTREP-certified **Laboratories** with equipment representative of current transformation processes applied in industrial units. The following steps should be performed:



Figure 4: Detailed description of blow extrusion steps

### Step 1: Preparing mixtures

Granulate GT, GM1, GM2, etc. prepared in advance according to the COTREP PE regeneration protocol (Flexible PE-1) should be mixed with a virgin LDPE resin at a rate of 50% by weight to produce mixtures GTD, GM1D, GM2D, etc. The mixtures should ideally be produced mechanically.

The virgin LDPE granulate used for these tests should be free-radical LDPE granulate of a density of 0.92 kg/m<sup>3</sup> and a grade close to 0.5. The following products have been identified as appropriate:

- EXXON 171BA
- SABIC 2801TH00W
- DOW 535E

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

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

### Step 2: Blow extrusion

The moisture content of each batch produced from the extrusion/granulation process, GT, GM1, GM2, etc., should be measured and recorded in the report. The target moisture content for starting the blow extrusion process is < 0.3%.

If the moisture content of the GT batch does not meet this target, drying will be implemented for all the batches. The conditions applied, particularly the drying time, drying temperature and equipment used on the GT batch, will be recorded in the test report. The same conditions will be reproduced for all the batches. The moisture content of each batch, GT, GM1, GM2, etc., after drying should be measured and recorded in the report.

The prepared mixtures should be extruded to produce a film using the blow extrusion method. At least one zone should be 210°C during the blow extrusion stage. The following implementing conditions are recommended for testing:

- Blow-up ratio – between 2.5 and 3
- Filter mesh size 150µm
- Film thickness 50 microns - Tolerance: +/- 5% on average and occasionally +/- 20%

A filter change should be performed for each new mixture. The equipment used and the extrusion conditions applied should be recorded in the final report including the following information:

- Typical extruder: (screw diameter, L/D ratio)
- Blow film die diameter
- Temperatures of the different heating zones
- Pressures/amperage
- Air gap
- Extrusion speed
- Blow pressure
- Cooling type (internal/external, air/water)
- Bubble width
- Extrusion time

The parameters of the blow extrusion 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 20 linear meter sample of each film produced will be taken midway through production once “stabilised” and will be kept by the **Laboratory**.

**Blow extrusion: success criteria**

- No faults or damage to the line due to the nature of samples (clogging, etc.)
- No bubble deterioration during implementation

The final report should include the following observations:

PROPERTIES EXAMINED	ANTICIPATED RESULTS
IMPLEMENTATION	Observations
EMISSIONS OF FUMES OR ODOURS	Observations
EXTRUSION PARAMETERS	Variation compared to the control sample
FILTRATION	For each filter, provide the reference, visual assessment and photographs
BUBBLE SIZE	Bubble stability (video of 30 seconds duration taken from the same fixed point in each <b>Laboratory</b> )
FILM APPEARANCE	Observations (bubbles, holes, white marks, etc.)
DIE DEPOSIT	Observations

### Step 3: Characterisation of films produced

The samples of film for characterisation will be taken midway through production once “stabilised”. These samples should be characterised based on the tests set out below. The results should be included in the report.

#### → Dimensional properties

PROPERTIES EXAMINED	STANDARDS	ANTICIPATED RESULTS
FLAT WIDTH OF THE EXTRUDED BUBBLE	ISO 4592	Values
SHRINKAGE RATE (LONGITUDINAL/TRANSVERSE)	NFT 54-115	Values
THICKNESS PROFILE	ISO 4593	Values
MEAN THICKNESS	ISO 4591	Values

#### → Mechanical properties

PROPERTIES EXAMINED	STANDARDS	ANTICIPATED RESULTS
ELONGATION AT BREAK	NF EN ISO 527-3	Values
BREAKING STRESS	NF EN ISO 527-3	Values
BREAKING STRENGTH	NF EN ISO 527-3	Values
COEFFICIENT OF FRICTION (STATIC/DYNAMIC)	NF EN ISO 8295	Value
SUITABILITY FOR SEALING	/	Measurable with KOPP equipment

#### → Optical and visual properties

PROPERTIES EXAMINED	STANDARDS	ANTICIPATED RESULTS
COLORIMETRIC TESTING	/	Testing of Delta E versus a standard sample
FILM APPEARANCE AFTER ROLLING	/	Observations regarding gels, surface defects, etc.
GELS	/	Visual observation
SURFACE DEFECTS	/	Observations

#### Characterisation of films: success criteria

- Comparison of surface defects observed versus control sample\*
- Variation < 10% for dimensional properties versus control sample
- Variation < 10% for mechanical properties versus control sample

\*Note that there is currently no protocol concerning assessment of film appearance.

## 6. TEST REPORT

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

- The report concerning regeneration protocol Flexible PE-1.
- 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 photographs for each step.
- Any observations to be made during the tests should be included in the report and are provided in **APPENDIX 2**.
- 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:

*"The tests were performed according to COTREP protocol Flexible PE-2 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-2 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**.

# 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*

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

**PACKAGING  
STRUCTURE:**

**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*

*Any other potentially useful information for the test*

**COMMENTS:**

<b>Company stamp:</b>	<b>Date:</b>	<b>Last name, first name and signature:</b>
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## APPENDIX 2: Observations to include in the report

The Flexible PE-2 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.

### Blow extrusion:

- Line operation relating to the nature of samples (clogging, etc.)
- Bubble appearance and behaviour during implementation
- Bubble stability, presence of porosity

### Characterisation of films:

- Surface defects observed versus control sample
- Dimensional properties versus control sample
- Mechanical properties versus control sample