



TEST PROTOCOL RIGID PE-2

MANUFACTURE OF RECYCLED RIGID PE BOTTLES BY EXTRUSION BLOW MOULDING

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	October 2024	Initial version

1. CONTEXT

COTREP has drawn up this protocol in collaboration with French manufacturers of extrusion blow moulded polyethylene bottles. It is representative of industrial practices adopted by producers receiving rPE granulate from streams of rigid PE sourced from French selective collection. Its purpose is to specify tests to be performed for assessing the suitability of recycled rigid PE granulate produced during regeneration tests in accordance with protocol Rigid PE-1 for conversion into bottles by extrusion blow moulding (currently one of the two most common outlets for recycled rigid PE and the most demanding in terms of expected characteristics).

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.

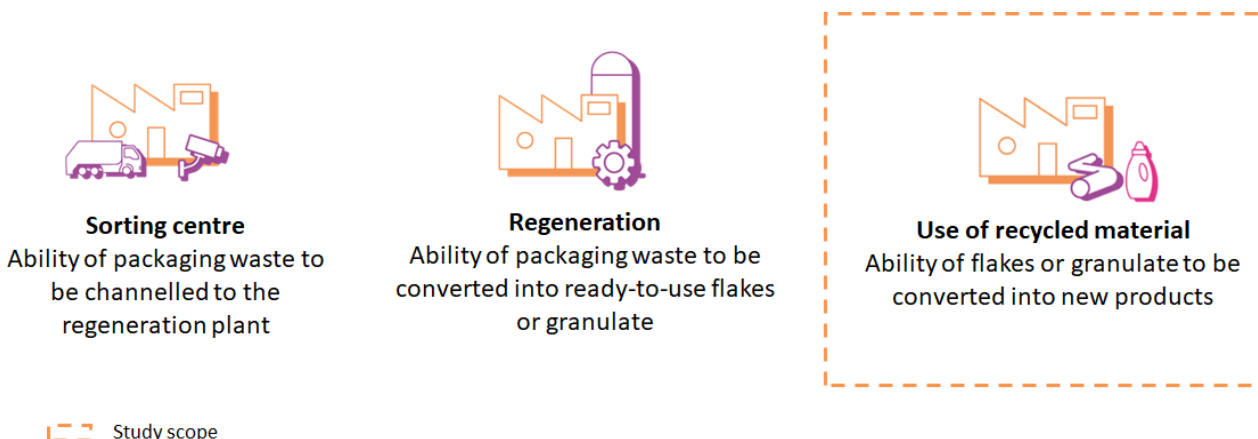


Figure 1: Scope of the Rigid PE-2 protocol

This protocol takes account of current technical knowledge and processes for transforming rPE granulate into bottles.

COTREP may review this protocol with regard to developments in the use of granulate from recycled rigid 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 bottles and provides no basis for judging the suitability of packaging for sorting.

2. AIMS

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

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

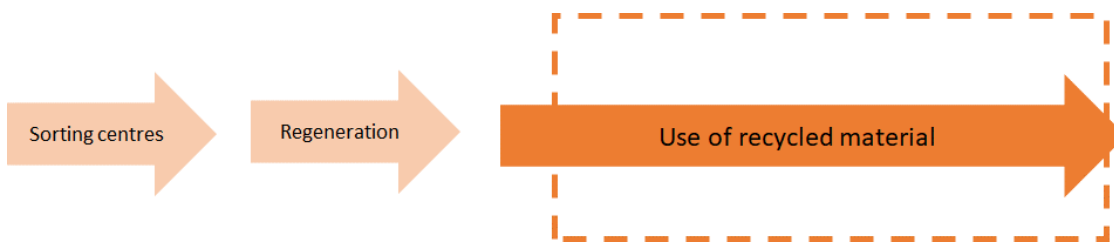


Figure 2: Analytical scope of the extrusion blow moulding protocol

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 Rigid PE-1 impacts the process of forming by extrusion blow moulding can use this protocol to perform testing.

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

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

Step 2: Prepare for application of protocol Rigid PE-2

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

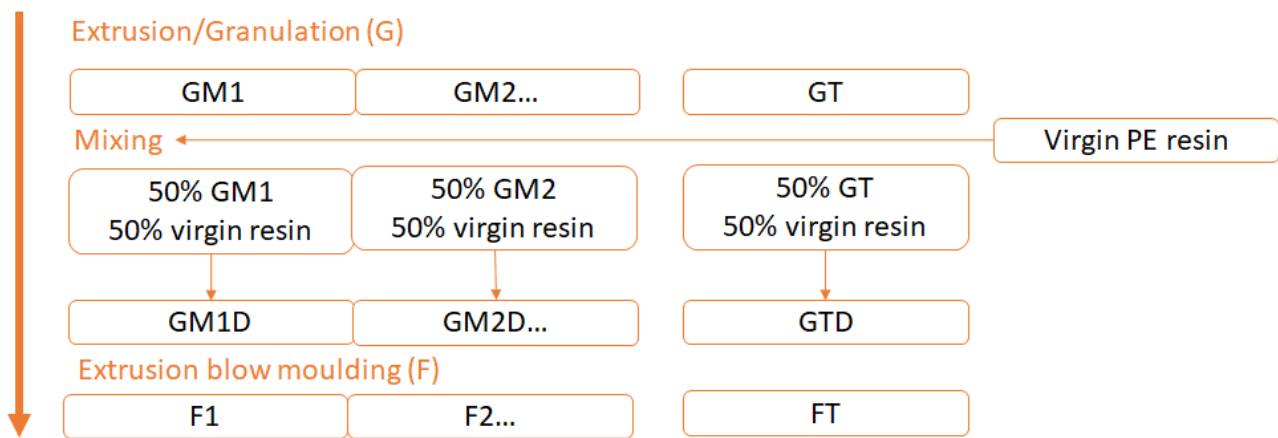


Figure 3: Description of extrusion blow moulding protocol steps

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:

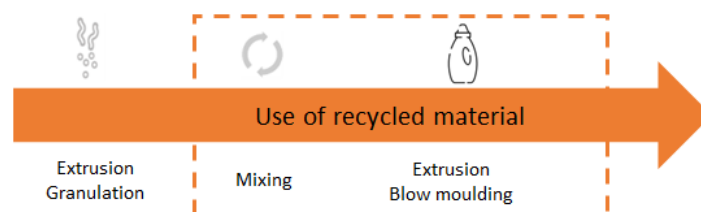


Figure 4: Detailed description of extrusion blow moulding steps

Step 1: Preparing mixtures

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

The virgin HDPE granulate used for these tests should be HDPE granulate of a density of $\sim 0.954 \text{ kg/m}^3$ and a grade close to 0.2. The following products may be used:

		MFI (190 °C / 2.16kg)	MFI (190 °C / 21.6kg)	Density
TOTAL	HDPE 5502	0.25	22	0.954
INEOS	RIGIDEX HD 5502 S	0.2	-	0.954
TOTAL	ACESO HDPE 5502 R3	-	22	0.954
CHEVRON PHILLIPS	MARLEX HHM 5502 BN	0.35	-	0.955

The **Laboratory** should order the materials required for testing and specify the product used in its final report. A sample of approximately 150 g of each mixture will be kept by the **Laboratory**.

Step 2: Extrusion blow moulding

The prepared mixtures should be extruded to produce a bottle using the extrusion blow moulding process. The following implementing conditions are recommended for testing:

- Bottle thickness: 1mm for specimen cut-out and ≤ 0.9 mm in size
- Extrusion blow moulding temperatures $180\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$

The equipment used, extrusion conditions applied and bottle properties should be recorded in the final report including the following information:

- Extruder type (screw diameter, L/D ratio) and punch/die tooling
- Temperatures of the different heating zones
- Pressures/amperage/speed
- Bottle properties (thickness, weight, capacity, shape)
- Production length and cycle time
- Smell if unusual

The parameters of the extrusion blow moulding 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.

3 copies of bottles for each mixture will be kept by the Laboratory.

Extrusion blow moulding: success criteria

- No faults or damage to the line due to the nature of samples (clogging, etc.)
- No deterioration of the parison or bottles 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
BOTTLE APPEARANCE	Observations (bubbles, holes, white marks, etc.)
TOOL CLOGGING	Observations

Step 3: Characterisation of bottles produced

All bottles produced should be characterised based on the tests set out below. Results should be included in the report.

→ Dimensional properties

PROPERTIES EXAMINED	STANDARDS	ANTICIPATED RESULTS
THICKNESS PROFILE	According to the determined inspection plan)	Values
MEAN THICKNESS	ISO 4591	Values
BOTTLE WEIGHT	/	Values
BRIM VOLUME	/	Values
OVERALL HEIGHT	/	Values

→ Mechanical properties

PROPERTIES EXAMINED	STANDARDS	ANTICIPATED RESULTS
TOP LOAD	ASTM D2659*	Values
DROP IMPACT	ASTM D2463*	Values
BENDING	/	Values
TRACTION / MODULUS / TENSILE STRESS / ELONGATION	NF EN ISO 527-3	Value
STRESS CRACKING	ASTM D1693	Values

* Vertical compression force on the bottle ring

→ Optical and visual properties

PROPERTIES EXAMINED	STANDARDS	ANTICIPATED RESULTS
COLORIMETRIC TESTING	/	Testing of Delta E versus a standard sample
BOTTLE APPEARANCE	/	Observations regarding gels, surface defects, etc.
GELS	/	Visual observation
SURFACE DEFECTS	/	Visual observation
LEAK-TIGHTNESS	/	Number of non-leak-tight bottles

Characterisation of bottles: success criteria

- Variation < 10 % for dimensional properties versus control sample
- Variation < 10 % for mechanical properties versus control sample

6. TEST REPORT

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

- The report concerning regeneration protocol Rigid 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 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 2**
- The samples collected by the **Laboratory** at the various stages will be available on request by the **Requester**. All materials related to the campaign will be kept by the Laboratory for 6 months after 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, and send the test reports to COTREP.

The report should include the following declaration:

"The tests were performed according to COTREP protocol Rigid PE-2 for rigid 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

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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 Rigid 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*

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
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APPENDIX 2: OBSERVATIONS TO INCLUDE IN THE REPORT

The Rigid 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.

Extrusion blow moulding:

- Line operation due to the nature of samples (clogging, etc.).
- Appearance of parison or bottles during implementation.

Characterisation of bottles:

- Variation for dimensional properties versus control sample.
- Variation for mechanical properties versus control sample.