CEN WORKSHOP AGREEMENT

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Challenge test for analysing presence of contaminants before / after samples decontamination

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Foreword

This CEN Workshop Agreement (CWA 18305:2025) has been developed in accordance with the CEN-CENELEC Guide 29 "CEN/CENELEC Workshop Agreements – A rapid way to standardization" and with the relevant provisions of CEN/CENELEC Internal Regulations - Part 2. It was approved by the Workshop CEN "Challenge test for analysing presence of contaminants before / after samples decontamination", the secretariat of which is held by "UNE" consisting of representatives of interested parties on 2025-07-02, the constitution of which was supported by CEN following the public call for participation made on 2025-05-30. However, this CEN Workshop Agreement does not necessarily include all relevant stakeholders.

The final text of this CEN Workshop Agreement was provided to CEN for publication on 2025-11-11.

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Introduction

This CEN Workshop Agreement establishes a new harmonized process to check the efficiency of a plastic waste decontamination process in providing clean samples (granulates, material samples, or other forms) with a challenge test analyzing presence of contaminants before / after decontamination. The challenge test process was created and validated during SURPASS Horizon Europe project. FDA guidance document “Use of Recycled Plastics in Food Packaging (Chemistry Considerations): Guidance for Industry” (July 2021) served as reference for selecting the challenge test principles. No existing EU guidance was identified during preliminary work to establish this protocol. Different applications are possible for the challenge test and are given in the scope paragraph. The process establishes concrete elements on the artificial contaminants and define the challenge test method. This challenge test uses ingredients of lower toxicity to obtain better working conditions for laboratory manipulations in terms of health protection. The possibility that chemical contaminants in plastic waste intended for recycling may remain in the recycled material and could migrate into the food the material contacts is one of the major considerations for the safe use of recycled plastics for food-contact applications. Other aspects of plastics recycling, such as microbial contamination and structural integrity of the recycled plastic, are also important, but are not discussed in this document.

Important note: The use of the word “should” in CEN Workshop Agreement means recommendation, the word “may” means permission, the word “can” means possibility, and the word “shall”, must mean obligation.

1 Scope

This CEN Workshop Agreement establishes a challenge test (artificial contamination) process for plastic samples in form of granulates. Challenge tests can also be applied to plastics material samples, or other forms. The challenge test comprises a selection of contaminants, an artificial contamination process, completed by the verification of obtained contamination level. Quantitative analytical techniques to measure contamination before and after a recycling decontamination process are also included to define the performance level of the recycling decontamination process. As such, this challenge test is not applicable for chemical recycling processes. The decontamination process itself is not part of this challenge test.

The challenge test can be applied on polymers, and in a limited manner on metallic compounds. Application on metallic compounds was not investigated during SURPASS Horizon Europe project; adequacy with samples preparation process and protection of workers has therefore not been examined in this case.

The challenge test has been verified during SURPASS Horizon Projects for a selection of contaminants and a polyethylene (PE) polymer. As the process uses an extrusion process at polymer melt temperature in the range of 200°C to 220 °C, it is only adequate for contaminants that are not volatile at such temperatures.

NOTE For other application purposes, i.e. other categories of contaminated samples and/or other selection of contaminants, other polymers, the challenge test needs to be verified before implementation.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp/

— IEC Electropedia: available at http://www.electropedia.org/

3.1
twin screw extruder
co-rotating twin screw extruder for industrial compounding tasks

3.2
screw configuration
screw configuration for the twin screw extruder can be adapted to the tasks for the compounding process

3.3
barrel configuration
barrel of the twin screw extruder can be set up according to the compounding task

3.4
recycling material
material which will be tested for its recycling capabilities

3.5
surrogate substance

substance used to simulate or represent real plastic additives, monomers, or contaminants in testing processes, such as migration tests, material compatibility studies, or safety evaluations

3.6
quantitative analytical method

analytical technique used to **determine the exact amount or concentration of a substance** in a sample

Note 1 to entry: It measures **how much** of a particular component is present, usually expressed in units such as ppm or percentage.

Note 2 to entry: This includes both the extraction method used to isolate the substance from the sample and the analytical method employed to detect and quantify it using the standard corresponding to the substance of interest.

4 Challenge test (artificial contamination) process

4.1 General

The challenge test described in this CWA is an artificial contamination process. Several approaches for the decontamination of polymer materials have been developed, among them dissolution technologies or extractive technologies. As such, this challenge test is not applicable for chemical recycling processes.

In order to be able to compare these technologies concerning their applicability for a specific material stream, an evaluation procedure needs to be established.

This relays on an artificial contamination of the material stream with surrogate substances, which are well analysable in the material.

4.2 Artificial contaminants

Various surrogates may be chosen according to their chemical properties, such as polarity (polar or non-polar) and volatility (volatile or non-volatile).

Different sources propose substances as surrogates for this compound:

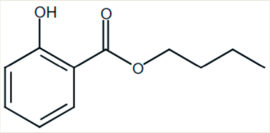
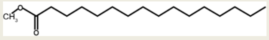
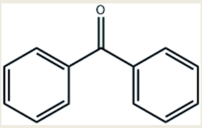
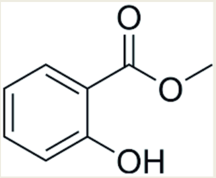
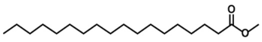
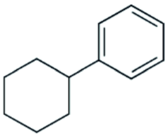
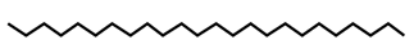
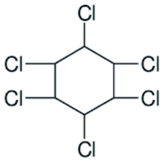
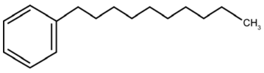
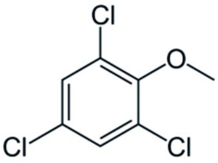
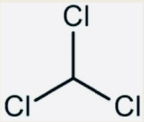
- FDA (FDA - Office of Food Additive Safety, Division of Food Contact Substances in the Center for Food Safety and Applied Nutrition at the U.S. Food and Drug Administration, July 2021);
- Publication of (WELLE, 2013). The following three substances from Table 1 are well suitable for the evaluation using an extrusion process.

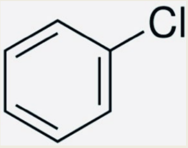
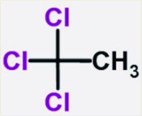
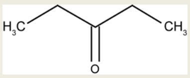
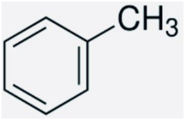
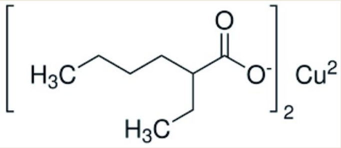
For the SURPASS PROJECT only three substances were selected and verified at a polymer melt temperature of 200 °C to 230 °C, as they have high boiling points, are not volatile in an extrusion process at approx. 200 °C to 220 °C and have a low hazardous potential for the worker. The three substances have different polarity and do therefore not behave in the same way in an extruded sample. They are listed below:

- Butyl salicylate,
- Phenylcyclohexane,
- Methylpalmitate.

However, other substances may be retained depending on whether they are polar or non-polar in nature. Table 1 provides a list of possible substances. For the polymer melt processing, the boiling point and decomposition temperature of the contaminant has to be taken into account.

Table 1 — Model contaminants for the decontamination process development

Category	Surrogate substances	CAS-Nr	Structure	Electric dipole moment (D)	Boiling point (°C)
Non-volatile - polar	Butylsalicilate liquid	2052-14-4		Between 2,41 (me-salicylate) and 3,15 (phenyl salicylate)	268
Non-volatile - polar	Methylpalmitate solid	112-39-0		1,55	185
Non-Volatile Polar	Benzophenone solid	119-61-9		3,0	305
Non-Volatile Polar	Methyl salicylate liquid	119-36-8		2,7	222
Non-volatile - Non polar	Methyl stearate	112-61-8		2,0	298
Non-volatile - Non polar	Phenylcyclohexane liquid	827-52-1		0,60	240
Non-volatile non polar	Tetracosane liquid	646-31-1		~0	391
Non-volatile non polar	Lindane solid	58-89-9		2,3	323
Non-volatile non polar	1-Phenyldecane liquid	104-72-3		0,35	315
Non-volatile non polar	2,4,6-Trichloroanisole solid	87-40-1		1,9	242
Volatile Polar	Chloroform liquid	67-66-3		1,04 to 1,15	61

Category	Surrogate substances	CAS-Nr	Structure	Electric dipole moment (D)	Boiling point (°C)
Volatile Polar	Chlorobenzene liquid	108-90-7		1,69	132
Volatile Polar	1,1,1-Trichloroethane liquid	71-55-6		2,17	74
Volatile Polar	Diethyl ketone liquid	96-22-0		2,70	102
Volatile Non Polar	Toluene liquid	108-88-3		0,36	102
Heavy Metal	Copper(II) 2-ethylhexanoate solid	149-44-0		2 to 4	Not applicable

The list of substances is not conclusive but can be widened. The SURPASS project e.g. has not investigated to use of any metallic compounds. For each contaminant it needs to be carefully examined, that it does not harm the worker carrying out the experiment e.g. due to low boiling point of the substance resulting in harmful decomposition products or vapours. Furthermore the equipment used needs to be protected from e.g. abrasive materials, like ceramics or metals that can harm the extrusion line.

Furthermore it must be noted that the substances can react with the polymer. In SURPASS project only the decontamination of PE was examined. Substances not reacting with the polymer should be chosen to be sure, that the analysis is able to detect the decontamination efficiency of the process by defining the content of the original substance.

4.3 Controlled contamination process

The contamination process in SURPASS Project is described hereafter.

The contaminants are compounded in an extrusion line to the materials aiming for a concentration of 1000 ppm of each material. As the dosing of small amounts in extrusion can be a challenge, the contaminant mixture is firstly dissolved to acetone, in a ratio of 3:7 contaminant:acetone.

NOTE The soaking of the pellets is an alternative, requiring however higher amounts of the contaminant and storing times. It would be an interesting alternative process to examine in future.

However, other contamination methods exist, such as soaking granules in contaminants, but this approach requires larger amounts of contaminant and longer storage times. It would be interesting to explore these two approaches in future work.

This process is applicable for all types of polymers until the extrusion temperature gets too high (i.e. above 220 °C) and the contamination will evaporate from the polymer at the die or the process becomes impossible to handle.

For the dosing of the low amounts of contaminant, we propose to use acetone as a carrier, as it is easily removed in the extrusion line due to the operating temperature. The acetone-contaminations ratio should not be seen as a fixed ratio. For example, in the SURPASS project case we used 10 kg/h polymer throughput and needed a minimum of 100 g/h throughput for the liquid dosing system to ensure a stable run (contaminants 30 g/h needed). With other throughputs or maybe other dosing equipment the ratio with acetone can be changed. Butyl salicylate and phenylcyclohexane are liquid, the methylpalmitate is a solid at room temperature. To be able to dose the methylpalmitate with a liquid dosing system the acetone was also added as a carrier solvent. The acetone is evaporated before the melting zone with the target to have no foaming polymer at the die and no degassing of the melt is needed. This would or may lead to a significant and unknown loss of contaminants in the degassing zone regarding the present melt temperature.

This acetone-contaminant mixture is used as feed in the extrusion. The mixture is fed into an open block section to the unmolten granules. An atmospheric degassing section is set up with low barrel temperatures (below the melting point of the polymer but significantly higher than the boiling point of the acetone). The acetone evaporates in this zone instantly and the contaminant retains in the polymer. The contaminants are mixed into the polymer in the melting zone of the extruder. In the screw section with the molten polymer the melt is softly mixed and not degassed. The temperatures and shear energy in the extruder (screw-concept) is kept as low as possible to ensure a low melt temperature at the extruder die. The melt is then cooled in a water bath and pelletized.

After pelletization the granules are stored in sealed metal buckets to prevent a possible emission of the contaminants during storage.

4.4 Process design for contamination

The polymer and the contaminants-acetone mixture are fed into a twin-screw extruder. The acetone evaporates before the melting zone of the extruder while the contaminants are mixed into the polymer from the beginning of the melting zone. The melt with the contaminants is thoroughly mixed and then pelletized.

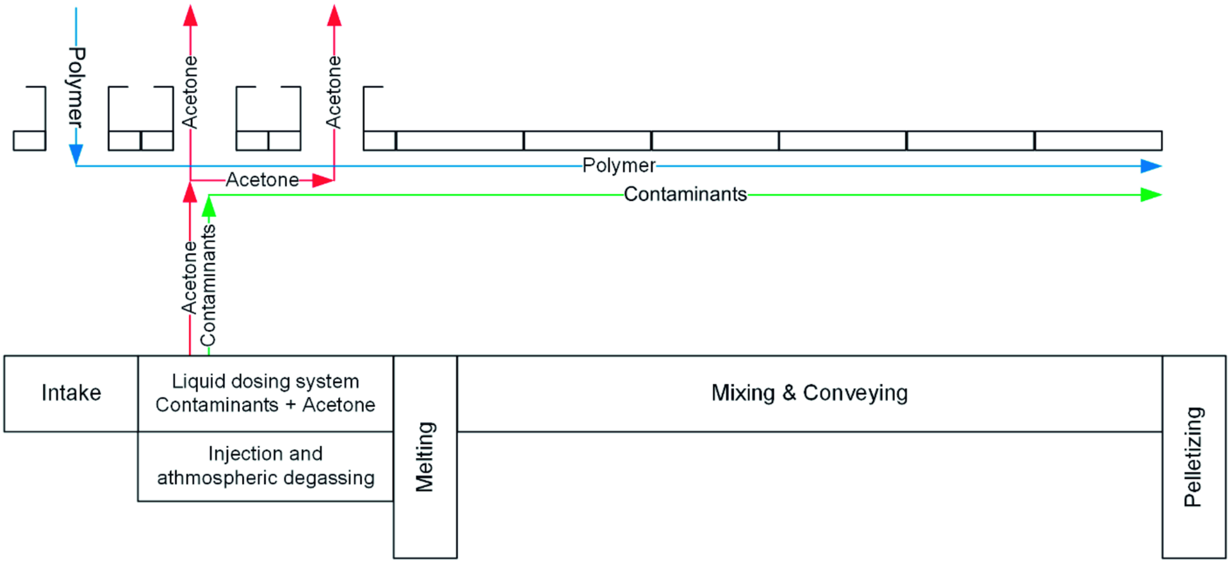


Figure A.1 — Contamination process

The verification of the level of contamination in the SURPASS project was carried out with the same methods as the determination of the contamination after the decontamination (s. 4.6 for the analysis method). The goal is to determine the real concentration on contaminated materials because a loss of surrogate substances occurs during the processing phase.

In SURPASS project the concentration of the contaminant in the pelletized polymer was measured after the contamination step to have an exact contamination level of the polymer. In the dosing of the contaminant a concentration of 1000 ppm was aimed. Depending on the polymer recipe and therefore the processing temperature (between 200 °C and 220 °C), the resulting concentration varied. Common values for Phenylcyclohexane were between 975 ppm and 1050 ppm, for Butylsalicilate between 650 and 930 and for Methylpalmitate between 800 ppm and 930 ppm.

4.5 Verification of quantity of contaminant in polymer before and after recycling or decontamination process

The accurate quantification of targeted substances in a plastic matrix requires a rigorous methodological approach, combining efficient extraction techniques with sensitive analytical methods. The same method shall be used before and after decontamination process in order to have comparative results.

The steps are described below:

— Sample preparation:

  - Selection of a representative portion of the plastic material,
  - Cryogenic grinding into particles with size between 250 µm and 500 µm to increase surface area if necessary,
  - Precise weighing for subsequent quantitative calculations.

— Extraction of Targeted Substances:

  - **Solvent extraction**: Using organic solvents suited to the polarity of the target compounds.
  - **Thermal desorption**: For volatile compounds without solvent use.

— Treatment of extract: if necessary, by different methods in order to optimize the next analytical step:

  - Filtration to remove particles interfering with the analysis,
  - Concentration or dilution in order to respect the sensibility of the analytical method,
  - Fractionation to simplify complex matrices.

— Targeted Analytical Techniques

  - Chromatography HPLC/UHPLC (High-Performance Liquid Chromatography):
    1) Ideal for non-volatile, thermolabile compounds,
    2) Paired with various detectors (UV, fluorescence, MS),
    3) Applications: additives, plasticizers, antioxidants, residual monomers.
  - Chromatography GC-MS (Gas Chromatography–Mass Spectrometry):
    4) For volatile or semi-volatile compounds,
    5) Excellent sensitivity and specificity,
    6) Applications: residual solvents, volatile plasticizers, degradation by-products,

- Py-GCMS (Pyrolysis-GC-MS): Direct analysis without prior extraction,
- ICP-MS: Used for heavy-metal and metallic additive analysis.

4.6 Test report of challenge test

The test report should include the following information:

- A reference to this document (including its year of publication);
- All information necessary for the complete identification of used ingredients to prepare artificially contaminated samples (artificial contaminants, contaminated material);
- Information on the employed equipment and settings;
- Information on the source of polymer material
- Information on contamination process setting parameters;
    - amount of contaminant dosed, carrier solvent used,
    - doing equipment, throughput,
    - extrusion line, throughput, operating temperature and barrel and screw configuration.
- Information on contamination analysis setting parameters;
- Results of the verification done on artificially contaminated samples;
- Results of contaminants quantification before and after recycling or decontamination process;
- Interpretation of contaminants quantification results;
- Any deviations from the procedure;
- Any unusual features observed;
- The date of the testing;
- Pictures of samples.

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