



# D3.1 Waste Feedstock Supply



Co-funded by  
the European Union



## PROJECT INFORMATION

GRANT AGREEMENT NUMBER	101137792
PROJECT TITLE	A novel multi-stage steam gasification and syngas purification demonstration plant for waste to hydrogen conversion
PROJECT ACRONYM	HYIELD
FUNDING SCHEME	Horizon Europe
START DATE OF THE PROJECT	01/01/2024
DURATION	48 months
CALL IDENTIFIER	HORIZON-JTI-CLEANH2-2023-1
PROJECT WEBSITE	<a href="https://hyield.eu/">https://hyield.eu/</a>

## DELIVERABLE INFORMATION

DELIVERABLE N° & TITLE	D3.1. Waste Feedstock Supply
WP NO.	WP3 Waste management methodology for waste to hydrogen applications
WP LEADER	ACES
DELIVERABLE CREATOR	ACES
CONTRIBUTING PARTNERS	-
AUTHORS	Ferran Barrera, Adrián Francés, Albert Badell
REVIEWERS	Matilde Santiago, Sònia Quintana (VEOLIA) Álvaro Sánchez Sánchez de Puerta (MAG)
CONTRACTUAL DEADLINE	M12 (January 2025)
DELIVERY DATE TO EC	24/04/2025
DISSEMINATION LEVEL	PUBLIC

## DOCUMENT LOG

VERSION	DATE	AUTHOR	DESCRIPTION OF CHANGE
V1.1	18/12/2024	Ferran Barrera, Adrián Francés, Albert Badell	Table of contents, initial version
V1.2	31/12/2024	Matilde Santiago, Sònia Quintana	Draft for revision
V1.3	29/01/2025	Luis Sanchez (WTE), Sergio Sanz (CMX)	Internal review feedback
V2.0	04/02/2025	Matilde Santiago	Second version
V2.1	11/02/2025	Javier Jiménez (MAG)	Internal review feedback
V2.2	25/02/2025	Mario Diaz (WTE)	Internal review feedback
V3.0	02/04/2025	Matilde Santiago	Third version
V3.1	16/04/2025	Alexandru Cornel Vlad (MAG)	Review feedback
V3.2	23/04/2025	Matilde Santiago	Implementation feedback
V3.4	24/04/2025	Laia Mencia (INV)	Final version approval / Ready for submission

## DISCLAIMER



**Co-funded by  
the European Union**



Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

# Content

Content .....	3
1 Introduction .....	6
2 Project planning .....	7
2.1 Stage 0. Market Study.....	7
2.2 Stage 1. General Definition of Characterization Plan.....	7
2.2.1 Waste Stream Selection Criteria .....	7
2.2.2 Preliminary Sampling Strategy.....	8
2.2.3 Definition of Sampling Methodology .....	8
2.2.4 Analytical Parameters .....	8
2.2.5 Development of Long-term Sampling and Characterization Plan.....	8
2.3 Stage 2. Execution of the characterization plan .....	9
2.3.1 2.1 Identification of possible suppliers and establishment of contact .....	9
2.3.2 On-site Waste Plant Inspection and Specific Sampling Protocol Definition .....	10
2.3.3 Periodical sampling, characterization, and analysis .....	10
2.3.4 Data Recording and Management.....	10
2.3.5 Continuous Adaptation of Characterization Process:.....	10
2.4 Stage 3: Final Waste Selection .....	11
2.4.1 Analysis and Reporting:.....	11
2.4.2 Comprehensive Evaluation.....	11
2.4.3 Results Interpretation .....	11
2.4.4 Final Selection and Implementation Plan .....	11
2.4.5 Monitoring and Continuous Improvement Plan.....	11
3 Conclusions.....	12
4 ANNEX I - Protocol for Sampling of Waste .....	13
4.1 Introduction.....	13
4.2 Sampling Types .....	13
4.3 Materials and Equipment for Sampling.....	13
4.3.1 Work materials .....	13
4.3.2 Equipment .....	13
4.3.3 Complementary office materials .....	13
4.4 Risk Prevention .....	13
4.4.1 Identification risks .....	14
4.4.2 Preventive measures .....	14
4.4.3 Required PPE.....	14
4.4.4 Training.....	14
4.5 Methodology .....	15
4.5.1 Conduct Coordination of Business Activities (CBA) prior to sampling .....	15
4.5.1.1 Sampling methodology .....	15
4.5.1.1.1 Location of Sampling Point .....	15
4.5.2 Sampling Conditions .....	15
4.5.3 Sampling Procedures .....	15
4.5.3.1 Static Sampling.....	15
4.5.3.2 Dynamic Sampling.....	16
4.5.3.3 Sample size determination .....	16

4.5.4	Material Homogenization and Sample reduction.....	17
4.5.4.1	Homogenization .....	17
4.5.4.2	Quartering Technique.....	18
4.5.5	Additional Considerations.....	18
4.5.6	Sample conservation and transport .....	19
5	ANNEX II - Determinations .....	20
5.1	Determination of apparent density .....	20
5.2	Pre-Drying and determination of humidity.....	20
5.3	Particle size distribution .....	20
5.4	Approximate characterization of the waste and obtaining the recomposed sample 21	
5.5	Crushing with cutting mill (FRITSCH pulverisette 15)(MO-3.0) .....	21
5.6	Sample storage.....	22
5.7	Ash content.....	22
5.8	Analysis of the Lower Heating Value (LHV) .....	22
5.9	Determination of volatile matter .....	22
5.10	Fixed carbon .....	22
5.11	Elemental Analysis (CHONS).....	22
6	ANNEX III - Standards .....	24

## Figure index

Figure 1.	Sample type diagram.....	16
Figure 2.	Quartering diagram, showing the “n” iterations until the final required sample.....	18

## Table index

Table 1.	Feedstock Characteristics and Operating Guidelines for the Pilot Plant.....	7
Table 2.	Minimal mass determination of Refuse derived fuel (RDF) flakes.....	17
Table 3.	Minimal mass determination of granular type Refuse derived fuel (RDF).....	17

# Consortium Partners

	Country	Country	Short name
1	MAGTEL OPERACIONES	ES	MAG
2	CEMEX ESPAÑA OPERACIONES	ES	CMX
3	VEOLIA (ACES)	ES	ACES
4	WASTE-TO-ENERGY ADVANCED SOLUTIONS	ES	WTE
5	H2SITE	ES	H2S
6	MINCATEC	FR	MIN
7	FUNDACIÓ EURECAT	ES	EUT
8	SINTEF AS	NO	SIN
9	ARISTENG	LU	ARI
10	SYNHELION	BE	SYN
11	CETAQUA-CENTRO TECNOLÓGICO DEL AGUA, FUNDACIÓN PRIVADA	ES	CET
12	INVENIAM GROUP	ESE	INV
13	LA FARGA	ES	FAR
14	ARCELORMITTAL	DE	ARC
15	ENAGAS	ES	ENG
16	CONSEJO SUPERIOR DE INVESTIGACIONES CIENTÍFICAS	ES	CSIC

# 1 Introduction

This document presents a Comprehensive Waste Feedstock Management and Characterization Plan for an innovative hydrogen production project utilizing diverse waste streams. In response to the growing global demand for sustainable energy sources and the pressing need for effective waste management, this project aims to contribute significantly to the field of waste-to-hydrogen production.

The primary objective of this plan is to establish a robust framework for identifying, analyzing, and evaluating various waste streams as potential feedstock for hydrogen production. By focusing on waste classified under European Waste Codes (EWC), the objective is to systematically characterize a wide range of waste materials, with particular emphasis on those with high organic content.

The plan outlines a structured approach encompassing:

- Preliminary sampling strategy development
- Strategic waste mapping and supplier engagement
- Comprehensive sampling and waste characterization
- Periodic waste sampling and evaluation
- Final waste selection based on rigorous criteria

To assess the efficacy of this approach, the following key performance indicators have been established:

- Development of at least 1 new methodology for waste characterization and control.
- Characterization and identification of more than 20 types of waste, showcasing the diversity of potential feedstocks.
- Exploitation of more than 10 types of waste streams for waste-to-hydrogen production, highlighting the versatility of our approach.
- Achieving less than 50% proportion of waste non-suitable for processing (rejection rate).
- Maintaining waste acquisition costs below 20€ per tonne, ensuring economic viability.

This plan adheres to relevant European standards and incorporates best practices in waste management and characterization. It is designed to be adaptable to the inherent variability in waste composition and availability, ensuring a robust and scientifically sound approach to waste-to-hydrogen production.

Note: The storage strategy is outside the scope of this deliverable and will be addressed separately by the pilot project designers.

## 2 Project planning

The Feedstock Supply Strategy for this Project is structured into four distinct stages, each designed to address specific aspects of waste identification, characterization, and selection. This structured approach ensures a comprehensive and adaptable feedstock management system.

These are the different stages defined for the process:

### 2.1 Stage 0. Market Study

The market study provides the foundation for identifying potential waste sources, which informs the subsequent characterization process. For more details, see deliverable 3.2 Market study.

### 2.2 Stage 1. General Definition of Characterization Plan

The primary objectives of this characterization plan are to:

- Identify suitable waste streams for the gasification process
- Establish a comprehensive understanding of waste composition and variability
- Develop long-term relationships with reliable waste suppliers
- Optimize the waste selection process for efficient hydrogen production

The characterization plan employs a systematic approach to waste sampling and analysis, adaptable to various waste types. This methodology is designed to be flexible, allowing for adjustments based on ongoing results and any significant changes in waste composition or generation processes.

This stage is subdivided into five fundamental steps:

#### 2.2.1 Waste Stream Selection Criteria

The parameters for waste selection will be determined based on the specifications of the results obtained during the analysis of different wastes. The following key determinations will be taken into consideration. The project will work to ensure that most of the waste supplied to the pilot falls within the ranges specified in Table 1.

Table 1. Feedstock Characteristics and Operating Guidelines for the Pilot Plant.

Key determinations	Units	MIN	MAX	TARGET	Comments
<b>Proximate analysis</b>					
Humidity	%	10	25	20	Short run tests: max. 40%
Ash content	% D.B.	-	10	5	If LHV >15 MJ/kg a.r., max. 15% Short run tests: max. 40%
Fixed carbon	% D.B.	2	-	8	
<b>Ultimate analysis</b>					
N	% D.B.	1	5	2	
S	% D.B.	0	1.5	0.1	
<b>Particle size</b>					

Granulometric study	mm	3	60	40	
Fines fraction	% W.B.	5	10	8	Short run tests: max 30%
<b>Other determinations</b>					
LHV	MJ/Kg	11.25	-	15	Short run tests: min 9 MJ/kg a.r.
Density	Kg/m3	200	450	200	Short run tests: min 100 kg/m3
Large impropers (Stones, glass, other inserts, etc)	% W.B.	0	2	None	Short run tests: max 10 %

\*Note: These are reference values with an acceptable deviation of 10%. While individual batches or deliveries may vary, the project team will work towards maintaining these specifications within operational periods.

## 2.2.2 Preliminary Sampling Strategy

Due to the accelerated project execution timeline, an initial sampling strategy was developed prior to the full characterization and sampling plan. This pre-plan stage leveraged ACES' knowledge and experience from previous projects to establish a preliminary framework. It allowed for the initiation of crucial sampling and characterization activities while the comprehensive plan was being developed, ensuring no delays in the project's progress.

## 2.2.3 Definition of Sampling Methodology

A detailed sampling methodology is established to ensure representative and consistent sample collection across all waste types. This methodology outlines the procedures for sample selection, collection, handling, and preservation. The specific details of this sampling methodology are thoroughly explained in [Annex I](#) of this document. This step is critical for ensuring that all subsequent analyses are based on reliable and representative samples.

## 2.2.4 Analytical Parameters

The collected samples undergo comprehensive analysis, including:

- Approximate analysis (humidity, ash, volatile matter and fixed carbon content).
- Determination of density and theoretical heating value.
- Ultimate analysis (carbon, nitrogen, hydrogen, sulphur and oxygen content).
- Approximate characterization.
- Particle size distribution study.

The procedure of all these determinations is specified on the [Annex II](#) of this document.

## 2.2.5 Development of Long-term Sampling and Characterization Plan

A long-term sampling plan is established. This plan defines the optimal sampling frequency for each selected waste type, taking into account the specific characteristics of the residues. For homogeneous waste samples, sampling and analysis periods of 3-4 months are typically established. In contrast, for heterogeneous waste samples and those subject to seasonal variations, more frequent sampling intervals of approximately 2 months are implemented.

The plan considers the observed variability in waste characteristics and determines key parameters for regular monitoring. A detailed sampling and analysis schedule is created. This tailored approach ensures that the sampling frequency adequately captures the waste stream's variability while optimizing resource allocation. The schedule is designed to be flexible, allowing for adjustments based on ongoing results and any significant changes in waste composition or generation processes.

## 2.3 Stage 2. Execution of the characterization plan

This stage involves the implementation of the refined sampling and analysis plan, focusing on continuous monitoring and comprehensive data collection.

The stage is structured into the following key components:

### 2.3.1 2.1 Identification of possible suppliers and establishment of contact

Based on the mapping conducted, a systematic identification of potential waste suppliers is undertaken. This process is integrated with the initial outreach, combining the request for information and the presentation of the project in a single, comprehensive approach. The initial contact and information gathering phase includes:

Formal communication with potential suppliers, presenting:

- A synthesis of the project and its objectives
- Detailed information about the project's environmental benefits
- Potential economic advantages for the suppliers

Simultaneous request for crucial documentation and information:

- Previous analytical reports or general waste characteristics
- Photographs of the waste materials
- Process diagrams from the supplier's facilities
- Mass balance or annual production data
- Current waste management practices and disposal methods
- Estimated volume of waste generated
- Ability to consistently supply the required waste types
- Compliance status with relevant regulations and standards

Arrangement of an initial site visit, if deemed necessary based on the supplier's profile and the nature of the waste.

This integrated approach allows for a comprehensive preliminary assessment of the suppliers' operational capacity and the potential suitability of their waste streams. By combining the introduction of the project with the request for information, we streamline the process and demonstrate the seriousness and professionalism of our initiative from the outset.

The goal of this comprehensive approach is to foster long-term partnerships while efficiently gathering the necessary data to evaluate the most promising waste sources. This method enhances the efficiency of subsequent stages, particularly the physical sampling and characterization processes, by providing a solid foundation of information upfront.

Through this integrated initial contact and information gathering phase, we aim to build a thorough understanding of each potential supplier and their waste streams, setting the stage for effective collaboration and successful project implementation.

## 2.3.2 On-site Waste Plant Inspection and Specific Sampling Protocol Definition

---

Technical visits to selected waste plants are conducted. These on-site inspections involve:

- Visual evaluation of facilities
- Discussions with technical personnel about available waste types and volumes
- Visual inspection of waste
- This information is used to refine the initial parameters of the sampling protocol.

The stage is structured into the following key components:

## 2.3.3 Periodical sampling, characterization, and analysis

---

Regular sampling expeditions are conducted at predetermined intervals, considering seasonal variations and other factors that might influence waste composition. Each sampling event is meticulously documented, recording physical samples, relevant environmental conditions, and observations that might impact waste quality.

Samples undergo a battery of analytical tests in line with previously established procedures, encompassing a wide range of parameters defined in Stage 2. The analytical process is rigorous and standardized to ensure consistency and comparability of results over time.

## 2.3.4 Data Recording and Management

---

Data from periodic sampling and analysis is systematically recorded and managed through:

### a) Individual Sample Reports:

Detailed reports for each sample, including:

- Sample identification and collection details
- Analytical methods used
- Detailed results of all tests performed
- Observations or anomalies noted during analysis
- Photographic report of the sample

### b) Centralized Data Compilation:

A comprehensive spreadsheet featuring:

- Columns for each analytical parameter measured
- Chronological listing of all samples analyzed
- Automated calculations for derived parameters
- Data visualization tools for quick trend identification
- Filtering and sorting capabilities for easy data manipulation

## 2.3.5 Continuous Adaptation of Characterization Process:

---

The sampling and analysis process is subject to ongoing review and adaptation, including:

- Regular assessment of the relevance and utility of each analytical parameter
- Evaluation of sampling frequency and methodology effectiveness
- Incorporation of feedback from the operational team to address practical challenges

A formal review of the characterization process is conducted quarterly, with flexibility to implement immediate changes if significant issues or opportunities for improvement are identified.

## 2.4 Stage 3: Final Waste Selection

This culminating stage represents the synthesis of all previous, where the accumulated data, analyses, and insights are leveraged to make critical decisions about the waste streams to be used in the gasification process. Key components of this stage include:

### 2.4.1 Analysis and Reporting:

A comprehensive analytical report will be generated at least at the conclusion of the characterization plan. This report will provide:

- Summary of all samples analyzed in the period.
- Trend analysis comparing current data with historical results.
- Identification of significant findings or concerns.
- Recommendations for process adjustments or further investigations.

These reports serve as the basis for informed decision-making and discussions with project partners.

### 2.4.2 Comprehensive Evaluation

- Review of all data collected throughout the project.
- Assessment of each waste stream's performance against predefined criteria.
- Consideration of long-term availability and supply chain stability.

### 2.4.3 Results Interpretation

- Trend Analysis: Examination of temporal and spatial trends in waste characteristics.
- Variability Assessment: Evaluation of consistency within and between waste streams.
- Process Implications: Assessment of gasification suitability, pretreatment needs and blending opportunities.

### 2.4.4 Final Selection and Implementation Plan

- Documentation of the final waste stream selections with detailed justifications.
- Development of a comprehensive implementation plan, including sourcing strategies, logistics, pre-treatment protocols, and quality control measures.
- Establishment of contingency plans for supply disruptions or quality variations.

### 2.4.5 Monitoring and Continuous Improvement Plan

- Establishment of ongoing monitoring protocols to ensure consistent waste quality.
- Development of a feedback loop for continuous optimization of waste selection and utilization.
- Regular review and update of waste selection criteria based on operational experience and technological advancements.

This comprehensive approach to final waste selection ensures that the chosen feedstocks are suitable for the gasification process.

### 3 Conclusions

The implementation of this Comprehensive Waste Feedstock Management and Characterization Plan is crucial for the success of the waste-to-hydrogen production project. By following this structured approach, the project team can:

1. Systematically identify and evaluate a wide range of potential waste feedstocks, ensuring a diverse and reliable supply for hydrogen production.
2. Develop and refine robust sampling and characterization methodologies, contributing to the advancement of waste-to-energy technologies.
3. Establish strong relationships with waste suppliers, fostering long-term collaborations essential for sustainable feedstock supply.
4. Continuously monitor and analyze waste characteristics, allowing for adaptive management and process optimization.
5. Make informed decisions on waste selection based on comprehensive data analysis, ensuring the most suitable feedstocks are used for hydrogen production.
6. Maintain flexibility in the face of potential variability in waste composition and availability, through well-defined contingency plans and continuous improvement strategies.
7. Contribute significantly to sustainable waste management and renewable energy production, aligning with global efforts to reduce carbon emissions and promote circular economy principles.

## 4 ANNEX I - Protocol for Sampling of Waste

### 4.1 Introduction

This section details the sampling protocol for selected waste at the waste treatment plant facilities. It is important to note that variability between waste families can be considerable, so a generic sampling scenario is considered, without peculiarities that may arise in a specific family.

This protocol adheres to the UNE-EN ISO 21645 standards - "Solid Recovered Fuels. Sampling Methods".

### 4.2 Sampling Types

The procedure distinguishes between static and dynamic sampling:

- Dynamic sampling is considered when collected directly from a continuous flow of discharging material.
- Static sampling is considered for all material stored in a discharge pit, container, or in piles on the pavement.

### 4.3 Materials and Equipment for Sampling

#### 4.3.1 Work materials

- Volumetric containers
- Manual shovel for initial handling, homogenization, and sample quartering
- Rake for handling during quartering
- Buckets or drums for sample storage or transport

#### 4.3.2 Equipment

- Braxtan ZW stainless steel single-cell scale, with a maximum weight of up to 6 kg and a precision of 0.50 g, equipped with an electronic display.
- Forced convection oven

#### 4.3.3 Complementary office materials

- Digital camera
- Notebook
- Calculator
- Smartphone (as an alternative to the above)

### 4.4 Risk Prevention

Sampling execution involves various occupational risks, both in the usual environment and when traveling to external facilities. These risks derive from the work environment, use of equipment and products, manual handling of loads, environmental conditions, and the nature and organization of tasks. It's crucial to identify and evaluate these risks, especially those arising outside the usual work environment, as they may present unforeseen dangers. To ensure workers' safety and health, it's necessary to establish a comprehensive action protocol that defines risks, implements preventive measures, provides training, and establishes emergency procedures. The goal is to eradicate or minimize these risks, ensuring a safe working environment for all involved in the project.

### 4.4.1 Identification risks

---

The main defined risks are:

- Exposure to biological agents
- Risks caused by living beings
- Exposure to harmful substances
- Falling objects
- Overexertion
- Falls at the same and different levels
- Electrical contacts
- Blows and cuts
- Run-overs
- Burns
- Traffic accidents/in itinere
- Extreme temperatures
- Anomalous or emergency situations

### 4.4.2 Preventive measures

---

Action methods, in order to avoid these risks:

- Establish efficient communication protocol with plant operators, especially the control room shift manager.
- If possible, all work carried out in the plant should involve 2 operators from the company that is carrying out the sampling and one operator belonging to the waste management plant.
- Establish a suitable work site, close to the sampling point and as far away as possible from the maximum number of existing risks.
- Use necessary Personal Protective Equipment (PPE).

### 4.4.3 Required PPE

---

- Anti-perforation, non-slip, high-top protective footwear
- Anti-pinch gloves
- Helmet
- Protective glasses
- Vinyl gloves
- Masks with filters for organic vapours, acid gases, ammonia, and dust particles
- Work overalls
- Reflective vest
- Tall rubber boots
- Kevlar gloves
- First aid kit

### 4.4.4 Training

---

All personnel involved in sampling must undergo comprehensive training on the sampling protocol, risk prevention measures, and emergency procedures. Regular refresher courses should be conducted to ensure up-to-date knowledge and skills.

## 4.5 Methodology

### 4.5.1 Conduct Coordination of Business Activities (CBA) prior to sampling

Prior to sampling, a CBA (Coordination of Business Activities) must be carried out with the entity or facility where the sampling is carried out. The objective is for the institution to communicate to all parties about the preventive measures of the work facilities, so that the different employees who coincide in the same space are informed and thus eliminate possible risks.

The management of the CBA is carried out with the support of the PRL department.

The necessary documentation is requested from the centre where the work is carried out and, on the other hand, the required documentation is provided.

### 4.5.2 Sampling methodology

#### 4.5.2.1 Location of Sampling Point

The selection of the sampling point is crucial to ensure both sample representativeness and personnel safety. The following aspects should be considered:

- **Marked area:** The area should be clearly designated as a sampling point.
- **Adequate lighting:** Ensure optimal visibility for safe and accurate sampling.
- **Ventilation:** The area should have appropriate ventilation to minimize exposure to gases or particles.
- **Away from machinery movement zones:** To avoid risks of accidents with moving vehicles or equipment.
- **Accessibility:** The point should be easily accessible for sampling personnel and necessary equipment.
- **Stable surface:** Ensure the area has firm and level ground to prevent falls.
- **Weather protection:** If possible, choose a covered area or with protection against rain or excessive wind.

### 4.5.3 Sampling Conditions

The sampling process requires a minimum of two trained individuals from the sampling team to be present at all times. These team members should possess complementary skills, with at least one having extensive experience in waste sampling techniques. Whenever possible, a qualified operator from the waste treatment plant should accompany the sampling team.

### 4.5.4 Sampling Procedures

#### 4.5.4.1 Static Sampling

Static sampling is applicable to materials stored in piles, containers, or pits. The procedure begins with a visual inspection to evaluate the apparent homogeneity of the material. Multiple sampling points are then identified, distributed throughout the pile or container. Sampling is conducted using appropriate shovels or sampling probes, with samples taken at different depths and locations to ensure representativeness. It's crucial to avoid taking samples only from the surface, as this can lead to biased results. The frequency of sampling is determined based on the variability of the material and the specific requirements of the project.

#### 4.5.4.2 Dynamic Sampling

Dynamic sampling is used for materials in continuous movement, such as those on conveyor belts. The process starts by identifying the optimal point in the process line for sampling. Once this is determined, the sampling equipment is prepared, ensuring it is correctly calibrated and positioned. Sampling involves collecting increments at regular intervals, with the interval determined by the flow speed and material variability. A minimum of 24 increments per batch is required to ensure statistical validity. The sample size is adjusted according to the flow speed and particle size of the material being sampled.

#### 4.5.4.3 Sample size determination

To ensure representative sampling, the quantity of material collected depends on the nature and particle size of the waste. The following guidelines are used to determine the appropriate sample size:

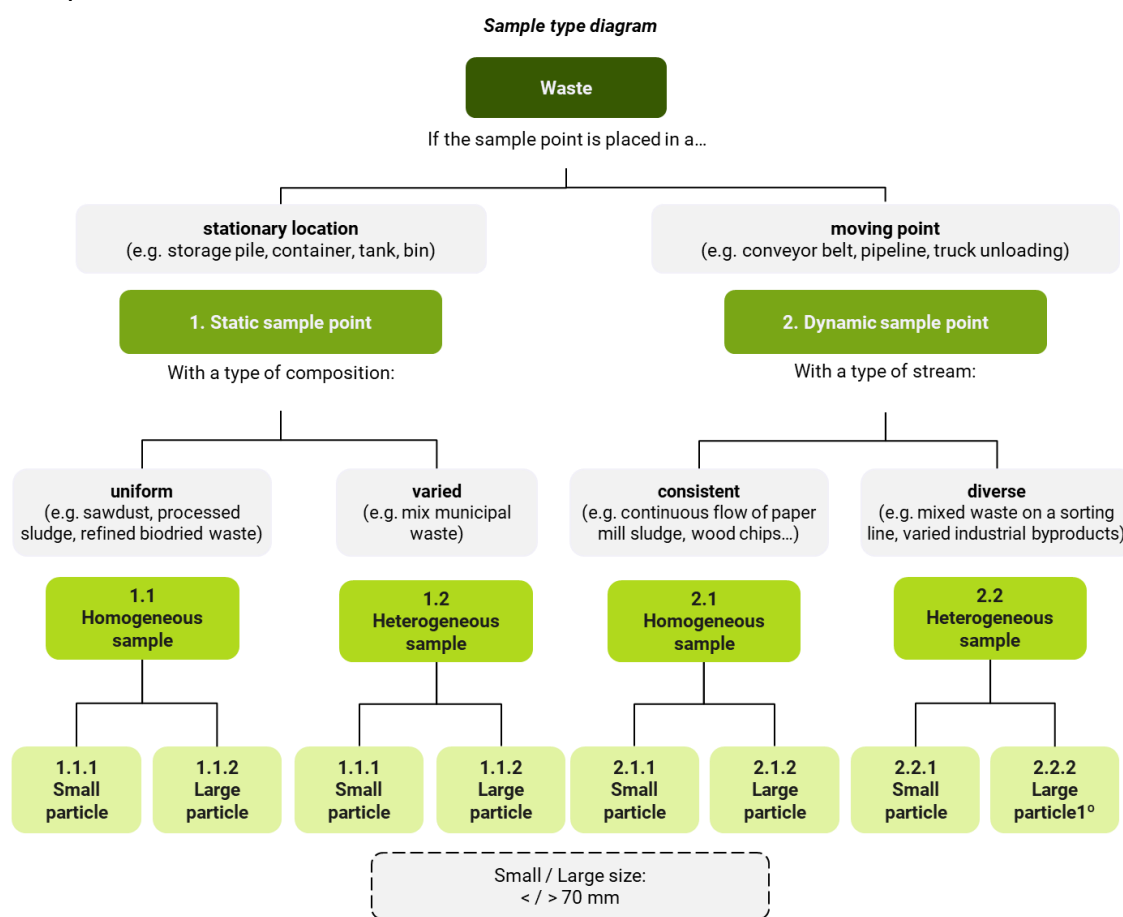


Figure 1. Sample type diagram.

Samples are categorized into four groups based on homogeneity and particle size:

- Homogeneous samples with large particle size (>70mm)
- Homogeneous samples with small particle size (<70mm)
- Heterogeneous samples with large particle size (>70mm)
- Heterogeneous samples with small particle size (<70mm)

The minimum quantities required for the sampling process will be determined depending on the following tables for Refuse Derived Fuel (RDF):

Tables 1 and 2 provide quick ways to determine the minimum sample mass based on sample type.

Table 3. Minimal mass determination of Refuse derived fuel (RDF) flakes.

$d_{95,l}$ <sup>b</sup> mm	Densidad a granel kg/m <sup>3</sup>	50	75	100	125	150	175	200	225	250	275	300
	Masa mínima de muestra kg	Volumen mínimo de muestra c										
		l										
50	0,8	16	11	8	6	5	5	4	4	3	3	3
75	2,5	50	33	25	20	17	14	12	11	10	9	8
100	6	120	79	59	47	39	34	29	26	24	21	20
125	12	230	150	120	92	77	66	58	51	46	42	38
150	20	400	270	200	160	130	110	99	88	80	72	66
200	47	940	630	470	380	310	270	240	210	190	170	160
250	92	1 800	1 200	920	740	610	530	460	410	370	330	310
300	159	3 200	2 100	1 600	1 300	1 100	910	800	710	640	580	530

Table 2. Minimal mass determination of granular type Refuse derived fuel (RDF).

$d_{95,b,c}$ mm	Densidad a granel kg/m <sup>3</sup>	50	75	100	125	150	175	200	225	250	275	300
	Masa mínima de muestra kg	Minimum sample volume d										
		l										
10	0,8	15	10	8	6	5	4	4	3	3	3	3
12,5	0,9	18	12	9	7	6	5	5	4	4	3	3
20	4	75	50	38	30	25	22	19	17	15	14	13
25	7	150	98	74	59	49	42	37	33	29	27	25
30	13	250	170	130	100	85	73	64	57	51	46	42
40	30	600	400	300	240	200	170	150	130	120	110	100
50	59	1 200	790	590	470	390	340	290	260	240	210	200

These tables serve as quick references to determine the minimum sample mass based on the waste type and particle size. Adhering to these quantity guidelines ensures that the samples collected are representative and sufficient for all subsequent analyses required in the waste-to-hydrogen project.

By incorporating this information, you provide a comprehensive guide for determining sample sizes, which is crucial for ensuring the reliability and representativeness of the samples collected. This addition enhances the overall quality and precision of the sampling procedure.

## 4.5.5 Material Homogenization and Sample reduction

After obtaining the initial sample, it's crucial to ensure its representativeness through proper homogenization and reduction techniques. This process is carried out near the sampling area to minimize material transport and potential segregation.

### 4.5.5.1 Homogenization

The collected material is thoroughly mixed using a combination of manual tools:

- A shovel is used for larger movements and initial mixing.
- A rake is employed for finer distribution and breaking up of any clumps.

The goal is to create a uniform mixture where all particles have an equal chance of being included in the final sample. This process should be carried out on a clean, flat surface to prevent contamination and ensure even distribution.

#### 4.5.5.2 Quartering Technique

Quartering is a reliable method for reducing the sample size while maintaining representativeness. The process involves the following steps:

1. Spread the homogenized material on a clean, flat surface in a circular shape with uniform thickness.
2. Divide the circle into four equal quarters using a cross-shaped divider or by drawing lines.
3. Select two opposite quarters to keep and discard the other two. The selected quarters should be chosen randomly to avoid bias.
4. Thoroughly mix the two selected quarters.
5. Repeat the process (steps 1-4) as many times as necessary until the desired sample size is reached. Conventionally, the target weight is about 5 kg, but this may vary depending on the specific requirements of the analysis to be performed.
6. The final reduced sample should be checked to ensure it meets the minimum mass requirements for the intended analyses, as specified in relevant standards or project protocols.

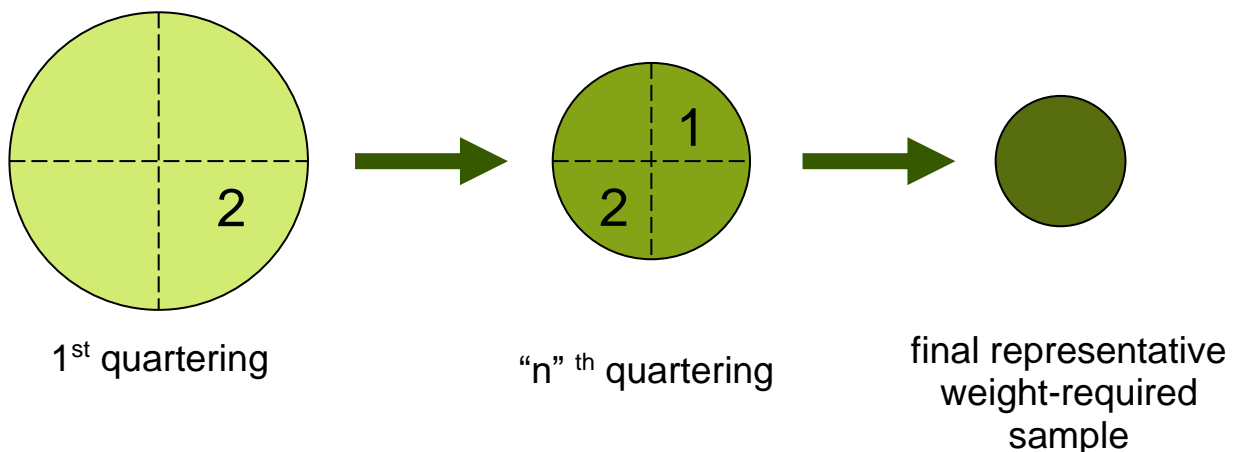


Figure 2. Quartering diagram, showing the "n" iterations until the final required sample.

#### 4.5.6 Additional Considerations

- Documentation: Record each sampling event in detail, including date, time, environmental conditions, and any relevant observations.
- Labelling: Immediately label each sample with a unique identifier, date, and sampling location.
- Safety: Strictly follow established safety protocols at all times.
- Equipment cleaning: Clean and decontaminate sampling equipment between each collection to avoid cross-contamination.

By following these detailed homogenization and quartering procedures, we ensure that the final sample accurately represents the larger batch of waste material, providing reliable data for subsequent analyses in the waste-to-hydrogen project.

## 4.5.7 Sample conservation and transport

---

This section addresses the specific protocols for laboratory sample handling and transport. For more information or comprehensive logistics information regarding industrial-scale waste transport and supply chain management, please refer to deliverable D3.2.

During this characterization phase, the transport involves small samples of approximately 15 kg from waste producers to the laboratory. Samples are stored in hermetic, watertight containers made of inert materials, filled to minimize headspace, and labelled with unique identifiers and handling instructions.

A specific safety protocol has been developed for these sample-sized quantities, including proper packaging, detailed documentation, use of appropriate personal protective equipment, and established emergency procedures. Transport preferably uses company vehicles with GPS and temperature control, though personal vehicles or couriers meeting handling requirements are acceptable alternatives. Transport should not exceed 24 hours from collection to laboratory arrival, with pre-approved exceptions for distant locations and/or stable samples.

Upon laboratory reception, samples are checked against the chain of custody form, assigned a laboratory ID, and stored in a refrigerator until analysis, which ideally begins within 24-48 hours. This protocol ensures careful handling and preservation of waste characteristics for accurate analysis, while maintaining safety standards and environmental protection throughout the transport process.

## 5 ANNEX II - Determinations

The determinations could be adapted for new types of samples.

### 5.1 Determination of apparent density

The apparent density is measured on:

- Raw material
- Dried sample
- Partially dried sample

Density determination is carried out following the internal procedure (L-IE-001) defined in accordance with ISO 17828: 2015. Applicable to samples in which sufficient quantity is not available. The followed procedure uses a graduated and transparent 5 L beaker, together with a precision balance that has a maximum deviation of 1g. The beaker is filled so the sample occupies a 5 L volume but without compacting it, then the sample is weighted. This way the volume and the mass of the sample are known so the density can be calculated. The test sample is deposited between the net weight and the normalised volume, expressing the result in conjunction with the previously measured moisture content.

### 5.2 Pre-Drying and determination of humidity

Because the sample presents humidity, it is subjected to drying in a stove following the internal protocol (L-IE-002) based on the UNE-EN-ISO 21660 - 3:2021 standard, and UNE-EN-ISO 14780-2018.

Pre-Drying

Samples must be pre-dried according to the standard UNE-EN-ISO 14780-2018. Solid biofuels. Sample preparation. Samples are subjected to thermal treatment at 40 °C for or the time required to reach approximately 20% moisture content.

Determination of humidity is carried out in two consecutive stages

1. Initial stage at low temperature ( $60\pm 5^{\circ}\text{C}$ ) for 20 hours. This first phase at moderate temperature aims to avoid calcination or carbonization problems, due to the characteristics of the sample, since it is composed of dry leaves, branches and many plant fibres.
2. Final stage at high temperature ( $105\pm 5^{\circ}\text{C}$ ) for an additional 4 hours. After preliminary drying at low temperature, the temperature is increased to  $105^{\circ}\text{C}$  to complete the elimination of residual moisture present in the sample.

This two-stage procedure, with temperatures and times, seeks to guarantee complete and adequate drying.

In parallel with the stove drying process, the moisture content is determined following the same regulatory protocol.

### 5.3 Particle size distribution

The granulometric study is carried out starting from 1000 g of raw material, pre-dry or/and dry material.

The determination of the granulometry of a sample of solid aggregates is carried out following the internal protocol (L-IE-004) based on the bibliographical references UNE 7050:1997 and UNE-EN 1015-1:1999, as well as the guidelines of NE-CEN/TR 15149- 3:2015 IN. This standardised method involves separating the aggregates into different sizes by passing them

through a series of sieves, placed from lowest to highest light and agitated with a sieving machine, the Filtra FTL-0200.

The sieves selected have been: 0.9mm, 2.8mm, 10mm, 31.5mm, 40mm, 100mm

## 5.4 Approximate characterization of the waste and obtaining the recomposed sample

The characterization is performed with raw material starting from 2 - 5 kg depending on the sample. To do this, the fractions to be separated are identified through an initial visual inspection of the sample. With this information the separation criteria are defined. The identified fractions are separated into well-identified trays. The weight of each fraction and the content as a percentage of each of the fractions, with respect to the total sample, are determined.

The fractions that may be present in the sample typology are:

- Organic matter
- Vegetable matter
- Wood
- Paper
- Plastic mix (plastic remains)
- Textile
- Cellulosic Material
- Not identified matter
- Fines 30-10mm
- Fines 10-2.8mm
- Fines 2.8-0.9mm
- Fines <0.9mm
- Inerts:
  - Multi Composite materials
  - Aluminized papers
  - Glass
  - Bones and shells
  - Ceramics and stones

The inserts fraction is not included in the recomposition because it can make the trituration process more difficult, and they are considered non-combustible for the gasification treatment.

This list has been created with the information obtained from the received samples, it is possible that with new samples more fractions will be added, as a possible fraction, to the list.

The representative sample used to make all the determinations is prepared by excluding the groups included in the "Improper material fractions".

For its recomposition, the percentage of each fraction is calculated, and the sample is reconstructed based on said percentages starting from a total weight of 1 kg.

## 5.5 Crushing with cutting mill (FRITSCH pulverisette 15)(MO-3.0)

The crushing is carried out with 500-1000g of: raw material, dried/predried material or recomposed sample. The previously pre-dried out sample is frozen with liquid nitrogen and introduced into the cutting mill to obtain a homogeneous sample of lower particle size. Particle size reduction is carried out in several stages, using a finer sieve in the grinding at each stage and finishing with the appropriate sieve. Specifically, it has been crushed first at 6 mm, then a representative sample is taken from the crushed at 6mm quartering it and then

the final sample is crushed to 0.5 mm. From this last crushing, a homogeneous sample of 50 g is taken to carry out the corresponding analyses.

## 5.6 Sample storage

The sample is stored in a vacuum to avoid its degradation or that it gains humidity. In case of the sample crushed to 0.5mm it is stored around 50g of sample to perform the corresponding analyses. The rest of the recomposed sample (around 1 kg or less) is stored in the same conditions. These samples were archived for 2 years after the project closure.

## 5.7 Ash content

The ash content is carried out with sample crushed to 0.5mm or 1 mm. This determination is carried out following the internal protocol (L-IE-014) that is based on the UNE-EN ISO 21656:2021 standard. The ash content can be determined at 3 different temperatures: 550 °C, 815 °C and 900 °C.

## 5.8 Analysis of the Lower Heating Value (LHV)

The determination of PCI is carried out solely theoretically. To determine this parameter the next formula is used to calculate it using the CHONS % of the sample:

$$LHV = HHV - (579 \times 9 \times \%H)$$

In order to obtain the High Heating Value (HHV) the following formula is used:

$$HHV = \%C \times 7835.7124 + (\%H - (\%O \div 7.9366)) \times 33912.2951 + \%S \times 2214.52046$$

These formulas are a small variation of dulong's formula, these variations are made in order it adapts better to the material it is being worked on.

## 5.9 Determination of volatile matter

The volatile matter is carried out with sample crushed to 0.5mm or 1 mm. The determination of volatile matter is carried out following the internal procedure (L-IE-015) that is based on UNE-EN 18123:2024 standard: Solid biofuels: Determination of volatile after. The process involves heating the sample to 900 °C ± 10 °C for 7 minutes in an environment with minimal air contact; to obtain these conditions the melting pots used are covered up. This percentage of volatile matter is calculated from the mass loss, discounting the loss due to moisture.

## 5.10 Fixed carbon

To determine the fixed carbon of the sample a formula is applied using the content of ashes and volatile matter:

$$fC = 100 - (\%Volatile\ matter + \%Ashes)$$

## 5.11 Elemental Analysis (CHONS)

This determination is carried out with sample crushed to 0.5mm, using the VELP EMA 502 Elemental Analysis equipment, which performs a combustion analysis.. To perform this analysis a quantity between 8 and 20 mg is weighted using a precision scale. Quantification is carried out by external calibration of different standards:

- Quantification of Nitrogen (N), Hydrogen (H) and Carbon (C) is performed by external calibration with a certified sulphanilic acid standard
- Quantification of Oxygen is performed by external calibration with a certified Ethylenediaminetetraacetic acid (EDTA)

- Quantification of Sulphur (S) is performed through external calibration with a certified standard of BBOT (2,5-Bis(5-tert-butyl-benzoxazol-2-yl)thiophene).

This method is based on ISO 16948:2015 Solid biofuels. Determination of total carbon, hydrogen and nitrogen, which is also considered valid for total sulphur analysis.

## 6 ANNEX III - Standards

### Standards for the sampling plan

1. UNE-EN 14899:2007 - "Characterization of waste - Sampling of waste materials - Framework for the preparation and application of a sampling plan"
2. UNE-EN 15002:2015 - "Characterization of waste - Preparation of test portions from the laboratory sample" - Method preparation
3. UNE-CEN/TR 15310-1:2008 IN - "Characterization of waste - Sampling of waste materials - Part 1: Guidance on selection and application of criteria for sampling under various conditions"- Method evaluation, error control, etc...
4. UNE-CEN/TR 15310-2:2008 IN - "Characterization of waste - Sampling of waste materials - Part 2: Guidance on sampling techniques"
5. UNE-CEN/TR 15310-3:2008 IN - "Characterization of waste - Sampling of waste materials - Part 3: Guidance on procedures for sub-sampling in the field"
6. UNE-EN ISO 21645:2022 - "Recovered solid fuels. Sampling methods"

### Standards for the analysis procedures

7. ISO 17828:2015 - "Solid biofuels – Determination of bulk density"
8. UNE-EN-ISO 14780:2018. Solid biofuels. Sample preparation.
9. UNE-EN-ISO 21660 - 3:2021 - "Solid recovered fuels - Determination of moisture content using the oven dry method - Part 3: Moisture in general analysis sample (ISO 21660-3:2021)"
10. Particle size distribution study
  - UNE 7050:1997 - "Test sieves. Technical requirements and testing. Part 1. Test sieves of metal wire cloth"
  - UNE-EN 1015-1:1999 - "Methods of test for mortar for masonry - Part 1: Determination of particle size distribution (by sieve analysis)"
  - UNE-CEN/TR 15149-3:2015 IN - Solid biofuels - Determination of particle size distribution - Part 3: Rotary screen method
11. UNE-EN ISO 21656:2021 - Solid recovered fuels - Determination of ash content
12. UNE-EN 18123:2024 - Solid biofuels - Determination of volatile matter
13. ISO 16948:2015 - Solid biofuels - Determination of total content of carbon, hydrogen and nitrogen