



BSI Standards Publication

Collection, logistics & treatment requirements for end-of-life household appliances containing volatile fluorocarbons or volatile hydrocarbons

Part 2: Specification for de-pollution

National foreword

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Collection, logistics & treatment requirements for end-of-life household appliances containing volatile fluorocarbons or volatile hydrocarbons - Part 2: specification for de-pollution

Exigences de collecte, logistique et traitement pour la fin de vie des appareils domestiques contenant des fluorocarbures volatils ou des hydrocarbures volatils - Partie 2: Spécifications de dépollution

Anforderungen an die Sammlung, Logistik und Behandlung von Altgeräten aus dem Haushalt, die flüchtige Fluorkohlenwasserstoffe oder flüchtige Kohlenwasserstoffe enthalten - Teil 2: Spezifikation zur Schadstoffentfrachtung

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Foreword

This document (CLC/TS 50574-2:2014) has been prepared by CLC/TC 111X “Environment”.

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Introduction

This Technical Specification is intended to support EN 50574:2012 by providing further normative requirements for the assessment of de-pollution for treatment of end-of-life household appliances containing volatile fluorocarbons or volatile hydrocarbons.

Any characteristic numbers and target values within this Technical Specification are based on evidence gathered by technical experts over a time period of more than two years when performing test according to EN 50574:2012.

1 Scope

EN 50574:2012 gives the responsible take-back parties the task of defining target values. This Technical Specification provides applicable target values, characteristic numbers; sampling and analysis procedures, as well as monitoring and reporting requirements. Furthermore, this Technical Specification provides validation methodologies for tests and the daily business of the treatment plants as defined in EN 50574:2012.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 50574:2012, *Collection, logistics & treatment requirements for end-of-life household appliances containing volatile fluorocarbons or volatile hydrocarbons*

3 Terms and definitions

For the purposes of this document the terms and definitions given in EN 50574:2012 and the following apply.

3.1

characteristic numbers

values of parameters (except target values and limit values) used to determine VFC and VHC recovery performance for step 1 (e.g. q_{Mtot} , S_k), step 2 (e.g. f_{VFC} , f_{VHC}) and step 3, and values of parameters used to assess certain results of the plant performance audit (e.g. t_{max} in step 1 and $w_{i,VFC}$ and $w_{i,VHC}$ in step 2)

3.2

monitoring

system to acquire and store quantitative information about the material input and material output streams of the treatment facility (step 1, step 2, step 3) and related calculation of the recovery performance

Note 1 to entry: The scope of the required monitoring procedures is detailed in 5.5 in EN 50574:2012.

3.3

reporting

all information used to notify the results of the monitoring system

Note 1 to entry: The scope and content of the information to be reported is detailed in 5.6 in EN 50574:2012.

4 Target values

4.1 Step 1

A.2.7 of EN 50574:2012 define the target values for step 1 treatment.

4.2 Step 2: tv_{VFC} , tv_{VHC}

4.2.1 General

Clause 6 of EN 50574:2012 requires that target values for the recovery of VFCs and VHCs for Step 2 treatment (tv_{VFC} , tv_{VHC}) shall be defined.

The target values (tv_{VFC} and tv_{VHC}) represent the minimum masses of VFC and VHC to be recovered per kilogram of VFC-PU or VHC-PU foam respectively. The target value tv_{VFC} is calculated either from M_{VFC} , which is the "potentially recoverable" mass of VFC based on the VFC-PU foam in the plant's PU input stream, or from D_{VFC} , which is the mass of VFC blowing agent determined based on the plant's PU output stream. The

target value tv_{VHC} is defined analogously in those cases where there will be no on-site step 3 treatment after step 2 treatment.

4.2.2 Target values based on the quantity of potentially recoverable VFC and VHC blowing agent calculated from the plant's PU input stream

The total amount of potentially recoverable blowing agent (VFC and VHC) is calculated as: $M_{tot} = M_{VFC} + M_{VHC}$.

NOTE The formulas for calculating M_{VFC} and M_{VHC} are presented in Annex B of EN 50574:2012.

The target value tv_{VFC} is calculated using the formula: $0,9 \times M_{VFC} / PU_{VFC}$ (expressed as g VFC per kg PU_{VFC}).

The target value tv_{VHC} is calculated using the formula: $0,9 \times M_{VHC} / PU_{VHC}$ (expressed as g VHC per kg PU_{VHC}).

4.2.3 Target values based on the theoretical mass of VFC and VHC blowing agent calculated from the PU output fraction

The total theoretical quantity of blowing agent based on the PU output fraction is calculated as: $D_{tot} = D_{VFC} + D_{VHC}$

NOTE The formulas for calculating D_{VFC} and D_{VHC} are given in Annex B of EN 50574:2012.

The target value tv_{VFC} is calculated using the formula: $0,9 \times D_{VFC} / PU_{VFC}$ (expressed as g VFC per kg PU_{VFC}).

The target value tv_{VHC} is calculated using the formula: $0,9 \times D_{VHC} / PU_{VHC}$ (expressed as g VHC per kg PU_{VHC}).

5 Characteristic numbers

5.1 Step 1

5.1.1 Maximal ratio of VFC (according to rating plate) to mass of mixture of VFC and oil sucked off: t_{max}

According to A.2.6 c) in EN 50574:2012, a value (t_{max}) for the parameter t shall be specified, above which an individual appliance undergoing step 1 treatment shall be deemed to be defective. The parameter t is computed as the ratio of the potentially recoverable quantity of VFC to the effective reduction in mass (S_k) resulting from the vacuum extraction of the refrigerant and oil mix from the refrigerating system. A threshold value of $t_{max} = 0,57$ shall be used in case of completely emptied compressors.

NOTE If the step 1 process does not completely remove the refrigerants and oil, then the t value is not applicable and has no plausible relation with t_{max} .

In addition to the evaluation based on t_{max} , the quantity of VFC and oil removed by vacuum extraction shall be assessed. A completely emptied refrigerating system shall be deemed as defective if $S_k < 0,24$ kg.

5.1.2 Ratio of total quantity of VFC and oil recovered by vacuum extraction to the reduction in the appliance mass after vacuum extraction has been completed: q_{Mtot}

According to A.2.6 a) in EN 50574:2012, a mass balance analysis shall be carried out by calculating the ratio (q_{Mtot}) of the sum of the total mass of VFC recovered and the total mass of oil recovered ($outR + outL$) to the total mass reduction (S) of all appliances after vacuum extraction has been completed. This ratio q_{Mtot} is a measure of the total system performance in terms of the overall quantity of oil and refrigerant recovered.

A value of $q_{Mtot} > 0,95$ and $< 1,05$ is deemed plausible.

5.1.3 Number of oil containing refrigerating system: N

According to A.2.6 e) in EN 50574:2012, the average quantity of oil per refrigerating system is calculated by dividing the total mass of oil recovered ($outL$) by the number of refrigerating systems containing oil (N). The result (V_L) represents the quantity of oil recovered per refrigerating system. The parameter N refers to the number of appliances for which $S_k > 0,050$ kg.

5.1.4 Residual oil in compressors

According to 5.2 (1) in EN 50574:2012 all oil shall be removed from the refrigerating system. All oil shall be considered removed if the average residual amount of oil in the opened compressors is less than 10 g.

If the average residual amount of oil in the opened compressors according to A.2.6 of EN 50574:2012 exceeds the limit of 10 g then the residual amount of other compressors from the test shall be determined.

NOTE In order to get a representative figure one could measure the total amount of residual oil left in all the compressors.

5.2 Step 2

5.2.1 Masses of PU for the individual appliance categories VFC 1–3 (i) and VHC 1–3 (i): $w_{i,VFC}$ and $w_{i,VHC}$

According to Clause 6 in EN 50574:2012, the mass of PU originally present in the individual appliance categories (w_1, w_2, w_3) shall be defined. The masses of PU insulating foam in VFC-containing and VHC-containing appliances in the categories 1–3 vary from country to country or relevant playing field area.

Characteristic numbers for the individual areas are as follows:

- The values for $w_{i,VFC}$ and $w_{i,VHC}$ for the Northern European countries are:
 - $wN_{i,VFC}$: cat 1: 3,66 kg; cat 2: 4,88 kg; cat 3: 6,10 kg;
 - $wN_{i,VHC}$: cat 1: 4,36 kg; cat 2: 5,81 kg; cat 3: 7,26 kg;
- The values for $w_{i,VFC}$ and $w_{i,VHC}$ for the Mid European countries are:
 - $wM_{i,VFC}$: cat 1: 3,14 kg; cat 2: 4,18 kg; cat 3: 5,23 kg;
 - $wM_{i,VHC}$: cat 1: 4,36 kg; cat 2: 5,81 kg; cat 3: 7,26 kg;
- The values for $w_{i,VFC}$ and $w_{i,VHC}$ for the Southern and Eastern European countries are:
 - $wSE_{i,VFC}$: cat 1: 2,45 kg; cat 2: 3,26 kg; cat 3: 4,08 kg;
 - $wSE_{i,VHC}$: cat 1: 4,36 kg; cat 2: 5,81 kg; cat 3: 7,26 kg;

$w_{i,VHC}$ data are based on a limited number of tests and may be subject to change. If there are clear and comprehensible evidences, that in a single country numbers for $w_{i,VHC}$ are higher or lower than the average numbers in the geographical area these new numbers shall be used in the respective country.

In case of an appliance without doors the amount of foam of the appliance shall be reduced by 20 %.

NOTE 1 Countries are listed in Annex A.

NOTE 2 Categories are defined in 3.2 of the EN 50574:2012.

NOTE 3 A successful VFC test combined with tests on base of Annex B of EN 50574:2012 with solely VHC appliances of one category are deemed to be clear and comprehensible evidences.

5.2.2 Concentration of VFC in VFC-PU (input) and Concentration of VHC in VHC-PU (input): f_{VFC} / f_{VHC}

According to Clause 6 in EN 50574:2012, the concentration of VFC and VHC blowing agents present in the PU foam contained in appliances shall be defined. The following values are applicable in all European countries:

- $f_{VFC} = 8,5 \%$
- $f_{VHC} = 3,8 \%$

These values are needed in order to calculate the amounts of VFC and VHC that can potentially be expected to be recovered during plant performance tests and the target values derived from them.

6 Monitoring system and regular reporting (5.2 to 5.6 / Annex C of EN 50574:2012)

6.1 General

According to 5.5 of EN 50574:2012, the operator shall monitor the input and output information of step 1, step 2 and step 3 treatment of the plant.

The monitoring system shall demonstrate the recovery performance of the daily business. The role of the monitoring system is to provide evidence of the regular reporting, as well as an internal control system for the operator to measure and evaluate the daily recovery performance.

6.2 Information to be monitored

The monitoring information required may be found in Clause 5 and Annex C of the EN 50574:2012 and within other clauses of the standard.

6.3 Targets for day-to-day business

6.3.1 General

The monitoring data collected day-to-day shall be evaluated at least on a weekly basis.

The evaluation shall include the recovery rates and analysis of the performance of the plant in order to assess the achievements against the following:

If the operator doesn't reach these targets of the day-to-day business, failure will be justified and the operator shall activate corrective actions and monitor the related results.

6.3.2 Step 2

The mass of refrigerant recovered per intact refrigerating system in day-to-day business shall be not lower than 90 % of the expected mass.

The mass of oil recovered per oil containing refrigerating system in day-to-day business shall be not lower than 90 % by weight of the expected mass of oil.

NOTE 1 The expected mass of VFC is anticipated to be 126 g of refrigerant per intact refrigerating system. The expected mass of VHC is anticipated to be 54 g of refrigerant per refrigerating system.

The expected mass of oil is anticipated to be 240 g oil per VFC containing compressor. VHC containing compressors might have a different mass of oil. If there is clear and comprehensive evidence that the

expected mass of oil from VHC appliances is lower or higher, the respective value shall be used for further calculations.

NOTE 2 In order to set a fixed border for intact or defective refrigerating systems, a system pressure of less than 0,2 bar is assumed to indicate a defective appliance.

6.3.3 Step 2 and Step 3

The mass of blowing agent recovered (or destroyed) per kilogram PU_{VFC} and PU_{VHC} in day-to-day business shall be $> 90\%$ by weight of the expected mass of VFC and VHC respectively (tv_{VFC} and tv_{VHC} shall be calculated on the basis of 4.2.2 of this Technical Specification).

The analysis of 5.6.1, Annex A and Annex B of EN 50574:2012 has to be performed following the sampling and analytical methods settled in chapter 8, to give evidence of reaching the limit values for step 1 and step 2.

6.4 Reporting

According to 5.6 (1) in EN 50574:2012, the results of the monitoring system shall be structured on a monthly basis and reported annually. The results shall be built starting, as a minimum, from the weekly performance's results.

The table in Annex B shows the structure of the reporting system for the Step 1 and Step 2 activities.

7 Compliance evaluation

The results of the performance tests according to Annex A and Annex B of EN 50574:2012 are to be validated using the formulae in A.3 and B.3. In addition to that the references in Annex C shall be used to validate the test results.

8 Sampling and analytical methods

8.1 General

For concretisation of sampling and analysing requirements from EN 50574:2012, 5.2 to 5.7, Annex A, Annex B the following provisions shall apply.

8.2 Sampling methods

8.2.1 General

Sampling has to be carried out during the performance tests and for reporting according to EN 50574:2012, 5.6 and Annex A and B.

NOTE EN 14899:2005 (Characterization of waste, Sampling of waste materials, Framework for the preparation and application of a sampling plan) contains basic sampling requirements.

The sampling and subsequent sample preparation shall be carried out by someone who has the requisite competence. When conducting sampling, appropriate personal protective equipment shall be used, in accordance with the facility requirements and current regulations.

Two similar samples have to be taken in each case of sampling: One sample is for laboratory analysis and the other one has to be kept as backup for minimum 1 year.

Sample bottles and other sample vessels should be clearly labelled. The label shall contain at least the name of the treatment facility (or a code), WEEE stream, location of sampling, name of fraction and date of sampling.

Bottles and sample vessels shall be selected according to the requirements of the contents concerning pressure and gas tightness.

Because of the high-volatility of VFCs and VHCs, all samples containing these substances shall be sent as quickly as possible to the laboratory and store them between 1°C and 4°C whenever possible.

The control sample has to be stored at temperatures within a range from 1°C to 4°C.

Photos of samples shall be taken and recorded.

8.2.2 Sampling of oil as base for the determination of residual VFC content

This clause refers to 5.2 (3), 5.6 (1), 5.7 (2) and A.2.6 (f) and A.2.7 of EN 50574:2012.

To ensure that the sample is representative for the complete circle of step 1 production (excluding oil coming from other sources), the sample shall be taken from oil that has undergone full treatment.

The sampled oil shall be stored in a gastight vessel. The sample shall be taken at mid-height. Sampling vessel shall be filled up to top.

NOTE The oil from the step 1 test on base of Annex A of EN 50574:2012, taken out of the tank to establish the mass balance might not have been fully degassed yet.

Volume and mass of the sample should fit with the requirements of the laboratory.

8.2.3 Sampling of refrigerant as base for the determination of the chemical composition

This clause refers to 5.2 (5), 5.5 (3), 5.6 (1) and Annex A of EN 50574:2012. As the sampling process is a part of the analysing, it is described in 8.2.2.

NOTE Mobile gas analysers for refrigerants for sampling and analysing in one step can be used.

8.2.4 Sampling of PU fraction as base for the determination of residual VFC, VHC and foreign matters content

8.2.4.1 General

This clause refers to 5.3 (5), 5.6 (1), 5.7 (4) (5) and B.2.5.2 (a) (b) of EN 50574:2012.

8.2.4.2 Number and size of the sample

Table 1

PU foam form	Minimum vol. of single sample	Minimum vol. of mixed sample	Amount of laboratory sample
Briquette	1 unit (Min. 250 ml)	10 units	3 units
Pellets	250 ml	2 500 ml	250 ml
Powder	250 ml	2 500 ml	250 ml

PU foam has to be sampled from a minimum of 10 single samples. These 10 single samples have to be mixed in order to form a mixed sample, which then has to be reduced at the size of the sample to be analysed.

8.2.4.3 Sampling technique

Depending on the design of the process, one of the following techniques shall be used:

a) Method 1: Sampling from a falling stream

In this method, samples are to be taken regularly at the outlet of a continuous mechanical treatment process

The sampling period shall correspond to the processing time of 1000 appliances of input material. To define the sampling interval, the required processing time of the input material has to be divided by 10. The first sample has to be taken after half of the time of the first calculated interval. The rest of the samples have to be taken at the calculated sampling intervals.

If PU foam is in pellet form or powder samples shall be taken directly from the output flow of the fraction over the whole cross section of the flow profile by applying the calculated sampling interval.

If PU foam is in briquette form, one briquette per sampling interval shall be sampled.

b) Method 2: Sampling from the container

If it is not possible to sample the PU directly from a falling stream because PU is stored in closed containers, 10 single samples shall be taken directly in storage containers after the test. To determine the number of samples in each container, divide 10 by the number of containers containing the fraction corresponding to the input quantity.

The location of where the samples shall be taken in one container is indicated in the picture below, depending on the calculated number of samples to take in each container.

On the following pictures, the brown square stands for a top view of one container of the fraction to sample, and the red points indicate where, in this top view of the container, shall be taken the samples, depending on the calculated number of samples to take per container. Samples are always taken on the top layer of the container.

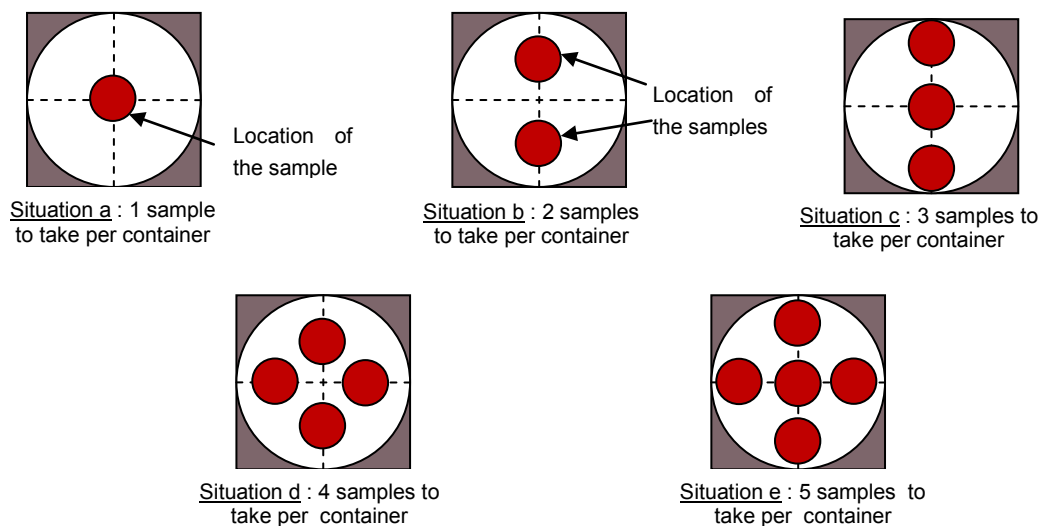


Figure 1 - Sampling from the container

8.2.4.4 Mixed sample reduction

The mixed sample has to be reduced to comply with the laboratory sample size.

For pellets and powder samples, after having applied the conical heap method, flatten the cone to form a flat heap of uniform thickness and diameter.

Using a shovel inserted vertically into the material, divide the flat heap into four along two diagonals, intersecting at right angles. Discard one pair of opposite quarters and shovel the remainder into a stockpile. Repeat this action until the volume of remaining subsample is equal to the desired size.

For briquette samples, randomly select 3 briquettes.

From the same reduction operation, form 2 samples:

- The first one for the analysis in laboratory
- The second one shall be retained for a minimum of 1 year in case further analysis is required.

8.2.5 Sampling of metallic and plastics fractions as base for the determination of remaining PU parts

8.2.5.1 General

This clause refers to 5.3 (4), 5.6 (1), 5.7 (5 and 6) and B.2.5.2 (e) of EN 50574:2012.

The sampling period shall correspond to the processing time of 1000 appliances of input material. This period varies depending on the recycling process.

8.2.5.2 Number and size of the sample

Metallic and plastics fractions are sampled with a minimum of 5 single samples. These 5 single samples are mixed in order to form a mixed sample, which is then reduced at the size of the sample to be analysed.

Table 2

Min. vol. of single sample	Min. vol. of mixed sample	Amount of sample to be analysed
1 l	5 l	1 l

8.2.5.3 Sampling technique

To define the sampling interval, the required processing time of the input materials has to be divided by 5. Sampling shall be carried out at regular intervals. The first sample has to be done after half of the time of the first sampling interval. This applies for each single fraction (ferrous-metal, NON - ferrous metals, plastics or mix of them).

Depending on the design of the process, one of the techniques as described in 8.2.4.3 shall be used.

8.2.5.4 Mixed sample reduction

The same method as in 8.2.4.4 is applied.

8.2.6 Sampling of blowing agents as base for the determination of chemical composition

This clause refers to the following clauses of EN 50574: 5.3:2012 (6), 5.5 (6), 5.6 (1), B.2.5.1 and B.2.5.2.

The water volume in the vessel has to be determined before the samples are taken.

NOTE Measurement systems in the vessel or implemented in the plant itself are deemed to be appropriate if there is a clear indication for the definite water content (in kg or litre) in the gross mass of blowing agents.

The gross mass of the blowing agents (mass of pure blowing agents *plus* water *plus* other substances) have to be measured and recorded. The mass of water shall be deducted from the gross mass of the blowing agents. The result of this calculation is the output mass of blowing agents (out_{BA}) as base for the further calculation according to B.2.5.1 (c) and B.2.5.2 (f) of EN 50574:2012.

Representative samples shall be taken in the blowing agent phase of the output liquids considering the high-volatility of VFCs and VHCs and to avoid blowing agent losses.

Two samples of 5 ml to 10 ml shall be taken. The sampled blowing agent shall be stored in a gastight vessel. Sample vessels shall be filled up to the top and closed immediately after filling in order to avoid any loss of the blowing agent.

8.3 Analytical methods

All the analyses shall follow the basic principle of double independent determination based on the sample and double determination of the analysis itself.

Analyses have to consider the blank test.

Because of the high-volatility of VFCs, the laboratory shall be advised that all samples containing these substances shall be stored between 1°C and 4°C.

The laboratory shall determine the volume of R12, R114 and R134a in the oil. The residual VFC in the oil shall be expressed as an R12 equivalent, calculated using molar mass. Annex D lists examples of proven procedures for analytical methods. Laboratories shall use the methods described there. If laboratories use other methods than as described in Annex D, they shall report the procedure in detail and supply the results of a validation process. The laboratory shall implement the standard EN 15002:2006 and follow the different step described in the decision tree.

Annex A (normative)

List of countries in the geographical areas

The values for $w_{i,VFC}$ and $w_{i,VHC}$ for different geographical areas are fixed in 5.2.1 of this Technical Specification.

NOTE 1 The countries are classified in the following geographical area, in the following paragraph are indicated with an ** the countries from which the information have been taken to build up the $w_{i,VFC}$ targets.

Northern European countries are:

- Denmark;
- Estonia *;
- Finland;
- Iceland;
- Ireland;
- Latvia;
- Lithuania;
- Norway *;
- Sweden *;
- United Kingdom.

Mid European countries are:

- Austria *;
- Belgium *;
- France *;
- Germany *;
- Luxembourg *;
- Netherlands *;
- Switzerland *.

Southern and Eastern European Countries are:

- Bulgaria;
- Croatia;

- Cyprus;
- Czech Republic *;
- Former Yugoslav Republic of Macedonia;
- Greece *;
- Hungary;
- Italy *;
- Malta;
- Poland;
- Portugal *;
- Romania;
- Slovakia;
- Slovenia;
- Spain *;
- Turkey;

NOTE 2 The composition of macro geographical (continental) regions, geographical sub-regions, and selected economic and other groupings is taken by the United Nation statistic division.

Annex B (informative)

Structure of the monitoring and reporting system for the Step 1 and Step 2 activities

The following table gives an example of the structure of a monitoring and reporting system for the Step 1 activities.

Table B.1

				Refrigerators						
				Refrigerant: VFC		Refrigerant: VHC		Refrigerant:		
Month	Week	Day	N. of cooling circuits treated	N. of intact circuits	N. of defective	N. of intact circuits	N. of defective	N. of intact circuits	N. of defective	...
1	---	---								---
2	---	---								---
3	---	---								---
4	---	---								---
...	---	---								---
total			0	0	0	0	0	0	0	

	... (professional appliances, air conditioners,...)								
...	...	Amount of VFC/VHC recovered	Amount of VFC/VHC expected	Amount of oil recovered	Amount of oil expected	Alarm	Corrective actions and results	Analysis of oil	Analysis of refrigerants
---	---	-	-	-	-				
---	---	-	-	-	-		---		
---	---	-	-	-	-				
---	---	-	-	-	-				
---	---	-	-	-	-				

The following table gives an example of the structure of a monitoring and reporting system for the Step 2 activities.

Table B.2

Month	Week	Day	N. of cabinets treated	Category 1		Category 2		Cat
				N. of VFC appliances	N. of VHC appliances	N. of VFC appliances	N. of VHC appliances	N. of VFC appliances	N. of VHC appliances	..
1
2
3
4
...
total	0				

..	... (professional appliances, air conditioners,...)	Amount of VFC & VHC recovered	Amount of VFC & VHC expected (see 6.2.1 and 6.2.2)	Alarm	Corrective actions and results	Analysis of PU	Analysis of blowing agents
..		
..	...						
..	...						
..	...						
..	...						
..	...						

Annex C (normative)

Compliance evaluation

C.1 Introduction

This annex is to describe the validation of the collected figures during the performance tests according to EN 50574:2012, Annex A (Methodology for running performance tests for step 1 treatment) and Annex B (Methodology for running performance tests for step 2 treatment).

The described procedure shall support supervised performance tests in getting plausible results and conclusions (or to prevent making non-plausible conclusions).

C.2 Calculations for performance test step 1

C.2.1 General

In principle, any calculations and formulae referring to the step 1 test shall be based on A.3. In addition to that the following considerations shall be taken in account.

C.2.2 Defective refrigeration systems

The number of defective refrigeration systems shall be calculated as defined in A.2.6 c) of EN 50574:2012 and this Technical Specification, 5.1.1 using the characteristic numbers t_{\max} and S_k .

C.2.3 Compressed air in refrigeration vessels

The volume, pressure difference and temperature from the used vessels to collect the VFC have to be considered in order to calculate the mass of the compressed air as part of the total mass. The result will be subtracted from the recovered total mass.

The total mass balance rate q_{Mtot} and the rate of VFC recovery (based on plates) q_r shall be calculated on base of the consideration regarding the compressed air.

C.2.4 Plausibility checks referring to the step 1 test

The following aspects should be checked:

Is $q_{Mtot} > 0,95$ and $< 1,05$?

If q_{Mtot} is not in this range the mass balance is not plausible the evaluator shall look for explanations and refer to the findings in an evaluation report.

Is the 80 % quantile of all t values between 0,54 and 0,57 ?

If no, the evaluator shall look for explanations and refer to the findings in an evaluation report.

Is the "total mass lost" ($S - (\text{outR} + \text{outL})$) higher than the "mass of VFC lost" ($R_i - \text{outR}$) ?

If no, the result is not plausible. Evaluators shall look for explanations and refer to the findings in an evaluation report.

Is the ratio of recovered VFC per refrigerating system (V_R) to the mean amount of totally filled in VFC reasonable considering the number of defectives ?

If no, evaluators shall look for explanations and refer to the findings in an evaluation report.

For example, evaluators should look if the refrigerating systems have been emptied completely or should consider the residual amount of oil in the refrigerating system.

VFC lost should be part of the total mass lost. Assuming the VFC lost as being recovered, the mass balance should be not higher than 100 %. VFC losses being higher than the total losses are not plausible.

C.3 Calculations for performance test step 2

C.3.1 General

In principle, any calculations and formulae referring to the step 2 test shall be based on B.3. In addition to that the following considerations shall be taken in account.

NOTE In the case of Step 3, the results from the taken salt/solution analysis can be taken into account, together with the weight of the total salts/solutions, for determining the mass of the recovered VFC.

C.3.2 Density of the blowing agents

For the output consideration, the results from the analysis report from the taken sample of blowing agent shall be compared with the theoretical density for the mixture based on its components. If the deviation of the two values is higher than 10 % the evaluator shall look for explanations and refer to the findings in an evaluation report.

C.3.3 Determination of water in the vessel

In any case there shall be a qualitative analysis of water in the blowing agent vessels. If present, the part of water in the vessel shall be subject of volumetric determination, the measured volume shall be recalculated to mass water and the mass water shall subtracted from the mass of the gross blowing agent (including water and substances other than blowing agents in the vessel).

The part of water shall be determined before the liquids are transferred to the tank, except there is strong evidence of having no water part at all.

C.3.4 Plausibility checks

Evaluators shall check the correct data areas, e.g. when calculating the input-output mass balance. Each plant can have its specific situation for considering output material as part of the input (e.g. water from insulation or from the air).

Further on, the following aspects shall be checked:

Are the results from input and output consideration in a similar range?

Is the calculated density of the Blowing Agent in a similar range as the analysis result? If no, check the used densities of single components. Check whether both values are fitting with the behaviour of the organic layer of blowing agent against a water layer (if known, otherwise the evaluator should check the back-up sample)

In case of combustion calculation: are the ratios of resulting R11 and R12 for all single analysis in a plausible and similar range? If not, explain.

The evaluator shall check if the installation has been in the same condition before and after the performance test (e.g. number of regenerations, number of defrosting steps, input mix the days before).

NOTE 1 In case of a high ratio of plastics and other materials in a PU output fraction, consequently the amount of Plastics output can be reduced. A high amount of PU and Plastics at the same time seems to be not plausible.

NOTE 2 Adding water to the sample will increase the volume of the water phase.

NOTE 3 Raw gas streams to combustion units consist of approximately of 95 % of R11 and 5 % of R12. The determination of R11 and R12 from the resulting salts NaCl and NaF is only valid if no relevant amounts of other VFCs have been present. Relevant deviations to the expected ratio can be an indicator for other sources of VHC than PU foams.

Annex D **(informative)**

Analytical procedures

D.1 Determination of Residual VFC Content in Oil – Example 1

D.1.1 Extraction

At the analysis laboratory, a sample of the oil has to be weighed (0,5 to 2,0 g). In a gastight 60 ml vessel, VFCs are extracted from oil in 50 ml of acetone.

After the extraction, 1 ml of Acetone is diluted in 9 ml of water in a 20 ml headspace vial. Two different test portions have to be prepared with two concentrations of doped water.

D.1.2 Analysis

The vial has to be heated at 80°C for at least 30 min. Then, a sample of the gas in the headspace has to be injected into a GC-MS instrument.

D.1.3 Chromatography Settings

The chromatographic analyses have to be recorded under the follow instructions:

- Chromatographic column: capillary column specific for the determination of volatile organic pollutants, particularly for chlorofluorinated pollutants;
- Advised thermal profile: plateau at 50°C for 10 min followed by a 10°C/min thermal ramp until 280°C (at the end of the analysis, a cleaning process at a high temperature depending on the column model is needed);
- Gas-transport flux and pressure: gas-transport flux and pressure shall guarantee good resolution and separation of the chromatographic peaks; gas-transport flux and pressure shall avoid the overlap chromatographic peaks on other peaks;
- Calibration: the calibration is obtained by the internal standard method, i.e. a response factor is determined by a calibration curve determined with at least five known-concentration samples of each of the analysed compounds and the internal standard (the internal standard should be a chemical compound similar to the analysed substances, its chromatographic peak should not overlap the chromatographic peaks or those of other compounds of interest in the mixture);

D.2 Determination of Residual VFC Content in Oil – Example 2

D.2.1 Analysis

Before the laboratory analysis, the oil samples need an ice/water cooling bath for 1 h.

A sample of oil is weighted (1-2 g) in a gastight 40ml vessel and CFCs are extracted/solved in 10 ml of Diacetone Alcohol (4-hydroxy-4-methyl-pentan-2-one, CAS. 123-42-2) for a night at room temperature, without stirring.

After the extraction/solubilization, 1 ml of Diacetone alcohol is diluted in 9 ml of water in a 20 ml headspace vial. A sample of the gas in the headspace is injected into a GC-MS instrument. The GC-MS analysis of the

head-space sample is suitable for the determination of the compounds at low concentration, such as Freon-11 and traces of other CFCs. Gas-chromatography (GC) equipped with a flame ionization detector (FID) and double column is preferred for compounds at high concentrations (Freon-12).

D.2.2 Analysis Repetition

The analyses are repeated twice for each sample and the analysis report shall show the two sets of non-duplicate values, avoiding the average.

D.2.3 Chromatographic Settings

The chromatographic analyses are recorded under the follow instructions:

- chromatographic column: capillary column specific for the determination of volatile organic pollutants, particularly for chlorofluorinated pollutants;
- advised thermal profile: plateau at 35°C for 3 min followed by a 7°C/min thermal ramp until 110°C (at the end of the analysis, a cleaning process at a high temperature depending on the column model is needed);
- gas-transport flux and pressure: gas-transport flux and pressure shall guarantee good resolution and separation (at least 0,8 min) of the chromatographic peaks corresponding to Freon-11 and Freon-12; gas-transport flux and pressure shall avoid the overlap of Freon-11 and Freon-12 chromatographic peaks on other peaks;
- calibration: the calibration is obtained by the internal standard method, i.e. a response factor is determined by a calibration curve determined with three known-concentration samples of each of three compounds Freon-11, Freon-12 and the internal standard (the internal standard should be a chemical compound similar to Freon-11 and Freon-12, its chromatographic peak should not overlap the Freon-11 and Freon-12 chromatographic peaks or those of other compounds of interest in the mixture);

The chromatograms should be available for two years after the analysis. In chromatograms, the peaks of Freon-11, Freon-12 and internal standard (eventually unknown) should be clearly identified.

D.3 Determination of Chemical Composition of the Output Refrigerants (VFCs/VHCs) from the Step 1

For the determination of the chemical composition of refrigerants mobile analysers are deemed to be appropriate tools. These devices are able to analyse R12, R134a, R22, hydrocarbons and air.

NOTE Such devices provide a fast, easy, and accurate means to determine refrigerant type and purity in refrigeration systems and refrigerant storage cylinders. These instruments utilize a combination of infrared and chemical fuel cell technologies to determine purity and composition of refrigerants.

D.4 Determination of residual VFC and VHC in PU fraction – Example 1

D.4.1 Preparation for Analysis in the Laboratory

Phase separation:	not necessary
Drying:	not necessary and not applicable for the analyse but the water content should be determined on a distinct subsample
Homogenization and sub sampling:	mechanical homogenization using cryogenic grinding in order to obtain a test portion of 3 to 6 g as a subsample.

NOTE 1 Drying would cause VFC losses.

NOTE 2 Homogenization without using nitrogen would cause VFC losses.

D.4.2 Analysis

At the analysis laboratory, a sample of the polyurethane matrix is weighted (3 g to 6 g). In an gastight 60 ml vessel, VFCs are extracted from Polyurethane matrix in 50 ml of ultrapure Methanol for a night at 20 °C room temperature without stirring.

After the extraction, 1 ml of methanol is diluted in 9 ml of water in a 20 ml headspace vial. A sample of the gas in the headspace is injected into a GC-MS instrument. Gas-chromatography (GC) equipped with a flame ionization detector (FID) and double column is preferred for compounds at high concentrations (Freon-11).

D.4.3 Analysis Repetition

The analysis repetition on several specimens from the same plant is needed in order to obtain a precise evaluation of the residual VFC content in the Polyurethane matrix.

Particularly, 3 specimens are needed for briquette plants and sampling during the laboratory analysis shall be done from the centre of the briquette; 3 specimens are needed also for pellet plant and for powder plants sampling is obtained by quartering.

D.4.4 Chromatographic Settings

The chromatographic analyses are recorded under the follow instructions:

Chromatographic column: capillary column specific for the determination of volatile organic pollutants, particularly for chlorofluorinated pollutants;

Advised thermal profile: plateau at 35 °C for 3 min followed by a 7°C/min thermal ramp until 110 °C (at the end of the analysis, a cleaning process at a high temperature depending on the column model is needed).

Gas-transport flux and pressure: gas-transport flux and pressure shall guarantee good resolution and separation (at least 0,8 min) of the chromatographic peaks corresponding to Freon-11 and Freon-12; gas-transport flux and pressure shall avoid the overlap of Freon-11 and Freon-12 chromatographic peaks on other peaks.

The calibration is obtained by the internal standard method, i.e. a response factor is determined by a calibration curve determined with three known-concentration samples of each of three compounds Freon-11, Freon-12 and the internal standard (the internal standard should be a chemical compound similar to Freon-11 and Freon-12, its chromatographic peak should not overlap the Freon-11 and Freon-12 chromatographic peaks or those of other compounds of interest in the mixture).

The chromatograms shall be available for two years after the analysis. In chromatograms, the peaks of Freon-11, Freon-12 and internal standard (eventually unknown) should be clearly identified.

D.5 Determination of Residual VFC in PU Fraction – Example 2

D.5.1 Analysis

At the analysis laboratory, a sample of the polyurethane matrix is weighted (3 g to 6 g). In a gastight 60 ml vessel, CFCs are extracted from polyurethane matrix in 50 ml of ultrapure Methanol for a night at room temperature without stirring. After the extraction, 1 ml of Methanol is diluted in 9 ml of water in a 20 ml headspace vial. A sample of the gas in the headspace is injected into a GC-MS instrument. The GC-MS analysis of the head-space sample is suitable for the determination of the compounds at low concentration, such as Freon-12 and traces of other CFCs. Gas-chromatography (GC) equipped with a flame ionization detector (FID) and double column is preferred for compounds at high concentrations (Freon-11).

D.5.2 Analysis Repetition

The analysis repetition on several specimens from the same plant is needed in order to obtain a precise evaluation of the residual CFC content in the Polyurethane matrix.

Particularly, 3 specimens are needed for briquette plants and sampling during the laboratory analysis shall be done from the centre of the briquette; 3 specimens are needed also for pellet plant and for powder plants sampling is obtained by quartering.

D.5.3 Chromatographic Settings

The chromatographic analyses are recorded under the follow instructions:

- chromatographic column: capillary column specific for the determination of volatile organic pollutants, particularly for chlorofluorinated pollutants;
- advised thermal profile: plateau at 35°C for 3 min followed by a 7°C/min thermal ramp until 110°C (at the end of the analysis, a cleaning process at a high temperature depending on the column model is needed);
- gas-transport flux and pressure: gas-transport flux and pressure shall guarantee good resolution and separation (at least 0,8 min) of the chromatographic peaks corresponding to Freon-11 and Freon-12; gas-transport flux and pressure shall avoid the overlap of Freon-11 and Freon-12 chromatographic peaks on other peaks;
- calibration: the calibration is obtained by the internal standard method, i.e. a response factor is determined by a calibration curve determined with three known-concentration samples of each of three compounds Freon-11, Freon-12 and the internal standard (the internal standard should be a chemical compound similar to Freon-11 and Freon-12, its chromatographic peak should not overlap the Freon-11 and Freon-12 chromatographic peaks or those of other compounds of interest in the mixture);

The chromatograms should be available for two years after the analysis. In chromatograms, the peaks of Freon-11, Freon-12 and internal standard (eventually unknown) should be clearly identified.

D.6 Determination of foreign matters content in PU fraction

D.6.1 Thermogravimetric Analysis- Example 1

Preparation of samples

Phase separation:	not necessary
Drying:	applicable
Homogenization and sub-sampling:	mechanical homogenization using cryogenic grinding to a powder in order to obtain a test portion as a subsample.

NOTE Homogenization of samples is absolutely necessary in order to get a representative subsample for the subsequent analysis.

The analysis is carried out in two steps, a semiquantitative analysis by IR spectrometry and a quantitative analysis by thermogravimetric analysis (TGA).

Step 1: Spectroscopy IR (FT-IR)

The first step should aim to determine the type of impurities which could be present in the PU fraction.

Spectra of Solids - The KBr Disc Technique

Before pressing, the PU sample has to be mixed with the KBr powder at a sample concentration level of 0,1 % to 2 %. The sample has to be grounded to a fine powder in order to reduce scattering losses and absorption band distortions.

Preparation of the mixture: 1/2 mm to 1 mm layer of the mixture should be transferred from the mortar to the die and the disc should be pressed. The disk should then be placed in the disk holder and the spectrum should be obtained.

Step 2: Thermogravimetric method (TGA)

TGA is a thermal analysis technique which consists of the measurement of the variation in weight of a sample as a function of temperature.

The analysis has to be conducted under the following conditions of the TGA device:

- The sample should be heated at 10 K/min to 1000 °C under an atmosphere of oxygen.
- Measurement in the thermal analyser.

D.6.2 Selective extraction Method- Example 2

Sample preparation

Phase separation:	not necessary
Drying:	applicable
Homogenization and sub sampling:	mechanical homogenization using mortar and pestle or cryogenic grinding to a powder in order to obtain a test portion of 50 g as a subsample.

NOTE Homogenization of samples is absolutely necessary in order to get a representative subsample for the subsequent analysis

Analysis

The analysis is carried out in two steps, an automated solid liquid extraction of non PU-polymers by pressurized liquid extraction (PLE) followed by a solid liquid extraction in a semiquantitative analysis by IR spectrometry and a quantitative analysis by thermogravimetric analysis (TGA).

Step 1: Pressurized liquid extraction of non PU polymers

The first step should aim to determine extract and weigh non PU-polymers from the PU fraction, which may include (PE, PP, PS, ABS, PMMA).

3 g to 8 g of the homogenized PU sample (exact input weight is determined: dm(IN)) are mixed with the ninefold amount of pre extracted and dried sea-sand and filled into in 22 ml cartridges. Extractions were performed with two solvents, dichloromethane and toluene:

DCM: 3 static cycles for 30 min at 80 °C

Toluene: 3 static cycles for 30 min at 80 °C

Extracts are combined in vessel vials and dried under a stream of Nitrogen, whereas the samples are placed in a heated alumina tray. The weight of the dry matter extracted with both, Dichloromethane and Toluene, is determined. (dm(DCM) and dm(TOL))

Step 2: Depolymerize and extract PU from the sample

The extraction residue of step 1 is filled into a 500 ml flask and depolymerized/extracted for 1 h at 230 °C in glycol using a heating mantle and a reflux condenser fixed on top of the flask. The extract is filtered with a paper filter using a suction filter aggregate. The filter residue is extracted a second time with 80 g Glycol in the same flask and again filtered. Both filter papers are washed with Ethanol and dried. The dry weight of the filter residues is determined (dm FR).

Calculation of PU content:

$$\text{PU (\%)} = 1 - (\text{dm(DCM)} + \text{dm(TOL)} + \text{dm(FR)}) / \text{dm(IN)}$$

D.7 Visual analysis method for residual PU on metal and plastics fractions

The analysis and verification has to be conducted (complete or partly) on operator's site.

The following operations have to be conducted for each of the samples:

- the sample to be separated has to be weighed;
- the sample has to be separated by the two categories foam and main fraction (metals or plastics);
- only the separated PU category should be weighed;

Remaining PU with light structure in metallic fractions and strongly compressed PU, with glue or foil structure and also wet PU foam should be sorted out. These parts should not be taken into account as remaining PU.

NOTE The expected foam weights based on input calculation can be used to correct for PU coming from VHC appliances.

In order to calculate the remaining PU parts on metals and plastics the following formula should apply:

j_i : PU impurity in the fraction i .

$M(\text{PU}_i)$: Mass of residual PU of the fraction i .

M_i : Mass of the sample to be separated.

i : fraction of Fe, AlCu, Plastic.

$$j_{\text{Fe}} = M_{(\text{PU}_{\text{Fe}})} / M_{\text{Fe}}$$

$$j_{\text{AlCu}} = M_{(\text{PU}_{\text{AlCu}})} / M_{\text{AlCu}}$$

$$j_{\text{PL}} = M_{(\text{PU}_{\text{PL}})} / M_{\text{PL}}$$

D.8 Determination of chemical composition of the output blowing agents (VFCs/VHCs) from the step 2

Direct determination of R11, R12, R134a, R141b and Cyclo-Pentane, N-Pentane as well as Iso-Pentane using the following method:

The relative amount of VFCs and VHCs in the blowing agent phase (exclusive water content) should be determined using gas chromatography or infrared spectroscopy. The types of VFCs and VHCs identified and their respective percentage mass fractions should be documented.

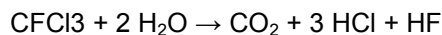
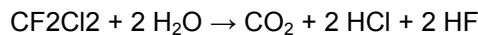
D.9 Determination of chemical composition of the collected acids or their neutralization products from the combustion in step 3

D.9.1 General

Several alkaline solid salts as neutralizing agent for acid gases can be used by the CFC disposal facilities:

- Sodium Carbonate, Na_2CO_3 ;
- Sodium Bicarbonate (also called sodium hydrogen carbonate), NaHCO_3 ;
- Sodium Hydroxide, NaOH .

During the disposal, the alkaline solid salts react with the acid gases, produced by combustion. The reactions of Freon-12 (Dichlorodifluoromethane), CF_2Cl_2 , and Freon-11 (Trichlorofluoromethane), CFCl_3 , are as the follow, respectively:



The produced gases are CO_2 , HCl e HF . Because of their acid properties, HCl and HF react with the alkaline salts forming NaCl and NaF , respectively. As the disposal process proceeds, the amount of NaCl and NaF increases. Thus, the composition of the gases is determined indirectly by the analysis of chlorine and fluorine content in a salt specimen. In addition, the sodium content determination and the pH measurement are used as complementary analyses for a higher precision.

D.9.2 Chlorine analysis

The quantitative analysis of chlorine in the solid specimens is based on the EN 15309 procedure, which requires the energy dispersive X-ray fluorescence (EDXRF) spectroscopy or the wavelength dispersive X-ray fluorescence (WDXRF) spectroscopy with calibration in a matrix. Additionally, the chlorine content is determined by ion-exchange chromatography. The chromatographic data are compared with the XRF data. If the difference between chromatographic and XRF data are lower than $\pm 10\%$, the results are accepted as valid; on the contrary the analyses have to be repeated.

D.9.3 Fluorine analysis

The quantitative analysis of fluorine is not possible directly in the solid specimens. Thus, a weighted sample is solved in a known-volume of water, accurately diluted and quantitatively analysed by a calibrated ion-exchange chromatography.

D.9.4 Sodium analysis

The quantitative analysis of sodium is complementary to the Chlorine and Fluorine analyses and it is measured by inductively coupled plasma atomic emission spectroscopy (AES-ICP).

D.9.5 pH measurement

For an efficient neutralization of the acid gases, the Alkaline solid salt should maintain its Alkaline properties meanwhile it is in the dry scrubber. The pH measurement of a solution obtained solving in water a sample of Alkaline solid salt allows the easy and effective evaluation of the quality of the Alkaline salt. A weighted sample of Alkaline salt is solved in a known volume of water and the pH is measured by a pH-meter with precision of ± 0.01 pH unit.

D.9.6 Analysis repetition

The analyses are repeated twice for each sample and the analysis report shall show the two sets of non-duplicate values, avoiding the average.

D.10 Determination of Chemical composition of the alkaline solution from the step 3

D.10.1 General

As described previously, Alkaline solid salts as neutralizing agent for acid gases are used by some CFC disposal facilities (see Alkaline Solid Salt Analysis for details). The composition of the outgoing gases can be indirectly determined also by the quantitative analyses of Chlorine and Fluorine amounts in an Alkaline solution obtained by solving a weighted sample of solid salt in a known volume of water. Before the analyses, the solution shall be homogenized by a suitable stirring.

Wet scrubbers use directly soda, i.e. a highly concentrated aqueous solution of sodium hydroxide, NaOH. In these facilities, an aqueous solution with NaCl and NaF is obtained. It is important to check the presence of a solid residue, due to low-soluble NaF (water solubility at 25°C: 42 g/l), in the soda recovery tank. Thus, the Alkaline solution needs an homogenization by a strong stirring before the sampling from the recovery tank. The solid residue is formed after a few of time and, consequently, the solution needs a homogenization by a 20 min strong stirring before the analysis.

D.10.2 Chlorine analysis

The content of Chlorine in the Alkaline solution is determined by X-ray fluorescence spectroscopy (XRF), using a specific polymeric sample-holder for liquids. In the sample holder, 4,00 ml of Alkaline solution are typically introduced. The quantitative analysis is obtained by means of certified calibration curves or prepared with standard-samples in the same range of conditions.

D.10.3 Fluorine and Sodium analysis

As described in D.9.3 and D.9.4.

D.10.4 pH measurement

For an efficient neutralization of the acid gases, the soda solution should maintain its alkaline properties meanwhile it is in the wet scrubber.

The direct pH measurement by a pH-meter with precision of $\pm 0,01$ pH unit allows the easy and effective evaluation of the quality of the soda solution, the indirect determination of the reacted soda and the estimation of the sodium content in the solution.

D.10.5 Analysis repetition

As described in D.9.6.

D.11 Determination of VFC and VHC mass flow in raw gas and clean gas for step 3

D.11.1 General

The determination of the amount of the blowing agent recovered in step 3 installation can be supported considering the concentration of VFC and VHC in the raw gas and clean gas.

D.11.2 Blowing agent in the raw gas

The calculation to determine the amount of blowing agent in the raw gas can be done daily using the data continuously recorded.

The operator should calculate the average concentration of VFC and VHC in an hour basis (in g/m^3 per hour).

The operator should measure the average air flow (in m^3/hour).

The blowing agent is calculated with the following formulas:

- VFC in input = average hourly concentration of VFC x average air flow
- VHC in input = average hourly concentration of VHC x average air flow

Attention should be made to the possible interactions in the measure between VFC and VHC.

D.11.3 Blowing agent in the clean gas

The calculation to determine the amount of blowing agent in the clean gas can be done daily using the data continuously recorded.

The operator should calculate the average concentration of VFC and VHC in an hour basis (in mg/m^3 per hour).

The operator should measure the average air flow (in m^3/hour).

The blowing agent is calculated with the following formulas:

- VFC in output = average hourly concentration of VFC x average air flow / 1000
- VHC in output = average hourly concentration of VHC x average air flow / 1000

Attention should be made to the possible interactions in the measure between VFC and VHC.

D.11.4 Blowing agent recovered

The blowing agent recovered is calculated with the following formulas:

- VFC recovered = VFC in input – VFC in output
- VHC recovered = VHC in input – VHC in output

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