TEXTE 120/2024

Annex

HP 14 Classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'

Sampling protocols

by: Ralf Ketelhut Stoffstromdesign, Neumünster

publisher: German Environment Agency



TEXTE 120/2024

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Annex

HP 14 Classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'

Sampling protocols

by

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On behalf of the German Environment Agency

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Table of content

List	List of figures			
List	of tables	7		
1	Flue-gas dust (10 09 09*) from iron and steel casting: batch 1	9		
2	Flue-gas dust (10 09 09*) from iron and steel casting: batch 2	. 14		
3	Flue-gas dust (10 09 10) from iron and steel casting: plant A	. 19		
4	Flue-gas dust (10 09 10) from iron and steel casting: plant B	. 24		
5	Soil and stones (17 05 03*): excavated geogenic material	. 29		
6	Soil and stones (17 05 03*): material from the side verges of a federal road	. 34		
7	Soil and stones (17 05 04): material from the side verges of a secondary road	. 39		
8	Fluff-light fraction and dust (19 10 04): material (sieved to <10 mm) from plant A, batch 1	. 44		
9	Fluff-light fraction and dust (19 10 04): material (sieved to <10 mm) from plant A, batch 2	. 49		
10	Fluff-light fraction and dust (19 10 04): material (sieved to <10 mm) from plant B	. 55		

List of figures

Figure 1:	Photographic documentation for flue-gas dust (10 09 09*) from iron and steel casting: batch 1 (aged material, storage
	neriod >4 weeks) 11
Figure 2.	Photographic documentation for flue-gas dust $(10.09.09^{\circ})$
inguie 2.	from iron and steel casting: batch 1 (aged material, storage
	period >4 weeks)
Figure 3.	Photographic documentation for flue-gas dust (10.09.09*)
inguie of	from iron and steel casting: batch 2 (fresh material, storage
	period <4 weeks)
Figure 4:	Photographic documentation for flue-gas dust (10 09 09*)
0	from iron and steel casting: batch 2 (fresh material, storage
	period <4 weeks)
Figure 5:	Photographic documentation for flue-gas dust (10 09 10) from
-	iron and steel casting: plant A21
Figure 6:	Photographic documentation for flue-gas dust (10 09 10) from
	iron and steel casting: plant A23
Figure 7:	Photographic documentation for flue-gas dust (10 09 10) from
	iron and steel casting: plant B26
Figure 8:	Photographic documentation for flue-gas dust (10 09 10) from
	iron and steel casting: plants B28
Figure 9:	Photographic documentation for excavated geogenic material
	(17 05 03)31
Figure 10:	Sieve curve for excavated geogenic material (17 05 03*)32
Figure 11:	Photographic documentation for excavated geogenic material
	(17 05 03)
Figure 12:	Photographic documentation for material from the side verges
	of a federal road (17 05 03*)36
Figure 13:	Sieve curve for material from the side verges of a federal road
	(17 05 03*)
Figure 14:	Photographic documentation for material from the side verges
	of a federal road (17 05 03)38
Figure 15:	Photographic documentation for material from the side verges
	of a secondary road (17 05 04)41
Figure 16:	Sieve curve for material from the side verges of a secondary
	road (17 05 04)42
Figure 17:	Photographic documentation for material from the side verges
	of a secondary road (17 05 04)43
Figure 18:	Photographic documentation for fluff-light fraction from plant
	A, batch 1 (19 10 04)46
Figure 19:	Sieve curve for fluff-light fraction (19 10 04) from plant A,
	batch 1

Figure 20:	Photographic documentation for fluff-light fraction (19 10 04)		
	from plant A, batch 148		
Figure 21:	Photographic documentation for fluff-light fraction from plant		
	A, batch 2 (19 10 04)51		
Figure 22:	Sieve curve for fluff-light fraction from plant A, batch 2 (19 10		
	04)53		
Figure 23:	Photographic documentation for fluff-light fraction from plant		
	A, batch 2 (19 10 04)54		
Figure 24:	Photographic documentation for fluff-light fraction from plant		
	B (19 10 04)57		
Figure 25:	Sieve curve for fluff-light fraction from plant B (19 10 04)59		
Figure 26:	Photographic documentation for fluff-light fraction from plant		
	B (19 10 04)60		

List of tables

Table 1:	Sampling9
Table 2:	Sample pretreatment and sample preparation according to DIN
	19747 (2009a)11
Table 3:	Sampling14
Table 4:	Sample pretreatment and sample preparation according to DIN
	19747 (2009a)16
Table 5:	Sampling19
Table 6:	Sample pretreatment and sample preparation according to DIN
	19747 (2009a)22
Table 7:	Sampling24
Table 8:	Sample pretreatment and sample preparation according to DIN
	19747 (2009a)27
Table 9:	Sampling29
Table 10:	Sample pretreatment and sample preparation according to DIN
	19747 (2009a)31
Table 11:	Sampling34
Table 12:	Sample pretreatment and sample preparation according to DIN
	19747 (2009a)36
Table 13:	Sampling
Table 14:	Sample pretreatment and sample preparation according to DIN
	19747 (2009a)41
Table 15:	Sampling44
Table 16:	Sample pretreatment and sample preparation according to DIN
	19747 (2009a)46
Table 17:	Sampling49
Table 18:	Sample pretreatment and sample preparation according to DIN
	19747 (2009a)52

Table 19:	Sampling55	5
Table 20:	Sample pretreatment and sample preparation according to DIN	J
	19747 (2009a)58	3

1 Flue-gas dust (10 09 09*) from iron and steel casting: batch 1

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018)¹, requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

	Waste code	10 09 09* (Flue-gas dust containing hazardous substances)		
	Identification	Flue-gas dust from iron and steel casting: batch 1		
	Sponsor	German Environment Agency/ECT Oekotoxikologie GmbH		
	Project	HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'		
	Overall objective	Collection of a probabilistic sample and preparation for ecotoxicological testing		
	Waste owner	Is known to the sampler		
	Project management	ECT Oekotoxikologie GmbH		
	Sampler	Ralf Ketelhut Stoffstromdesign		
Sampling date, time Weather conditions Relevant background for the sampled waste		14 July 2022, 10:00 a.m. to 10:20 a.m.		
		Sunny, 19°C, 54% relative humidity		
		Flue-gas dusts are mainly produced during the cleaning of furnace exhaust gases, process exhaust air from the waste sand treatment, casting part removal and post-processing and, to a small extent, exhaust air from the casting process as well as mould construction. They are generally removed from exhaust air with fabric filters. Dusts from the cleaning of furnace exhaust contain predominantly iron oxide, oxides of other metals used, ash of fuel, impurities adhering to the input material and unburnt aggregates. ² Here: Kupolo oven dust, approx. 85% grey cast iron, approx. 15% lamellar casting. Generally, huge fluctuations in chemical analysis.		
	Sampling point	Foundry in western Germany		
	Material description	Very fine-grained, grey		
	Variability	No specific spatial heterogeneity to be expected, large number of small influences, particulate heterogeneity rather low.		

Table 1: Sampling

 $^{^{\}rm 1}$ All references are included in the reference list (section 8) of the main report.

² See information portal waste assessment, waste sheet 1009 iron and steel foundries (<u>https://www.abfallbewertung.org</u>)

Specific information	The intended separation into grey cast iron and lamellar cast iron could not be realised. Therefore, two batches were sampled: older material (storage period >4 weeks) and fresh material (storage period <4 weeks). Here: batch 1 (aged material, storage period >4 weeks)			
Batch size	A total of 7 big bags of ap	prox. 1 m ³ or 5 Mg (estim	ated)	
Mass flow	Not known			
Bulk density	$\rho_{\rm B} = 0.7 \text{ kg/dm}^3$ (estimated	d)		
Raw density	$\rho_R = 2.8 \text{ kg/dm}^3$ (estimated	d)		
Particle dimension	d ₉₅ = 2 mm (estimated)			
Particle type	Grainy			
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D	The European working group on the characterization of waste (CEN/TC 292) states that an estimate of the minimum sample size can be obtained according to the equation indicated in Figure 13 of the report. The calculation was performed using the following values: M _{SAM} : Minimum sample size [kg], here: M _{SAM} = 0.003 kg d ₉₅ : Nominal screen size of particles [cm], here 0.2 cm p _p : Particle density [g/cm ³], here 2.8 g/cm ³ g: Correction factor for particle size, here g = 0.25, since d ₉₅ /d ₀₅ >4 p: Fraction of particles with a certain characteristic, if not known, then $p = 10\%^{a^3}$ CV: Coefficient of variation, if not known, then CV = 10%			
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples (CS):	Number of laboratory samples (LS):	
	8	2	2	
Minimum sample size	d ₉₅ :	Volume of RS:	Volume of LS:	
according to LAGA PN 98	≤2 mm	0.5 dm³	1 dm³	
	No sample size reduction of the composite samples to laboratory samples was made to avoid increasing the variance of the samples. The sample mass was increased to obtain sufficient material.			
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{CS} = (number of RS • V _{RS}) Number of RS: 17	M _{CS} = V _{CS} • ρ _B	
	0.0002 dm³	0.004 dm³	0.003 kg	
Type of sampling according to CEN/TR 15310-1	Systematic sampling. 14 individual samples were taken with a 50 mm sampling pipe from 7 big bags. Real sample: approx. 7.3 dm ³ , 5.1 kg			
Storage	None			
Transport at, start	None			
Sample preparation site	Foundry in western Germany			

³ Fraction (proportion) of particles with a certain characteristic (the characteristic to be determined). Generally, p is not known in advance. In addition, p is different for each active component. Therefore, p is usually assumed to be 10%.

Figure 1: Photographic documentation for flue-gas dust (10 09 09*) from iron and steel casting: batch 1 (aged material, storage period >4 weeks)



Big bags with old material, recognizable by deposits and standing water on the big bags Source: own illustration, Ralf Ketelhut, Stoffstromdesign

-		
Beginning		14 July 2022, 10:20 a.m.
	End	14 July 2022, 10:40 a.m.
	Place of treatment	Foundry in western Germany
Weather conditions		Sunny, 20°C
	Sample preparation	Creation of a sieve line using original material with hand screening on round-hole screens. Screen cut <4 mm round-hole sieve represents the laboratory sample.
	Sieve curve for original material and approximate distribution	The particle size distribution is speculative. The total sample can be almost completely rubbed through the 4 mm sieve.

Table 2:	Sample pretreatment	and sample preparatior	n according to DIN 1	9747 (2009a)
----------	---------------------	------------------------	----------------------	--------------

Data on the sieve curve	Weight [g]	Percentage by weight [%]	Cumulative percentage by weight [%]
<4 mm	5,125.0	99.99%	99.99%
<5 mm	0,8	0.01%	100.00%
Sample pretreatment	Small, possibly metallic o	contaminant particles are	contained.
Mass balance of pre-	Factions	Weight [g]	Share [%]
treatment	Input	5,126.8	100.0%
	Sample <4 mm	5,125.0	99.65%
	Oversized particles	0.8	0.015%
	Losses	1.0	0.020%
Storage	None		
Transport, start	14 July 2022, 12:15 p.m.		
Transport container	PE bag in cooled 60-liter PP barrel in a car		
Transport by	Ralf Ketelhut		
Handover	15 July 2022, 8:30 a.m.		
Recipient	ECT Oekotoxikologie GmbH		

Figure 2: Photographic documentation for flue-gas dust (10 09 09*) from iron and steel casting: batch 1 (aged material, storage period >4 weeks)



Above: impurities >4 mm, bottom: material <4 mm after screening Source: own illustration, Ralf Ketelhut, Stoffstromdesign

2 Flue-gas dust (10 09 09*) from iron and steel casting: batch 2

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018), requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

	Waste code	10 09 09* (Flue-gas dust containing hazardous substances)		
	Identification	Flue-gas dust (10 09 09*) from iron and steel casting: batch 2		
	Sponsor	German Environment Agency/ECT Oekotoxikologie GmbH		
	Project	HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'		
	Overall objective	Collection of a probabilistic sample and preparation for ecotoxicological testing		
	Waste owner	Is known to the sampler		
	Project management	ECT Oekotoxikologie GmbH		
	Sampler	Ralf Ketelhut Stoffstromdesign		
	Sampling date, time	14 July 2022, 10:45 a.m. to 11:15 a.m.		
	Weather conditions	Sunny, 19°C, 54% relative humidity		
Relevant background for the sampled waste		Flue-gas dusts are mainly produced during cleaning of furnace exhaust gases, process exhaust air from waste sand treatment, casting part removal and post-processing and, to a small extent, exhaust air from the casting process as well as mould construction. They are generally removed from exhaust air with fabric filters. Dusts from the cleaning of furnace exhaust contain predominantly iron oxide, oxides of the other metals used, ash of fuel, impurities adhering to the input material and unburnt aggregates. ⁴ Here: Kupolo oven dust, approx. 85% grey cast iron, approx. 15% lamellar cast iron. Generally, huge fluctuations in chemical analysis.		
	Sampling point	Foundry in western Germany.		
	Material description	Very fine-grained, grey		
	Variability	No specific spatial heterogeneity to be expected, large number of small influences, particulate heterogeneity rather low.		
	Specific information	The intended separation into grey cast iron and lamellar cast iron could not be realised. Therefore, two batches were sampled: older material		

Table 3: Sampling

⁴ See information portal waste assessment, waste sheet 1009 iron and steel foundries (<u>https://www.abfallbewertung.org</u>)

	(storage period >4 weeks) and fresh material (storage period <4 weeks). Here: batch 2 (fresh material, storage period <4 weeks)		
Batch size	A total of 25 big bags of approx. 1 m ³ or 17.5 Mg (estimated)		
Mass flow	Not known		
Bulk density	$\rho_B = 0.7 \text{ kg/dm}^3$ (estimated	d)	
Raw density	ρ _R = 2.8 kg/dm ³ (estimated	d)	
Particle dimension	d ₉₅ = 2 mm (estimated)		
Particle type	Grainy		
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D	The European working group on the characterization of waste (CEN/TC 292) states that an estimate of the minimum sample size can be obtained according to the equation indicated in Figure 13 of the report. The calculation was performed using the following values: M _{SAM} : Minimum sample size [kg], here: M _{SAM} = 0.003 kg d ₉₅ : Nominal screen size of particles [cm], here 0.2 cm ρ_p : Particle density [g/cm ³], here 2.8 g/cm ³ g: Correction factor for particle size, here g = 0.25, since d ₉₅ /d ₀₅ >4 p: Fraction of particles with a certain characteristic, if not known, then $p = 10\%^3$ CV: Coefficient of variation, if not known, then CV = 10%		
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples:	Number of laboratory samples (LS):
	8	2	2
Minimum sample size	d95:	Volume of RS:	Volume of LS:
according to LAGA PN 98	≤2 mm	0.5 dm³	1 dm³
	No sample size reduction of the composite samples to laboratory samples was made to avoid increasing the variance of the samples. The sample mass was increased to obtain sufficient material.		
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{cs} = (number of RS • V _{RS}) Number of RS: 17	$M_{CS} = V_{CS} \bullet \rho_B$
	0.0002 dm³	0.0035 dm³	0.0024 kg
Type of sampling according to CEN/TR 15310-1	 Systematic sampling. 16 individual samples with a 50 mm sampling pipe distributed over 16 b bags. Weight of the individual sample approx. 0.410 kg Real sample: approx. 9.3 dm³, 6.5 kg 		
Storage	None		
Transport at, start	None		
Sample preparation site	Foundry in western Germany		

Figure 3: Photographic documentation for flue-gas dust (10 09 09*) from iron and steel casting: batch 2 (fresh material, storage period <4 weeks)



Big bags with fresh material Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Beginning	14 July 2022, 11:20 a.m.		
End	14 July 2022, 11:45 a.m.		
Place of treatment	Foundry in western Gerr	nany	
Weather conditions	Sunny, 20°C		
Sample preparation	Creation of a sieve line u round-hole screens. Scre laboratory sample.	ising original material with een cut <4 mm round-hole	n hand screening on e sieve represents the
Sieve curve original material and approximate distribution	The particle size distribu almost completely rubbo	tion is speculative. The to ed through the 4 mm sieve	tal sample can be e.
Data on the sieve curve	Weight [g]	Percentage by weight [%]	Cumulative percentage by weight [%]
<4 mm	6,527.0	99.99%	99.99%
<5 mm	0.8	0.01%	100.00%
Sample pretreatment	The sample contains sm	all, possibly metallic conta	aminant particles.

Table 4: Sample pretreatment and sample preparation according to DIN 19747 (2009a)

Mass balance of pre-	Factions	Weight [g]	Share [%]
treatment	Input	6,529.8	100.0%
	Sample <4 mm	6,527.0	99.97%
	Oversized particles	0.81	0.013%
	Losses	1.1	0.017%
Storage	ge None		
Transport	14 July 2022, from 12:15 p.m.		
Transport container	PE bag in cooled 60-liter PP barrel in a car		
Transport by	Ralf Ketelhut		
Handover	15 July 2022, 8:30 a.m.		
Recipients	ECT Oekotoxikologie GmbH		

Figure 4: Photographic documentation for flue-gas dust (10 09 09*) from iron and steel casting: batch 2 (fresh material, storage period <4 weeks)



Above: impurities >4 mm, bottom: material <4 mm after screening Source: own illustration, Ralf Ketelhut, Stoffstromdesign

3 Flue-gas dust (10 09 10) from iron and steel casting: plant A

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018), requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

Waste code	10 09 10 (Flue-gas dust other than those mentioned in 10 09 09
Identification	Flue-gas dust from iron and steel casting: plant A
Sponsor	German Environment Agency/ECT Oekotoxikologie GmbH
Project	HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'
Overall objective	Collection of a probabilistic sample and preparation for ecotoxicological testing
Waste owner	Is known to the sampler
Project management	ECT Oekotoxikologie GmbH
Sampler	Ralf Ketelhut Stoffstromdesign
Sampling date, time	14 June 2022, 10:00 a.m. to 10:20 a.m.
Weather conditions	Sunny, 17°C, 60% relative humidity
Relevant background for the sampled waste	Flue-gas dusts are mainly produced during the cleaning of furnace exhaust gases, process exhaust air from waste sand treatment, casting part removal and post-processing and, to a small extent, exhaust air from casting process as well as mould construction. They are generally removed from exhaust air with fabric filters. Dusts from cleaning of furnace exhaust contain predominantly iron oxide, oxides of other metals used, ash of fuel, impurities adhering to the input material and unburnt aggregates. ⁵
Sampling point	Reserve area of the Cröbern Central Landfill in Saxony. Deferred, opened big bag (approx. 1 m ³) from one delivery.
Material description	Very fine-grained
Variability	No specific spatial heterogeneity to be expected, large number of small influences, particulate heterogeneity rather low.
Specific information	Very small batch size (deferral of one big bag)
Batch size	Piles of 1 m ³ or 1.3 Mg (estimated)

Table 5: Sampling

⁵ See information portal waste assessment, waste sheet 1009 iron and steel foundries (<u>https://www.abfallbewertung.org</u>)

Mass flow	Not known		
Bulk density	$\rho_{\rm B}$ = 1.3 kg/dm ³ (estimated	d)	
Raw density	$\rho_R = 2.8 \text{ kg/dm}^3$ (estimated	d)	
Particle dimension	d95 = 2 mm (estimated)		
Particle type	Grainy		
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D	The European working gro 292) states that an estima according to the equation calculation was performed M _{SAM} : Minimum sample d ₉₅ : Nominal screen size ρ_p : Particle density [g/cr g: Correction factor for p: Fraction of particles $p = 10\%^3$ CV: Coefficient of variation	bup on the characterization the of the minimum samp indicated in Figure 13 of d using the following value size [kg], here: $M_{SAM} = 0$ of particles [cm], here 0.2 m ³], here 2.8 g/cm ³ particle size, here g = 0.2 with a certain characterist	on of waste (CEN/TC le size can be obtained the report. The les: .003 kg 2 cm 25, since d ₉₅ /d ₀₅ >4 stic, if not known, then 2 = 10%
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples:	Number of laboratory samples (LS):
	8	2	2
Minimum sample size	d ₉₅ :	Volume of RS:	Volume of LS:
according to LAGA PN 98	≤2 mm	0.5 dm³	1 dm³
	No sample size reduction was made to avoid increas mass was increased to obt	of the composite sample sing the variance of the s tain sufficient material.	s to laboratory samples amples. The sample
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{cs} = (number of RS • V _{RS}) Number of RS: 9	$M_{CS} = V_{CS} \bullet \rho_B$
	0.0002 dm³	0.002 dm³	0.003 kg
Type of sampling according to CEN/TR 15310-1	Simple random sampling. 40 Individual samples with over the total sample. Real sa	n a 50 mm sampling pipe mple: approx. 3.9 dm ³ , 5	were taken, distributed .0 kg.
Storage	Transfer to PE bag and the closure.	en cooled 60-liter PP barr	el with clamping ring
Transport at, start	14 June 2022, 10:20 a.m.		
Transport by	Ralf Ketelhut		
Sample preparation site	Westsächsische Entsorgur	ngs- und Verwertungsges	ellschaft mbH, Cröbern
Arrival at the sample preparation site	14 June 2022, 10:40 a.m.		

Figure 5: Photographic documentation for flue-gas dust (10 09 10) from iron and steel casting: plant A



Above: deferred big bag with marking, below: detail Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Т	Table 6: Sample pretreatment and sample preparation according to DIN 19747 (2009a)			
	Beginning	14 June 2022, 10:30 a.m		
	End	14 June 2022, 11:15 a.m		
	Place of treatment	Westsächsische Entsorg	ungs- und Verwertungsge	sellschaft mbH, Cröbern
	Weather conditions	Sunny, 20°C		
	Sample preparation	Creation of a sieve line u round-hole screens. Scre laboratory sample.	ising original material witl een cut <4 mm round-hole	n hand screening on e sieve represents the
	Sieve curve original material and approximate distribution	The particle size distribu completely rubbed throu	tion is speculative. The to ugh the 4 mm sieve.	tal sample was
	Data on the sieve curve	Weight [g]	Percentage by weight [%]	Cumulative percentage by weight [%]
	<4 mm	5,000.0	100.0%	100.0%
	Sample pretreatment	No interfering material o	contained.	
	Mass balance of pre-	Factions	Weight [g]	Share [%]
	treatment	Input	5,000.0	100.0%
		Sample <4 mm	4,983.0	99.7%
		Oversized particles	0.0	0.0%
		Losses	17.0	0.3%
	Storage	None		
	Transport	14 June 2022, from 12:1	5 p.m.	
	Transport container	PE bag in cooled 60-liter	PP barrel in a car	
	Transport by	Ralf Ketelhut		
	Handover	15 June 2022, 8:30 a.m.		
	Recipients	ECT Oekotoxikologie Gm	ıbН	

Figure 6: Photographic documentation for flue-gas dust (10 09 10) from iron and steel casting: plant A



Material <4 mm after screening Source: own illustration, Ralf Ketelhut, Stoffstromdesign

4 Flue-gas dust (10 09 10) from iron and steel casting: plant B

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018), requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

Waste code	10 09 10 (Flue-gas dust other than those mentioned in 10 09 09)
Identification	Flue-gas dust from iron and steel casting: plant B
Sponsor	German Environment Agency/ECT Oekotoxikologie GmbH
Project	HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'
Overall objective	Collection of a probabilistic sample and preparation for ecotoxicological testing
Waste owner	Is known to the sampler
Project management	ECT Oekotoxikologie GmbH
Sampler	Ralf Ketelhut Stoffstromdesign
Sampling date, time	19 October 2022, 1:10 to 2:00 p.m.
Weather conditions	Clouded, 6°C, 80% relative humidity
Relevant background for the sampled waste	Flue-gas dusts are mainly produced during cleaning of furnace exhaust gases, process exhaust air from waste sand treatment, casting part removal and post-processing and, to a small extent, exhaust air from the casting process as well as mould construction. They are generally removed from exhaust air with fabric filters. Dusts from the cleaning of furnace exhaust contain predominantly iron oxide, oxides of other metals used, ash of fuel, impurities adhering to the input material and unburnt aggregates. ⁶
Sampling point	Reserve area of the Cröbern Central Landfill in Saxony. Deferred batch from a delivery of 21 big bags.
Material description	Very fine, dark, slightly greasy
Variability	No specific spatial heterogeneity to be expected, large number of small influences, particulate heterogeneity rather low.
Specific information	None
Batch size	A total of 21 big bags of 1 m ³ or 32.5 Mg (estimated)

Table 7: Sampling

⁶ See information portal waste assessment, waste sheet 1009 iron and steel foundries (<u>https://www.abfallbewertung.org</u>)

Mass flow	Not known		
Bulk density	$\rho_B = 1.3 \text{ kg/dm}^3$ (estimated	d)	
Raw density	$\rho_R = 2.8 \text{ kg/dm}^3$ (estimated	d)	
Particle dimension	d ₉₅ = 2 mm (estimated)		
Particle type	Grainy		
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D	The European working gro 292) states that an estima according to the equation calculation was performed M _{SAM} : Minimum sample d ₉₅ : Nominal screen size ρ_p : Particle density [g/cr g: Correction factor for p: Fraction of particles $p = 10\%^3$ CV: Coefficient of variation	bup on the characterization the of the minimum samp indicated in Figure 13 of d using the following value size [kg], here: $M_{SAM} = 0$ of particles [cm], here 0.2 m ³], here 2.8 g/cm ³ particle size, here g = 0.2 with a certain characteris	on of waste (CEN/TC le size can be obtained the report. The les: .003 kg 2 cm 25, since $d_{95}/d_{05} > 4$ stic, if not known, then T = 10%
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples:	Number of laboratory samples (LS):
	8	2	2
Minimum sample size	d ₉₅ :	Volume of RS:	Volume of LS:
according to LAGA PN 98	≤2 mm	0.5 dm³	1 dm³
	No sample size reduction of the composite samples to laboratory samples was made to avoid increasing the variance of the samples. The sample mass was increased to obtain sufficient material.		
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{cs} = (number of RS • V _{RS}) Number of RS: 9	$M_{CS} = V_{CS} \bullet \rho_B$
	0.0002 dm³	0.002 dm³	0.003 kg
Type of sampling according to CEN/TR 15310-1	Stratified random samplin pipe distributed over 16 o Total co	g. 16 individual samples f the 21 big bags. mposite sample approx.	with a 50 mm sampling 6.3 kg
Storage	Transfer to PE bag and the closure.	en cooled 60-liter PP barr	el with clamping ring
Transport at, start	19 October 2022, 1:45 p.n	n.	
Transport by	Ralf Ketelhut		
Sample preparation site	Westsächsische Entsorgur	ngs- und Verwertungsges	ellschaft mbH, Cröbern
Arrival at the sample preparation site	19 October 2022, 2:00 p.n	n.	

Figure 7: Photographic documentation for flue-gas dust (10 09 10) from iron and steel casting: plant B



Deferred big bags with marking, below: detail of a single sampling Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Т	Table 8: Sample pretreatment and sample preparation according to DIN 19747 (2009a)			
	Beginning	19 October 2022, 2:00 p	.m.	
	End	19 October 2022, 2:30 p	.m.	
	Place of treatment	Westsächsische Entsorg	ungs- und Verwertungsge	sellschaft mbH, Cröbern
	Weather conditions	Clouded, 7°C		
	Sample preparation	Creation of a sieve line u round-hole screens. Scre laboratory sample.	using original material witl een cut <4 mm round-hole	n hand screening on e sieve represents the
	Sieve curve original material and approximate distribution	The particle size distribu completely rubbed throu	tion is speculative. The to ugh the 4 mm sieve.	tal sample can be
	Data on the sieve curve	Weight [g]	Percentage by weight [%]	Cumulative percentage by weight [%]
	<4 mm	6,309.0	100.0%	100.0%
	Sample pretreatment	No interfering materials	contained.	
	Mass balance of pre-	Factions	Weight [g]	Share [%]
	treatment	Input	6,337.0	100.0%
		Sample <4 mm	6,309.0	99.6%
		Oversized particles	0.0	0.0%
		Losses	28.0	0.4%
	Storage	None		
	Transport	19 October 2022, from 4	l:15 p.m.	
	Transport container	PE bag in cooled 60-liter	PP barrel in a car	
	Transport by	Ralf Ketelhut		
	Handover	20 October 2022, 8:30 a	.m.	
	Recipients	ECT Oekotoxikologie Gm	ъbН	

Figure 8: Photographic documentation for flue-gas dust (10 09 10) from iron and steel casting: plants B



Material <4 mm after screening Source: own illustration, Ralf Ketelhut, Stoffstromdesign

5 Soil and stones (17 05 03*): excavated geogenic material

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018), requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

Waste code	17 05 03* (Soil and stones containing hazardous substances)
Identification	Excavated geogenic material
Sponsor	German Environment Agency/ECT Oekotoxikologie GmbH
Project	HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'
Overall objective	Collection of a probabilistic sample and preparation for ecotoxicological testing
Waste owner	Is known to the sampler
Project management	ECT Oekotoxikologie GmbH
Sampler	Ralf Ketelhut Stoffstromdesign
Sampling date, time	14 June 2022, 10:20 to 10:50 a.m.
Weather conditions	Sunny, 17°C, 60% relative humidity
Relevant background for the sampled waste	The excavated geogenic material originates from an open-cast lignite mine. The material has a pH in the acidic range. In the past, it was shown to contain increased levels of nickel. Since the pH of waste has proven to be a relevant parameter with regard to effects in bioassays, the geogenic material appeared of interest for the present study.
Sampling point	Reserve area of the Cröbern Central Landfill in Saxony. Between stockpiles of approx. 30 m ³ from a very large population.
Material description	Soil material without vegetation
Variability	No specific spatial heterogeneity to be expected, large number of small influences, particulate heterogeneity rather low
Specific information	Experience has shown that pH is between 2 and 3
Batch size	Pile of 30 m ³ or 36 Mg (estimated)
Mass flow	Not known
Bulk density	$\rho_B = 1.2 \text{ kg/dm}^3$ (estimated)
Raw density	$\rho_R = 1.8 \text{ kg/dm}^3$ (estimated)
Particle dimension	d95 = 20 mm (estimated)

Table 9: Sampling

Particle type	Grainy		
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D	The European working gro 292) states that an estima according to the equation calculation was performed M _{SAM} : Minimum sample d ₉₅ : Nominal screen size of ρ_p : Particle density [g/cm g: Correction factor for p: Fraction of particles of $p = 10\%^3$ CV: Coefficient of variation	bup on the characterization te of the minimum samp indicated in Figure 13 of d using the following value size [kg], here: $M_{SAM} = 1$ of particles [cm], here 2 c n ³], here 1.8 g/cm ³ particle size, here g = 0.2 with a certain characteris	on of waste (CEN/TC le size can be obtained the report. The es: .7 kg .5, since $d_{95}/d_{05} > 4$ tic, if not known, then T = 10%
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples:	Number of laboratory samples (LS):
	8	2	2
Minimum sample size	d ₉₅ :	Volume of RS:	Volume of LS:
according to LAGA PN 98	>2 to ≤20 mm	1 dm³	2 dm ³
	No sample size reduction was made to avoid increase mass was increased to obt	of the composite samples sing the variance of the sa tain sufficient material.	s to laboratory samples amples. The sample
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{cs} = (number of RS • V _{RS}) Number of RS: 7	$M_{CS} = V_{CS} \bullet \rho_B$
	0.22 dm³	1.4 dm³	1.7 kg
Type of sampling according to CEN/TR 15310-1	Restricted random samplin No wheel loader available accumulation process. 20 piercing tube. Re	ng (judgmental sampling . The pile indicates suffici Individual samples were al sample: 3.9 dm ³ , 4.7 kg	1). ent mixing during the taken with a 100 mm g
Storage	Transfer to PE bag and the closure.	en cooled 60-liter PP barr	el with clamping ring
Transport at, start	14 June 2022, 10:30 a.m.		
Transport by	Ralf Ketelhut		
Sample preparation site	Westsächsische Entsorgur	ngs- und Verwertungsges	ellschaft mbH, Cröbern
Arrival at the sample preparation site	14 June 2022, 10:40 a.m.		

Figure 9: Photographic documentation for excavated geogenic material (17 05 03)



Deferred heap with marking, right: detail Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Beginning	14 June 2022, 11:15 a.m	I.	
End	14 June 2022, 12:00 a.m	ı.	
Place of treatment	Westsächsische Entsorgungs- und Verwertungsgesellschaft mbH, Cröbern		
Weather conditions	Sunny, 20°C		
Sample preparation	Creation of a sieve line u round-hole screens. Scre laboratory sample. Over separated, as no jaw cru	using original material with een cut <4 mm round-hole rsized particles and interfe usher was available on site	h hand screening on e sieve represents the ering material were e.
Sieve curve original material and approximate distribution	The particle size of the s median is approx. 1.5 m	oil is approximately logno m. The d ₉₅ is approx. 4 mr	rmally distributed. The n (see also Figure 10).
Data on the sieve curve	Weight [g]	Percentage by weight [%]	Cumulative percentage by weight [%]
<4 mm	4.310,0	94.7%	94.7%
<5 mm	22,0	0.5%	95.2%
<10 mm	126,0	2.8%	98.0%
<15 mm	92,0	2.0%	100.0%
Sample pretreatment	It can be expected that s interfering materials cor	some drying of the materi ntained.	al will take place. No
Mass balance of pre-	Factions	Weight [g]	Share [%]
treatment	Input	4,700.0	100.0%
	Sample <4 mm	4,310.0	91.7%
	Oversized particles	240.0	5.1%
	Losses	150.0	3.2%
Storage	None		

Table 10: Sample pretreatment and sample preparation according to DIN 19747 (2009a)

Beginning	14 June 2022, 11:15 a.m.
Transport	14 June 2022, from 12:15 p.m.
Transport container	PE bag in cooled 60-liter PP barrel in a car
Transport by	Ralf Ketelhut
Handover	15 June 2022, 8:30 a.m.
Recipients	ECT Oekotoxikologie GmbH

Figure 10: Sieve curve for excavated geogenic material (17 05 03*)



Manual screening on round hole screens

Source: own illustration, Ralf Ketelhut, Stoffstromdesign





Top left: >15 mm; top right: >10 mm, center: >4/<5 mm, bottom: <4 mm Source: own illustration, Ralf Ketelhut, Stoffstromdesign

6 Soil and stones (17 05 03*): material from the side verges of a federal road

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018), requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

	Waste code	17 05 03* (Soil and stones containing hazardous substances)
	Identification	Material from the side verges of a federal road
Sponsor		German Environment Agency/ECT Oekotoxikologie GmbH
	Project	HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'
	Overall objective	Collection of a probabilistic sample and preparation for ecotoxicological testing
	Waste owner	Is known to the sampler
	Project management	ECT Oekotoxikologie GmbH
	Sampler	Ralf Ketelhut Stoffstromdesign
	Sampling date, time	19 October 2022, 7:20 a.m. to 7:35 a.m.
Weather conditions Relevant background for the sampled waste		Cloudy, 6°C, 80% relative humidity
		In the side verge area, impurities and deposits from vegetation residues and road dirt accumulate. Depending on the traffic volume, direct material input from traffic emissions as well as abrasion of tires, roadways and brakes are to be expected. In addition, road salt residues may be relevant. The side verge is periodically peeled off at a width of about 80 cm with a layer thickness of 3-10 cm approximately every 3-8 years. Here, the removal was carried out on both sides of a busy federal road to prepare for the use of an asphalt milling machine.
	Sampling point	Storage space for excavated side verge material in northern Germany. Intermediate stockpile of approx. 45 m ³ from a main unit of approximately 1,000 m ³ over a period of several months.
Material description		Older floor material with significant vegetation cover
	Variability	No specific spatial heterogeneity to be expected, large number of small influences, particulate heterogeneity rather low.
	Specific information	The material is classified as Z2 according to LAGA soil analysis. This means that the material may only be disposed of or installed to a limited extent. Reasons for classification according to LAGA and for classification as hazardous are not known.

Table 11: Sampling

Batch size	Pile of 45 m ³ or 40 Mg (estimated)		
Mass flow	None		
Bulk density	$\rho_B = 0.92 \text{ kg/dm}^3$ (estimated)		
Raw density	$\rho_R = 1.8 \text{ kg/dm}^3$ (estimated	d)	
Particle dimension	d95 = 20 mm (estimated)		
Particle type	Grainy		
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D	The European working group on the characterization of waste (CEN/TC 292) states that an estimate of the minimum sample size can be obtained according to the equation indicated in Figure 13 of the report. The calculation was performed using the following values: M_{SAM} : Minimum sample size [kg], here: $M_{SAM} = 1.7$ kg d95: Nominal screen size of particles [cm], here 2 cm ρ_p : Particle density [g/cm ³], here 1.8 g/cm ³ g: Correction factor for particle size, here g = 0.25, since d95/d05 >4 p: Fraction of particles with a certain characteristic, if not known, then $p = 10\%^3$ CV: Coefficient of variation, if not known, then CV = 10%		
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples:	Number of laboratory samples (LS):
	12	3	3
Minimum sample size	d95:	Volume of RS:	Volume of LS:
according to LAGA PN 98	>2 to ≤20 mm	1 dm³	2 dm³
	No sample size reduction of the composite samples to laboratory samples was made to avoid increasing the variance of the samples. The sample mass was increased to obtain sufficient material.		
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{cs} = (number of RS • V _{RS}) Number of RS: 9	M _{cs} = V _{cs} • ρ _B
	0.22 dm³	1.8 dm³	1.7 kg
Type of sampling according to CEN/TR 15310-1	Restricted random sampling (<i>judgmental sampling 1</i>). No wheel loader available. The pile indicates a sufficient mixing by accumulation. Since it is a guarded heap, superficial influence by erosion can be expected. Therefore, approximately 10 cm of surface material is taken and then 20 individual samples are taken with a 100 mm piercing tube. Real sample: approx. 15.4 dm ³ , 14.2 kg.		
Storage	Transfer to PE bag and then cooled 60-liter PP barrel with clamping ring closure.		
Transport at, start	19 October 2022, 7:40 a.m.		
Transport by	Ralf Ketelhut		
Sample preparation site	Westsächsische Entsorgungs- und Verwertungsgesellschaft mbH, Cröbern		
Arrival at the sample preparation site	19 October 2022, 2:00 p.m.		

Figure 12: Photographic documentation for material from the side verges of a federal road (17 05 03*)



Left: heap with vertical breakline, right: detail of the breakline Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Beginning	19 October 2022, 2:30 p	19 October 2022, 2:30 p.m.		
End	19 October 2022, 4:00 p.m.			
Place of treatment	Westsächsische Entsorgungs- und Verwertungsgesellschaft mbH, Cröbern			
Weather conditions	Clouded, 10°C			
Sample preparation	Creation of a sieve line using original material with hand screening on round-hole screens. Screen cut <4 mm round-hole sieve represents the laboratory sample. Oversized particles and interfering material were removed, as no jaw crusher was available on site.			
Sieve curve original material and approximate distribution	The particle size of the soil is approximately lognormally distributed. The median is approx. 3 mm. The d ₉₅ is approx. 18 mm (see also Figure 14).			
Data on the sieve curve	Weight [g]	Percentage by weight [%]	Cumulative percentage by weight [%]	
<4 mm	11,210	79.2%	79.3%	
<5 mm	93	0.7%	79.9%	
<10 mm	1,307	9.2%	89.1%	
<15 mm	540	3.8%	93.0%	
<20 mm	995 7.0% 100.0%			
Sample pretreatment	It can be expected that some drying of the material will take place. No metallic material is included. As contaminants, there are few organic plant parts. There are larger amounts of clay particles that escape hand screening by agglomeration. They are separated as oversized particles. The proportion of clay in the separated material >10 mm is 84%. Whether and to what extent is found in the sample material <4 mm clay is unknown.			

Table 12: Sample pretreatment and sample preparation according to DIN 19747 (2009a)

Mass balance of pre- treatment	Factions	Weight [g]	Share [%]
	Input	14,203.0	100.0%
	Sample <4 mm	11,210.0	78.9%
	Biogenic material	10.0	0.1%
	Oversized particles	2,925.0	20.6%
	Losses	58.0	0.4%
Storage	None		
Transport	19 October 2022, from 4:30 p.m.		
Transport container	PE bag in cooled 60-liter PP barrel in a car		
Transport by	Ralf Ketelhut		
Handover	20 October 2022, 8:30 a.m.		
Recipients	ECT Oekotoxikologie GmbH		





Manual screening on round hole screens

Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Figure 14: Photographic documentation for material from the side verges of a federal road (17 05 03)



Top left: >10 mm; top right: >5/<10 mm, center left: >4/<5 mm, center right: impurities, below: <4 mm Source: own illustration, Ralf Ketelhut, Stoffstromdesign

7 Soil and stones (17 05 04): material from the side verges of a secondary road

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018), requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

	Waste code	17 05 04 (Soils and stones other than those mentioned in 17 05 03)
	Identification	Material from the side verges of a secondary road
Sponsor Project Overall objective		German Environment Agency/ECT Oekotoxikologie GmbH
		HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'
		Collection of a probabilistic sample and preparation for ecotoxicological testing
	Waste owner	Is known to the sampler
	Project management	ECT Oekotoxikologie GmbH
	Sampler	Ralf Ketelhut Stoffstromdesign
	Sampling date, time	17 May 2022, 7:05 a.m. to 7:30 a.m.
Weather conditions Relevant background for the sampled waste		Light rain, 13°C, 60% relative humidity
		In the side verge area, impurities and deposits from vegetation residues and road dirt accumulate. Depending on the traffic volume, direct material input from traffic emissions as well as abrasion of tires, roadways and brakes are to be expected. In addition, road salt residues may be relevant. The side verge is periodically peeled off at a width of about 80 cm with a layer thickness of 3-10 cm approximately every 3-8 years. Here the removal was carried out on both sides of a district road to prepare for the use of an asphalt milling machine.
	Sampling point	Storage space for excavated side verge material in northern Germany. Intermediate stockpile of approx. 100 m ³ from a main unit of about 400 m ³ over a period of several weeks.
Material description		The heap has a limited vegetation cover.
	Variability	No specific spatial heterogeneity to be expected, large number of small influences, particulate heterogeneity rather low.
	Specific information	The material is classified as Z2, but not hazardous, according to LAGA soil analysis. This means that the material may only be disposed of or installed to a limited extent. The reasons for the classification according to LAGA are PAKs according to the information provided by the waste producer.

Table 13: Sampling

Batch size	Pile of 100 m ³ or 92 Mg (estimated)		
Mass flow	None		
Bulk density	$\rho_B = 0.92 \text{ kg/dm}^3$ (estimated)		
Raw density	$\rho_R = 1.8 \text{ kg/dm}^3$ (estimated)		
Particle dimension	d ₉₅ = 20 mm (estimated)		
Particle type	Grainy		
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D	The European working group on the characterization of waste (CEN/TC 292) states that an estimate of the minimum sample size can be obtained according to the equation indicated in Figure 13 of the report. The calculation was performed using the following values: M_{SAM} : Minimum sample size [kg], here: $M_{SAM} = 1.7$ kg d_{95} : Nominal screen size of particles [cm], here 2 cm p_p : Particle density [g/cm ³], here 1.8 g/cm ³ g: Correction factor for particle size, here g = 0.25, since $d_{95}/d_{05} > 4$ p: Fraction of particles with a certain characteristic, if not known, then $p = 10\%^3$ CV: Coefficient of variation, if not known, then CV = 10%		
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples:	Number of laboratory samples (LS):
	16	4	4
Minimum sample size	d95:	Volume of RS:	Volume of LS:
according to LAGA PN 98	>2 to ≤20 mm	1 dm³	2 dm³
	No sample size reduction of the composite samples to I was made to avoid increasing the variance of the sampl mass was increased to obtain sufficient material.		s to laboratory samples amples. The sample
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{CS} = (number of RS • V _{RS}) Number of RS: 9	M _{CS} = V _{CS} • ρ _B
	0.22 dm³	1.8 dm³	1.7 kg
Type of sampling according to CEN/TR 15310-1	Restricted random sampling (<i>judgmental sampling 1</i>). No wheel loader available. The pile indicates a sufficient mixing by accumulation. Since it is an overgrown heap, superficial influence due to erosion can be expected. Therefore, approximately 10 cm of surface material is taken away and 16 individual samples are taken with a 50 mm piercing tube. Real sample: 16.6 dm ³ . 15.3 kg		
Storage	Transfer to PE bag and then cooled 60-liter PP barrel with clamping ring closure.		
Transport at, start	17 May 2022, 7:30 a.m.		
Transport by	Ralf Ketelhut		
Sample preparation site	CTL Luers, Bremen		
Arrival at the sample preparation site	17 May 2022, 2:00 p.m.		

Figure 15: Photographic documentation for material from the side verges of a secondary road (17 05 04)



Sampled piles from two different perspectives Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Beginning	17 May 2022, 2:20 p.m.		
End	17 May 2022, 4:20 p.m.		
Place of treatment	CTL Luers, Bremen		
Weather conditions	Clouded, 17°C		
Sample preparation	Creation of a sieve line using original material with hand screening on round-hole screens. Sieve opening <4 mm round-hole screen. Oversized particles were repeatedly broken using a jaw crusher (Retsch type BB 1A). Interfering material was removed.		
Sieve curve original material and approximate distribution	The particle size of the s median is approx. 3 mm	oil is approximately logno . The d ₉₅ is approx. 25 mm	rmally distributed. The n (see also Figure 16).
Data on the sieve curve	Weight [g]	Percentage by weight [%]	Cumulative percentage by weight [%]
<4 mm	9,570.0	64.6%	64.6%
<5 mm	282.0	1.9%	66.5%
<10 mm	1,901.8	12.8%	79.3%
<15 mm	1,517.9	10.2%	89.6%
<20 mm	594.8	4.0%	93.6%
<30 mm	567.0	3.8%	97.4%
<40 mm	386.3	2.6%	100.0%
Sample pretreatment	It can be expected that some drying of the material will take place. The sample does not include metallic material. As contaminants, there are few organic plant parts.		
Mass balance of pre-	Factions	Weight [g]	Share [%]
treatment	Input	15,260.0	100.0%

Table 14: Sample pretreatment and sample preparation according to DIN 19747 (2009a)

Beginning	17 May 2022, 2:20 p.m.		
	Sample <4 mm	14,780.0	96.9%
	Biogenic material	12.6	0.1%
	Losses	467.4	3.1%
Storage	None		
Transport	17 May 2022, from 4:30 p.m.		
Transport container	PE bag in cooled 60-liter PP barrel in a car		
Transport by	Ralf Ketelhut		
Handover	18 May 2022, 8:30 a.m.		
Recipients	ECT Oekotoxikologie GmbH		





Manual screening on round hole screens

Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Figure 17: Photographic documentation for material from the side verges of a secondary road (17 05 04)



Top left: >20 mm, top right: >10 mm, center left: >4 mm, center right: impurities, below: sample <4 mm Source: own illustration, Ralf Ketelhut, Stoffstromdesign

8 Fluff-light fraction and dust (19 10 04): material (sieved to <10 mm) from plant A, batch 1

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018), requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

Waste code	19 10 04 (Fluff-light fraction and dust other than those mentioned in 19 10 03)	
Identification	Fluff-light fraction (material sieved to <10 mm) from plant A, batch 1	
Sponsor	German Environment Agency/ECT Oekotoxikologie GmbH	
Project	HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'	
Overall objective	Collection of a probabilistic sample and preparation for ecotoxicological testing	
Waste owner	Is known to the sampler	
Project management	ECT Oekotoxikologie GmbH	
Sampler	Ralf Ketelhut Stoffstromdesign	
Sampling date, time	17 May 2022, 9:30 a.m. to 10:30 a.m.	
Weather conditions	Cloudy, light rain, 14°C, 60% relative humidity	
Relevant background for the sampled waste	Fluff-light fractions are considered an extreme example of heterogeneous waste material. In modern plants, material recycling is significantly improved by automated sorting technology. This results in waste with a d ₉₅ ≤10 mm.	
Sampling point	Premises of a metal recycler in Germany	
Material description	Continuously occurring material (<10 mm) from processing of fluff-light fractions from Fe metals processing. Heterogeneous mixture of metal, mineral, biogenic and synthetic materials.	
Variability	Temporal-spatial heterogeneity is possible. Particular heterogeneity is very high but reduced by sieving.	
Specific information	The sample covers an operating time of about 2 h. It has, therefore, random sample characteristics.	
Batch size	Collection containers with a filling of 20 m ³ or 12 Mg (estimated)	
Mass flow	Not known	
Bulk density	$\rho_B = 0.6 \text{ kg/dm}^3$ (estimated)	

Table 15: Sampling

Raw density	$\rho_R = 1.2 \text{ kg/dm}^3$ (estimated)		
Particle dimension	d ₉₅ = 15 mm (estimated)		
Particle type	Heterogeneous		
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D	The European working group on the characterization of waste (CEN/TC 292) states that an estimate of the minimum sample size can be obtained according to the equation indicated in Figure 13 of the report. The calculation was performed using the following values: M _{SAM} : Minimum sample size [kg], here: M _{SAM} = 0.5 kg d ₉₅ : Nominal screen size of particles [cm], here 1.5 cm p_p : Particle density [g/cm ³], here 1.2 g/cm ³ g: Correction factor for particle size, here g = 0.25, since d ₉₅ /d ₀₅ >4 p: Fraction of particles with a certain characteristic, if not known, then $p = 10\%^3$ CV: Coefficient of variation, if not known, then CV = 10%		
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples:	Number of laboratory samples (LS):
	8	2	2
Minimum sample size	d95:	Volume of RS:	Volume of LS:
according to LAGA PN 98	>2 to 20 mm	1 dm³	2 dm³
	No sample size reduction of the composite samples to laboratory samples was made to avoid increasing the variance of the samples. The sample mass was increased to obtain sufficient material.		
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{CS} = (number of RS • V _{RS}) Number of RS: 9	$M_{CS} = V_{CS} \bullet \rho_B$
	0.09 dm³	0.8 dm³	0.5 kg
Type of sampling according to CEN/TR 15310-1	Restricted random sampling (<i>judgmental sampling 1</i>). Sampling of 4 subsamples of approx. 50 dm ³ with grabbed excavator. Mixing and spreading. Collection of 30 individual samples of approx. 0.4 dm ³ (0.25 kg) with a 50 mm piercing tube. Real sample approx. 12 dm ³ , 7.2 kg		
Storage	Transfer to PE bag and then cooled 60-liter PP barrel with clamping ring closure.		
Transport at, start	17 May 2022, 10:30 a.m.		
Transport by	Ralf Ketelhut		
Sample preparation site	CTL Luers, Bremen		
Arrival at the sample preparation site	17 May 2022, 2:00 p.m.		

Figure 18: Photographic documentation for fluff-light fraction from plant A, batch 1 (19 10 04)



Original material before sample pretreatment Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Beginning	17 May 2022, 4:30 p.m.			
End	17 May 2022, 5:45 p.m.			
Place of treatment	CTL Luers, Bremen			
Weather conditions	Clouded, 17°C			
Sample preparation	Creation of a sieve line using original material with hand screening on round-hole screens. Jaw crushers and cutters were available, but not suitable for the material.			
Sieve curve original material and approximate distribution	The particle size of the material is approximately lognormally distributed. The median is approx. 2.5 mm. The d ₉₅ is approx. 9 mm (see also Figure 19).			
Data on the sieve curve	Weight [g]Percentage by weightCumulative[%]percentage by weight[%]			
<4 mm	5,700.0	80.4%	80.4%	
<5 mm	293.0	4.1%	84.5%	
<10 mm	860.7	12.1%	96.6%	
<15 mm	105.5	1.5%	98.1%	
<20 mm	132.1 1.9% 100.0%			
Sample pretreatment	No shredding of the oversized particles. The material is fibrous and fluffy, so that – within the time available – shredding is not feasible. Material >4 mm is discarded as oversized particles.			

Table 16: Sample pretreatment and sample preparation according to DIN 19747 (2009a)

Mass balance of pre-	Factions	Weight [g]	Share [%]
treatment	Input	7,160.0	100.0%
	Sample <4 mm	5,700.0	79.6%
	Impurities	60.0	0.8%
	Oversized particles >4 mm	1,391.3	19.4%
	Losses	8.7	0.1%
Storage	None		
Transport	17 May 2022, from 5:45 p.m.		
Transport container	PE bag in cooled 60-liter	PP barrel in a car	
Transport byRalf KetelhutHandover18 May 2022, 8:30 a.m.			
Recipients	ECT Oekotoxikologie GmbH		

Figure 19: Sieve curve for fluff-light fraction (19 10 04) from plant A, batch 1





Source: own illustration, Ralf Ketelhut, Stoffstromdesign



Figure 20:Photographic documentation for fluff-light fraction (19 10 04) from plant A, batch 1

Top left: >20 mm, top right: >15 mm, center left: 10 mm, center right: 4 mm, bottom left: impurities, lower right: sample <4 mm

Source: own illustration, Ralf Ketelhut, Stoffstromdesign

9 Fluff-light fraction and dust (19 10 04): material (sieved to <10 mm) from plant A, batch 2

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018), requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

Waste code	19 10 04 (Fluff-light fraction and dust other than those mentioned in 19 10 03)	
Identification	Fluff-light fraction (material sieved to <10 mm) from plant A, batch 2	
Sponsor	German Environment Agency/ECT Oekotoxikologie GmbH	
Project	HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'	
Overall objective	Collection of a probabilistic sample and preparation for ecotoxicological testing	
Waste owner	Is known to the sampler	
Project management	ECT Oekotoxikologie GmbH	
Sampler	Ralf Ketelhut Stoffstromdesign	
Sampling date, time	14 February 2023, 8:30 a.m. to 8:45 a.m.	
Weather conditions	Cloudy, 3°C, 80% relative humidity	
Relevant background for the sampled waste	Fluff-light fractions are considered an extreme example of heterogeneous waste material. In modern plants, material recycling is significantly improved by automated sorting technology. This results in waste with a d95 ≤10 mm.	
Sampling point	Premises of a metal recycler in Germany	
Material description	Continuous removal from fluff-light fraction and dust from the processing of Fe metals. Heterogeneous mixture of metals, minerals, biogenic and synthetic materials.	
Variability	A temporal-spatial heterogeneity is possible. The partial heterogeneity is very high but is reduced by the sieve opening.	
Specific information	The sample covers an operating time of about 2 hours. It has, therefore, random sample characteristics.	
Batch size	Collection containers with a filling of 12 m ³ or 7.2 mg (estimated)	
Mass flow	Not known	
Bulk density	$\rho_B = 0.6 \text{ kg/dm}^3$ (estimated)	

Table 17: Sampling

Raw density	$\rho_R = 1.2 \text{ kg/dm}^3$ (estimated)		
Particle dimension	d_{95} = 10 mm (estimated from preliminary investigation batch 1)		
Particle type	heterogeneous The European working group on the characterization of waste (CEN/TC 292) states that an estimate of the minimum sample size can be obtained according to the equation indicated in Figure 13 of the report. The calculation was performed using the following values: M_{SAM} : Minimum sample size [kg], here: $M_{SAM} = 0.14$ kg d95: Nominal screen size of particles [cm], here 1 cm p_{p} : Particle density [g/cm ³], here 1.2 g/cm ³ g: Correction factor for particle size, here g = 0.25, since d95/d05 >4 p: Fraction of particles with a certain characteristic, if not known, then $p = 10\%^{3}$ CV: Coefficient of variation, if not known, then CV = 10%		
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D			
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples:	Number of laboratory samples (LS):
	8	2	2
Minimum sample size according to LAGA PN 98	d ₉₅ :	Volume of RS:	Volume of LS:
	>2 to ≤20 mm	1 dm³	2 dm³
	No sample size reduction of the composite samples to laborator was made to avoid increasing the variance of the samples. The mass was increased to obtain sufficient material.		s to laboratory samples amples. The sample
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{cs} = (number of RS • V _{RS}) Number of RS: 9	$M_{CS} = V_{CS} \bullet \rho_B$
	0.03 dm³	0.24 dm³	0.14 kg
Type of sampling according to CEN/TR 15310-1	Restricted random sampling (<i>judgmental sampling 1</i>). Sampling of 12 individual samples of approx. 1 dm ³ (0.6 kg) with a shovel from the container. Real sample: approx. 12 dm ³ , 7.3 kg.		
Storage	Transfer to PE bag and then cooled 60-liter PP barrel with clamping ring closure.		
Transport at, start	14 February 2023, 8:45 a.m.		
Transport by	Ralf Ketelhut		
Sample preparation site	Wookerkamp 61, 24536 Neumünster		
Arrival at the sample preparation site	17 May 2022, 12:00		



Figure 21:Photographic documentation for fluff-light fraction from plant A, batch 2 (19 10 04)

Original material before sample pretreatment, above: in the container, below: detail of the sample Source: own illustration, Ralf Ketelhut, Stoffstromdesign

			0	
	Beginning	14 February 2023, 1:00 p.m.		
	End	14 February 2023, 1:45 p.m.		
	Place of treatment	Ralf Ketelhut, Wookerkamp 61, 24536 Neumünster		
	Weather conditions	Cloudy, 4°C		
	Sample preparation	Sieve curve made using original material and hand screening on round- hole screens. Sieve opening <4 mm round-hole screen. Removal of oversized particles and interfering material.		
	Sieve curve original material and approximate distribution	The particle size of the material is approximately lognormally distributed. The median is approx. 2.3 mm. The d ₉₅ is approx. 8 mm (see also Figure 22).		
	Data on the sieve curve	Weight [g]	Percentage by weight [%]	Cumulative percentage by weight [%]
	<4 mm	5.828,0	80.6%	80.6%
	<5 mm	245,0	3.4%	84.0%
	<10 mm	866,5	13.4%	97.4%
	<15 mm	191,0	2.6%	100.0%
	Sample pretreatment	No shredding of the oversized particles. The material is fibrous and fluffy, so that – within the time available – shredding is not feasible. Material >4 mm is discarded as oversized particles.		
Mass balance of pre-		Factions	Weight [g]	Share [%]
	treatment	Input	7.258,0	100.0%
		Sample <4 mm	5.828,0	80.3%
		Impurities	0,0	0.0%
		Oversized particles >4 mm	1.402,5	19.3%
		Losses	27,5	0.4%
	Storage	None		
	Transport	14 February 2023, from 2:00 p.m.		
	Transport container	PE bag in cooled 60-liter PP barrel in a car		
	Transport by	Ralf Ketelhut		
	Handover	15 February 2023, 8:30 a.m.		
	Recipients	ECT Oekotoxikologie GmbH		

Table 18: Sample pretreatment and sample preparation according to DIN 19747 (2009a)





Manual screening on round hole screens

Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Figure 23: Photographic documentation for fluff-light fraction from plant A, batch 2 (19 10 04)



Top left: >10 mm, top right: >5 mm, center left: >4 mm, bottom: sample <4 mm Source: own illustration, Ralf Ketelhut, Stoffstromdesign

10 Fluff-light fraction and dust (19 10 04): material (sieved to <10 mm) from plant B

In accordance with the requirements of European standardisation in the framework of CEN/TC 292 (characterization of waste), careful sampling planning is required for the sampling of waste for physical, chemical or ecotoxicological characterization. This accompanying protocol on sampling and sample preparation is based on data provided by the waste owner and the sampler's assessments in the light of the recommendations of the 'Commission notice on technical guidance on the classification of waste' (EU 2018), requirements of CEN/TR 15310-1 (2006a), DIN EN 14735 (2022), LAGA PN 98 (2019), and the 'Recommendations for the ecotoxicological characterization of wastes' of the UBA (2013).

Waste code	19 10 04 (Fluff-light fraction and dust other than those mentioned in 19 10 03)	
Identification	Fluff-light fraction (material sieved to <10 mm) from plant B	
Sponsor	German Environment Agency/ECT Oekotoxikologie GmbH	
Project	HP 14 classification of mirror entries in the List of Wastes – elaboration of proposals for further developing the German 'Recommendations for the ecotoxicological characterization of wastes'	
Overall objective	Collection of a probabilistic sample and preparation for ecotoxicological testing	
Waste owner	Is known to the sampler	
Project management	ECT Oekotoxikologie GmbH	
Sampler	Ralf Ketelhut Stoffstromdesign	
Sampling date, time	14 February 2023, 10:40 a.m. to 11:00 a.m.	
Weather conditions	Cloudy, 4°C, 80% relative humidity	
Background to the waste sample	Fluff-light fractions are considered an extreme example of heterogeneous waste material. In modern plants, material recycling is significantly improved by automated sorting technology. This results in waste with a d ₉₅ ≤10 mm.	
Sampling point	Premises of a metal recycler in Germany	
Material description	Discontinuous removal from the processing of fluff-light fraction and dust from the processing of Fe metals. Heterogeneous mixture of metallic, mineral, biogenic and synthetic materials.	
Variability	A temporal-spatial heterogeneity is possible but reduced by sieving. The partial heterogeneity is very high but is also reduced by sieving.	
Specific information	The material is produced by discontinuous screening with a mobile rotary screen.	
Batch size	Collection pile of approx. 100 m ³ or 80 mg (estimated)	
Mass flow	Not known	
Bulk density	$\rho_B = 0.8 \text{ kg/dm}^3$ (estimated)	

Table 19: Sampling

Raw density	$\rho_R = 1.2 \text{ kg/dm}^3$ (estimated)		
Particle dimension	d ₉₅ = 10 mm (estimated)		
Particle type	Heterogeneous		
Minimum sample mass according to CEN/TC 292 CEN/TR 15310-1, Annex D	The European working group on the characterization of waste (CEN/TC 292) states that an estimate of the minimum sample size can be obtained according to the equation indicated in Figure 13 of the report. The calculation was performed using the following values: M_{SAM} : Minimum sample size [kg], here: $M_{SAM} = 0.14$ kg d95: Nominal screen size of particles [cm], here 1 cm p_p : Particle density [g/cm ³], here 1.2 g/cm ³ g: Correction factor for particle size, here g = 0.25, since d95/d05 >4 p: Fraction of particles with a certain characteristic, if not known, then $p = 10\%^3$ CV: Coefficient of variation, if not known, then CV = 10%		
Sample numbers according to LAGA PN 98	Number of individual samples (RS):	Number of composite samples:	Number of laboratory samples (LS):
	16	4	4
Minimum sample size according to LAGA PN 98	d ₉₅ :	Volume of RS:	Volume of LS:
	>2 to ≤20 mm	1 dm³	2 dm³
	No sample size reduction of the composite samples to laboratory was made to avoid increasing the variance of the samples. The sa mass was increased to obtain sufficient material.		s to laboratory samples amples. The sample
Sample size according to CEN/TR 15310-1	$V_{RS} = (3 \cdot d_{95})^3$	V _{cs} = (number of RS • V _{RS}) Number of RS: 7	$M_{CS} = V_{CS} \bullet \rho_B$
	0.02 dm³	0.18 dm³	0.14 kg
Type of sampling according to CEN/TR 15310-1	Restricted random sampling (<i>judgmental sampling 1</i>). Sampling of 16 individual samples of approx. 0.4 dm ³ (0.3 kg) sampling pipe 50 mm. Real sample: approx. 6.6 dm ³ , 5.3 kg.		
Storage	Transfer to PE bag and then cooled 60-liter PP barrel with clamping ring closure.		
Transport at, start	14 February 2023, 11:00 a.m.		
Transport by	Ralf Ketelhut		
Sample preparation site	Wookerkamp 61, 24536 Neumünster		
Arrival at the sample preparation site	17 May 2022, 12:00		



Figure 24: Photographic documentation for fluff-light fraction from plant B (19 10 04)

Original material before sample pretreatment, above: original heap, below: detail sample before pre-treatment Source: own illustration, Ralf Ketelhut, Stoffstromdesign

Beginning	14 February 2023, 12:00		
End	14 February 2023, 1:00 p.m.		
Place of treatment	Ralf Ketelhut, Wookerkamp 61, 24536 Neumünster		
Weather conditions	Cloudy, 4°C		
Sample preparation	Creation of a sieve line using original material with hand screening on round-hole screens. Sieve opening <4 mm round-hole screen. Removal of oversized particles and interfering material.		
Sieve curve original material and approximate distribution	The particle size of the material is approximately lognormally distributed. The median is approx. 2.3 mm. The d_{95} is approx. 7.5 mm.		
Data on the sieve curve	Weight [g]	Percentage by weight [%]	Cumulative percentage by weight [%]
<4 mm	4,524.5	86.1%	86.1%
<5 mm	231.5	4.4%	90.5%
<10 mm	485.0	9.2%	99.7%
<15 mm	14.5	0.3%	100.0%
Sample pretreatment	No shredding of the oversized particles. The material is fibrous and fluffy, so that – within the time available – shredding is not feasible. Material >4 mm is discarded as oversized particles.		
Mass balance of pre-	Factions	Weight [g]	Share [%]
treatment	Input	5,277.5	100.0%
	Sample <4 mm	4,524.5	85.7%
	Impurities	0.0	0.0%
	Oversized particles >4 mm	731.0	13.9%
	Losses	22.0	0.4%
Storage	None		
Transport	14 February 2023, from 2:00 p.m.		
Transport container	PE bag in cooled 60-liter PP barrel in a car		
Transport by	Ralf Ketelhut		
Handover	15 February 2023, 8:30 a.m.		
Recipients	ECT Oekotoxikologie GmbH		

Table 20: Sample pretreatment and sample preparation according to DIN 19747 (2009a)





Manual screening on round hole screens

Source: own illustration, Ralf Ketelhut, Stoffstromdesign





Top left: >10 mm, top right: >5 mm, center: >4 mm, bottom: sample <4 mm Source: own illustration, Ralf Ketelhut, Stoffstromdesign