PD CEN/TS 16011:2013

BSI Standards Publication

Plastics — Recycled plastics — Sample preparation

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Plastiques - Plastiques recyclés - Préparation des échantillons

Kunststoffe - Kunststoff-Rezyklate - Probenvorbereitung

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Contents

Foreword

This document (CEN/TS 16011:2013) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This Technical Specification is one part of a series of CEN publications on Plastics Recycling that is structured as follows:

- EN 15342, *Plastics — Recycled Plastics — Characterization of polystyrene (PS) recyclates*
- EN 15343, *Plastics — Recycled Plastics — Plastics recycling traceability and assessment of conformity and recycled content*
- EN 15344, *Plastics — Recycled Plastics — Characterisation of Polyethylene (PE) recyclates*
- EN 15345, *Plastics — Recycled Plastics — Characterisation of Polypropylene (PP) recyclates*
- EN 15346, *Plastics — Recycled plastics — Characterisation of poly(vinyl chloride) (PVC) recyclates*
- EN 15347, *Plastics — Recycled Plastics — Characterisation of plastics wastes*
- EN 15348, *Plastics — Recycled plastic — Characterization of poly(ethylene terephthalate) (PET) recyclates*
- CEN/TR 15353, *Plastics — Recycled Plastics — Guidelines for the development of standards for recycled plastics*
- CEN/TS 16010, *Plastics — Recycled plastics — Sampling procedures for testing plastics waste and recyclates*

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Recycling of plastics waste is one type of material recovery process intended to save resources (virgin raw materials, water, energy), while minimising harmful emissions into air, water and soil as well as their impacts on human health. The environmental impact of recycling should be assessed over the whole life cycle of the recycling system (from the waste generation point to the disposal of final residues). To ensure that recycling constitutes the best environmental option for treating the available waste, some prerequisites should preferably be met:

- the recycling scheme being contemplated should generate lower environmental impacts than alternative recovery options;
- existing or potential market outlets should be identified that will secure a sustainable industrial recycling operation;
- the collection and sorting schemes should be properly designed to deliver recyclable plastics waste fractions fitting reasonably well with the available recycling technologies and with the (changing) needs of the identified market outlets, preferably at minimum costs for society.

This Technical Specification has been produced in accordance with the guidance produced by CEN on Environmental Aspects and in accordance with CEN/TR 15353, *Plastics — Recycled Plastics — Guidelines for the development of standards for recycled plastics*.

NOTE CEN/TR 15353 considers the general environmental aspects which are specific to the recycling process.

The purpose of this Technical Specification is to define the procedures to be followed to prepare samples taken in accordance with CEN/TS 16010 in readiness for testing various material characteristics as set out in other relevant standards for recycled plastics (EN 15342 to EN 15348).

It is not the intention of this Technical Specification to develop new sample preparation methods.

1 Scope

This Technical Specification specifies the preparation of samples of recycled plastics prior to testing and takes account of the specifics of the material.

Sample preparation should avoid any process that causes 'de-mixing' of the sample. Following preparation, the sample should remain representative.

The behaviour of contaminants should be carefully analysed and observed to ensure this is homogeneous.

This Technical Specification does not address any legal or product safety issues.

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.

CEN/TR 15353:2007, *Plastics — Recycled plastics — Guidelines for the development of standards for recycled plastics*

CEN/TS 16010, *Plastics — Recycled plastics — Sampling procedures for testing plastics waste and recyclates*

EN ISO 472:2001, *Plastics — Vocabulary (ISO 472:1999)*

ISO 3085, *Iron ores — Experimental methods for checking the precision of sampling, sample preparation and measurement*

ISO 3086, *Iron ores — Experimental methods for checking the bias of sampling*

ISO 11648-1, *Statistical aspects of sampling from bulk materials — Part 1: General principles*

ISO 11648-2:2001, *Statistical aspects of sampling from bulk materials — Part 2: Sampling of particulate materials*

ISO 13909-7, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing*

ISO 13909-8, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias*

3 Terms and definitions

For the purposes of this document, the terms, definitions and abbreviated terms given in EN ISO 472:2001, in CEN/TR 15353:2007 apply.

4 Symbols and abbreviations

C_v coefficient of variation

5 General requirements

5.1 Implementation

Successful implementation of this Technical Specification depends on effective communication between the data user, the sampler and test laboratory. The aim is to close the information chain of the whole processes around the sampling in combination with the laboratory analysis.

NOTE The data user could be the supplier, purchaser or a third party.

5.2 Instructions

The appropriate instructions for sampling, sample preparation and testing shall be reviewed by all those involved in the sampling and testing process. If there are no instructions, the test laboratory should contact the data user.

5.3 Storage

Samples should be collected in appropriate containers for the tests required. The storage conditions shall ensure that the sample characteristics do not change.

5.4 Documentation

The whole sampling and sub-sampling process shall be documented with all relevant facts recorded. It is also useful to record the apparatus used and their calibration details.

5.5 Sample preparation rooms

All sample preparations shall be done in rooms or at places which do not influence the samples, or where the influence is negligible. All these areas shall be free from contamination, be easily decontaminated and be vented to control dust and remove fumes. It is good laboratory practice to avoid unnecessary flame, heat and fume.

5.6 Volatiles

If volatiles and non-volatiles are to be determined, then precautions shall be taken to ensure that the volatiles are not able to escape during the sampling and sample preparation process.

6 Sampling

Take the sample in accordance with CEN/TS 16010.

7 Methods

7.1 General

The general principles of sample preparation are described in Annex A and ISO 11648-2.

7.2 Determination of the minimum laboratory sample

The determination of the minimum laboratory sample is dependent on the characteristics and properties to be tested. It should be noted that the minimum mass of the laboratory analysis sample increases with the maximum grain size or mass of the individual particle of the sample.

In Table 1 the relationship is given between the maximum particle size and sample mass to achieve a different fundamental error with 15 %, 10 % and 5 %. The determination of this minimum value is dependent on the maximum mass/particle size of the material being analysed, the required measurement tolerance and the statistical probability value with which the result is correlated. If no information is available, the minimum value of the laboratory sample should be determined by Table 1. Note the different coefficients of variation C_v.

Samples for the determination of moisture should be a minimum of 1 kg, see 7.5.2.

Table 1 — Minimum value of the laboratory sample

Required minimum mass of the laboratory sample g	**Maximum allowable particle size**[a] cm $C_v < 15$ %	**Maximum allowable particle size**[a] cm $C_v < 10$ %	**Maximum allowable particle size**[a] cm $C_v < 5$ %	**Sieve size (ISO 565)**
0,1	0,05	0,04	0,03	500 µm
1	0,10	0,08	0,06	1,0 mm
2	0,13	0,10	0,08	1,4 mm
5	0,17	0,13	0,10	1,7 mm
10	0,21	0,16	0,13	2,0 mm
30	0,31	0,24	0,19	2,8 mm
50	0,37	0,28	0,22	3,35 mm
100	0,46	0,35	0,28	4,0 mm

[a] The particle size is dependent on the maximum linear dimension of the particle. It is the determined effective size or size distribution of particles, mostly done by manual or automatic sieves.

7.3 Homogenisation of samples

Suitable equipment for sample homogenisation and division are the riffle splitter or the sectorial splitter.

7.4 Sample preparation before mechanical testing

The preparation of samples before mechanical testing is dependent on the physical form of the material. Usually the aim is to produce a moulded part prior to mechanical testing. For flakes and agglomerates then additional granulation (size reduction) might be required before moulding. If the sample is in the form of a compound and information is available on the melt filtration used, then additional granulation might be unnecessary. See Table 2.

Table 2 — Sample preparation before mechanical testing

Powder	**Flake**	**Agglomerate**	**Compound**	**Moulded part**
				shredding
	granulation (optional)	granulation (optional)	granulation (optional)	granulation (optional)
moulding	moulding	moulding	moulding	moulding
testing	testing	testing	testing	testing

7.5 Preparation of samples for the determination of moisture

7.5.1 Sample Type

The sample to be tested shall be either:

a) a sample collected exclusively for the determination of moisture; or

b) a sample on which determinations of moisture and other quality characteristics are required.

7.5.2 Mass

The mass of each moisture sample shall not be less than 1 kg. Recommended masses of samples at various nominal maximum particle sizes are given in Table 3 (taken from ISO 11648-2:2001). Samples of nominal maximum particle size greater than 22,4 mm may be subjected to a two-stage drying procedure, as described in Annex A.4.1 b).

Table 3 — Example of recommended minimum mass of sample for the determination of moisture

Nominal maximum particle size of material mm	Maximum layer thickness mm	Minimum mass kg	Maximum allowable difference between subsequent weightings of dried samples g	Accuracy of weighing g	Minimum drying time h
63,0	70	110	110	10	16
45,0	50	40	40	4	12
31,5	35	14	14	1	8
22,4	25	5	5	0,5	6
16,0	20	2	2	0,2	4
11,2	13	1	1	0,1	4

7.5.3 Precautions against loss of moisture

One of the main difficulties in determining moisture is that of minimising changes in the moisture content of the sample when preparing the moisture sample. Take every precaution to minimise changes in moisture content due to unsuitable containers and by evaporation during handling, particularly if the bulk material is extremely wet. Keep all moisture samples in sealed containers in a cool place and out of direct sunlight before and after preparation as well as during any interval between stages of sample preparation.

Take care to minimise changes in moisture content during particle size reduction, by using equipment in which there is no appreciable heating, and by reducing the amount of air passing through the mill to a minimum.

Take care to minimise changes in moisture content when carrying out sample division; carry out such operations as quickly as possible. In some circumstances, it might be necessary to carry out moisture determinations on each increment, to minimise moisture changes (see Annex B).

Where moisture samples are to be retained for any length of time; for example for more than five to seven days in the case of umpire and shipping samples, place them into moisture-impervious plastic bags which are sealed so as to minimise free air space. Then store them in an airtight container.

7.5.4 Samples for determination of moisture

Moisture in bulk material may be determined by heating a sample of material at 105 °C in air until constant mass is achieved.

Preparation of the sample may include preliminary air-drying (see A.3) if the material is visibly wet.

7.6 Preparation of samples for chemical analysis

7.6.1 General

The procedure for preparation of the chemical analysis sample is described in 7.5.2 and shown schematically in Annex B. The moisture sample may be used as the chemical analysis sample after drying, provided it is sufficiently representative.

7.6.2 First stage in the preparation

If necessary, the sample may be dried in accordance with A.3. Pass the whole sample through the size reduction equipment (see A.4), mix it, then divide it (see A.6) to decrease the mass to the value appropriate to the nominal maximum particle size of the size reduced bulk material (see A.2).

7.6.3 Further stages in the preparation

In the further stages of preparation, reduce the particle size of the portion retained from the first stage to the required nominal maximum particle size for analysis (see A.4), then divide it to obtain as many samples as contractually required, each having a recommended mass of no less than 50 g.

Then place the chemical analysis sample in a sealed container and label it with all necessary identification details.

8 Precision of sample preparation

Check the precision of sample preparation periodically and whenever a new scheme is brought into operation.

Test procedures for the determination of precision in sample preparation are given in ISO 3085 and ISO 13909-7 and these procedures may be applied to other bulk materials.

9 Bias in sample preparation

9.1 General

Check the bias in sample preparation periodically and whenever a new scheme is brought into operation.

Procedures for checking bias in sample preparation are given in ISO 3086 and ISO 13909-8 and these procedures may be applied to other bulk materials.

Contamination is often a major source of bias during sample preparation. Contamination might be attributable to preparation equipment or cross-contamination.

9.2 Cross-contamination

Potential causes of cross-contamination include the following:

a) **Dust:** Dust settling on open samples can be a possible cause of contamination. Minimise dust settling by ensuring as much work as possible is carried out using a dust extraction system. If possible, any dust escaping from size reduction equipment should be extracted downwards and to the rear. Keep all ducting, machinery and floors clean.

b) **Sample:** To prevent cross-contamination, clean all equipment used between samples, ensuring that the sample has been removed first.

10 Packing and marking of samples

The samples for distribution shall be tightly sealed in air-tight containers. The label, and a card placed in the container, shall contain the following particulars:

a) the type of recycled plastic and the name of the lot (e.g. lot reference number);

b) the mass of the lot;

c) the sample reference number;

d) the place and the date of sampling;

e) the moisture content of the sample (if known);

f) the place and the date of sample preparation;

g) the particle size of the sample;

h) any other consideration (if necessary), for example any special purpose or test for which the sample is taken.

Annex A
(normative)

Principles of sample preparation

A.1 General

Sample preparation involves several distinct operations, which are sometimes preceded by drying. These are as follows:

a) reduction, i.e. to decrease the particle size by crushing, grinding, or pulverisation;

b) mixing;

c) division, i.e. to decrease the sample mass by dividing the sample into two or more parts.

These operations are generally considered to constitute one stage of sample preparation.

As a general rule, reduction should always precede division. An exception occurs where high capacity streams are being sampled mechanically; it is then permissible to divide large primary increments, as specified in ISO 11648-2:2001, Clause 10.

The stages of sample preparation should be chosen to minimise errors during sample preparation without having to retain too large a mass.

NOTE Examples of sample preparation schemes are given in Annex B.

All surfaces over which the sample passes should be constructed of abrasion-resistant material which will not be eroded in such a way as to contaminate the sample.

A.2 Minimum mass of sample to be retained after division

The procedure for sample preparation should involve two or more stages. The amount of sample to be retained at a given stage depends upon the nominal top size of the bulk material at that stage, and will be in accordance with the minimum mass of the gross sample, as determined by the method given in 7.1.

A.3 Drying

For samples other than those used for moisture determination, the sample may be air-dried or oven-dried.

NOTE A typical method for treatment of moisture samples is specified in ISO 11648-2:2001, Clause 20.

If the sample is wet or sticky, preliminary drying is often the first operation carried out in the first sample preparation stage. In this situation, preliminary drying (also called pre-drying) is necessary so that the sample will pass through the reduction and sample division equipment freely and without loss or contamination.

Drying may be carried out at any stage of sample preparation; e.g. drying prior to pulverisation. Drying is continued until the sample is sufficiently dry.

For some materials, it is necessary to dry at ambient temperature to avoid changes in the quality characteristic.

Materials that are susceptible to oxidation should be dried in an inert atmosphere, not at an excessive temperature.

No sample should be exposed to a temperature exceeding 105 °C.

The following methods may be used for air-drying a sample to ensure that it will pass through size reduction equipment and sample dividing equipment freely and without significant loss or contamination.

a) **Air-drying oven:** Pass heated air, not exceeding 40 °C, over the sample in an air-drying oven. Ensure the oven is able to make a complete change of air at least three times per hour, but at an air velocity which will not dislodge the sample from its tray.

   Place the sample in the oven on corrosion-resistant trays and spread it evenly in a layer of uniform thickness to a depth not exceeding the greater of:

   1) twice the nominal maximum particle size of the bulk material;

   2) 20 mm, except for lumps greater than this size.

b) **Drying floor:** Ensure that the drying floor is a smooth, clean surface, protected from direct sunshine, rain and excessive breeze.

   Spread the bulk material uniformly to a depth not exceeding twice its nominal maximum particle size. To aid drying, stir or rake the bulk material periodically, without loss of material.

A.4 Reduction of particle size

A.4.1 General

Shredders, granulators or pulverising equipment, referred to as “mills” in ISO 11648 (all parts), are used to reduce the nominal maximum particle size of the bulk material to a suitable level for subsequent division.

The purpose for which the sample is to be used will determine whether it is permissible to reduce the particle size during sample preparation. The several cases considered are as follows.

a) **Chemical analysis sample:** The bulk material will invariably require size reduction to meet the test requirements. The sample may be reduced as appropriate to facilitate the operations of division described in A.6.

   It is permissible, when handling samples intended for chemical analysis, to dry the material as received so that handling characteristics are improved. When drying is used, observe the following precautions.

   1) Avoid any contamination, oxidation or physical loss.

   2) Do not heat the sample to a temperature at which combined water or any other volatile component can be lost. Do not exceed a maximum temperature of 105 °C for any portion of the sample.

b) **Moisture sample:** Do not submit bulk material having a nominal top particle size of less than 10 mm to size reduction prior to moisture determination.

   When handling bulk material with a nominal top size of greater than 10 mm, and which is not adhesive or excessively wet, the bulk material may be reduced in particle size to less than 10 mm, care being taken to minimise any change in moisture level.

   When handling bulk material with a nominal top size of greater than 10 mm, and which is adhesive or excessively wet, the total sample may be weighed and air-dried. When it has dried to a free-flowing state, reweigh the sample. The sample may then be reduced in particle size prior to division and determination of residual moisture.

   When this procedure is followed, the final moisture value will take into account the moisture lost in the air drying stage.

c) **Physical testing sample:** Do not submit the sample to size reduction when samples are to be used for:

   1) determination of particle size distribution; or

   2) determination of bulk density.

   Feed the sample uniformly into size reduction equipment in such a way as to avoid choking or changes in drive speed, which can result in variation in the size distribution of the product.

   Errors of sample division and analysis are increased by the presence of oversize material. Therefore regularly check reduction equipment performance to ensure that the product meets the required nominal maximum particle size.

   During preparation of the chemical analysis sample, do not use screening to remove oversize particles for secondary size reduction. Material which is difficult to size reduce is usually different in composition from the remainder of the sample and cannot be satisfactorily mixed back into the sample.

A.4.2 Size reduction equipment

Particle size reduction equipment that may be used for particulate material samples includes shredders, granulators and pulverisers.

Factors which might influence the choice of equipment for any stage of sample preparation are the type of size reduction action of the mill and the requirements of the particular testing procedure.

Those parts of the apparatus that come into contact with the sample should be made of wear-resistant material, to minimise contamination. This is particularly important with samples in which trace elements are to be determined, every effort being made to use equipment which does not contain any of those elements.

Certain particle-size-reduction apparatus, such as high speed granulators and pulverisers, become heated, so avoid keeping samples in them long enough to become affected. If equipment is used for a series of samples, cool it between each operation.

High-speed granulators and pulverisers can be seriously damaged by the presence of hard extraneous material in the sample, so prevent such material from entering the equipment. A magnetic separator may be placed on the chute leading to the machine to safeguard against the entry of ferrous materials.

Although high-speed granulators and pulverisers are the most efficient for a wide range of materials, they generate dust owing to the fan-like effect of the rotating blades. To avoid loss of fine material, limit the volume of fresh air passing through the equipment (for example by using closed inlet or outlet hoppers with or without an air recycle tube, or by fitting a breather bag to the outlet).

Make sure all equipment is easy to clean and is cleaned between each sample.

A.5 Sample mixing

A.5.1 General

Errors in sample division can be reduced by thoroughly mixing the sample prior to division. The need for mixing is particularly important where samples from more than one source are combined. Some methods of mixing, for example forming and reforming into a conical pile, or some rotary methods of mixing, might have the opposite effect to that intended, leading to increased segregation. Consequently, the choice of the most appropriate method of mixing is critical, and depends on the nature of the bulk material.

Except where a moisture sample is to be taken, dry samples that are not free-flowing at a temperature not exceeding 105 °C before mixing.

Where samples are taken for moisture determination, carry out the mixing as quickly as possible to minimise changes in moisture content.

A.5.2 Methods of mixing

Mixing may be carried out using any of the following methods.

a) **Passing the sample through a riffle or rotary sample divider:** Pass the sample through a riffle or rotary sample divider at least three times in succession, and recombine the sample after each pass.

b) **Use of strip mixing:** The sample material is formed into a strip by careful distribution of the bulk material from a shovel. The length-to-width ratio of the strip shall be no less than 10:1. Take a complete cross-section of the ore strip randomly and spread it out to form a new strip. Take successive cross-sections randomly and spread them out on top of the preceding cross-section, layer upon layer, until the old strip has been converted into a new strip. Repeat the process of taking cross-sections and reforming a new strip twice.

c) **Use of a mechanical mixer:** See examples in ISO 11648-2:2001, Annex G.

A.6 Division

A.6.1 General

Sample division may be carried out by a variety of mechanical or manual methods. In all cases, collect the divided samples by taking and combining a large number of small increments. The minimum number of increments is 20.

Sample division may be carried out in as many passes as are needed so as to obtain the required divided sample mass; for example, when using a riffle, three passes are required to obtain a one-eighth fraction.

Wet material might not flow freely, or might adhere to contact surfaces. In such circumstances, dry the sample as described in A.3 before proceeding to sample division.

Examples of sample division apparatus are illustrated in Annex C. Where division is performed for the purpose of extracting moisture samples, the preferred methods are mechanical division (see A.6.2) or increment division [see A.6.3 a)].

A.6.2 Mechanical sample division

The main advantage of mechanical sample division is that the divided sample is obtained by taking a much larger number of increments than is generally obtainable manually. The minimum number of increments is 20.

The design criteria for acceptable mechanical sample dividers follow the same general design principles as for mechanical sample cutters (see ISO 11648-2:2001, Clause 13). Although cross-stream cutters are used, rotary dividers are more common in sample preparation.

Examples of acceptable mechanical rotary sample dividers are as follows.

- **Rotating cone (Figure A.1)**: The machine consists of a feed hopper, a low-speed slotted rotating cone, a reject chute and a sample pipe. Bulk material is allowed to flow from the feed hopper onto the rotating cone which diverts it into the reject chute. The slot in the cone allows the bulk material to fall directly into the sample pipe for part of each revolution.
- **Rotary sample divider (Figure A.2)**: The machine comprises a number of sector-shaped canisters positioned on a platform, and a feeding device. The uniform bulk material stream flows to the hopper spout and, by relative rotation of these two components, the flow is intercepted by the top edge of the sector-shaped canisters, dividing the sample into representative parts.

- **Rotating chute (Figure A.3)**: A hollow shaft, to which is attached one or more cutters, rotates in an offset cone-shaped housing. A feed pipe is positioned above the rotating cutter. As bulk material falls through the feed pipe, the cutter intersects the stream and diverts an increment through the hollow shaft.

The procedure for dividing by rotary division is as follows.

a) Place the sample into the hopper of the rotary divider, ensuring that the opening at the bottom of the hopper is large enough to prevent bridging.

b) Ensure that the opening of the segment or cutter lips at any point where it intersects the bulk material stream is a minimum of three times the nominal top size of the material.

c) Ensure that the speed of the divider at any point where it intersects the bulk material stream is constant and not more than 0,6 m/s.

d) Activate the cutter prior to commencing the feed of the bulk material.

e) Allow a minimum of 20 passes of the receiving segment or cutter during the bulk material feed.

f) Continue rotation of the cutter until the bulk material feed has been completed.

Other types of mechanical sample dividers may be used, provided that they conform to the requirements of ISO 11648 (all parts) and do not introduce bias.

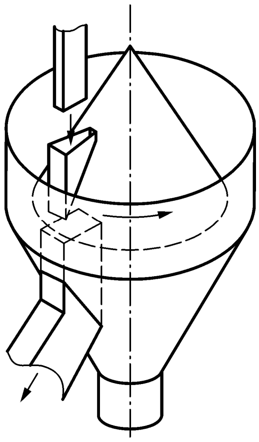
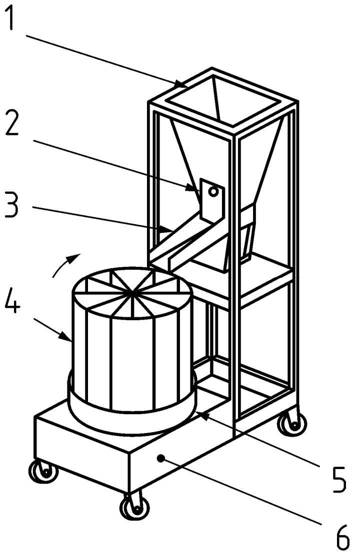


Figure A.1 — Rotating cone



Key

1	feed hopper
2	slide gate
3	vibratory feeder
4	removable canisters
5	turntable
6	drive (enclosed)

Figure A.2 — Rotary sample divider

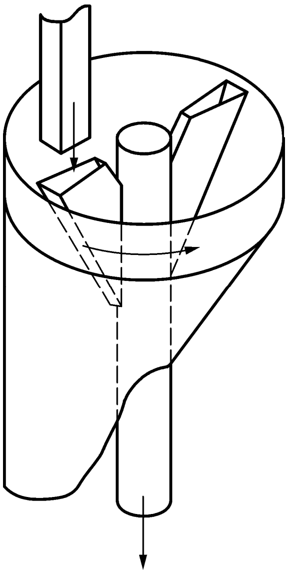


Figure A.3 — Rotating chute

A.6.3 Manual sample division

Sample division may be carried out manually using one of the following procedures.

a) **Increment division,** is carried out as follows.

   1) Mix the bulk material thoroughly and spread on a flat plate in the form of a rectangle of uniform thickness, as given in Table A.1.

Table A.1 — Cutter having cutter lips normal to the cutter path

Nominal maximum particle size mm	**Minimum mass of increment** kg	**Thickness of flattened sample** mm
≤ 11,2	Masses need to be determined; refer to ISO 11648-2:2001, Clause 9.	30 to 35
16,0		40 to 50
22,4		55 to 65
31,5		80 to 90
45,0		110 to 120

   2) Mark a matrix on the spread sample (see Figure A.4) with a minimum of 20 parts.

      The matrix should be designed in conjunction with Table A.1 to give the total amount of sample required for subsequent analysis. The ratio of the length to the width should never be more than 5:4.

   3) Collect one increment of mass given in Table A.1 from each part of the matrix with an appropriate flat bottomed scoop.

   4) Insert a flat bump plate vertically through the spread material until it comes into contact with the mixing plate. Then insert the scoop to the bottom of the spread material and take the increment by moving the scoop horizontally until its open end comes into contact with the bump plate, ensuring that all particles of the material are collected off the top of the mixing plate.

   5) Lift the scoop and bump plate together so that material is prevented from falling from the open end of the scoop by the bump plate.

   6) If the mass of the combined increments is less than that determined in accordance with A.2, collect further complete sets of increments, as stated in 3), until the minimum mass is exceeded.

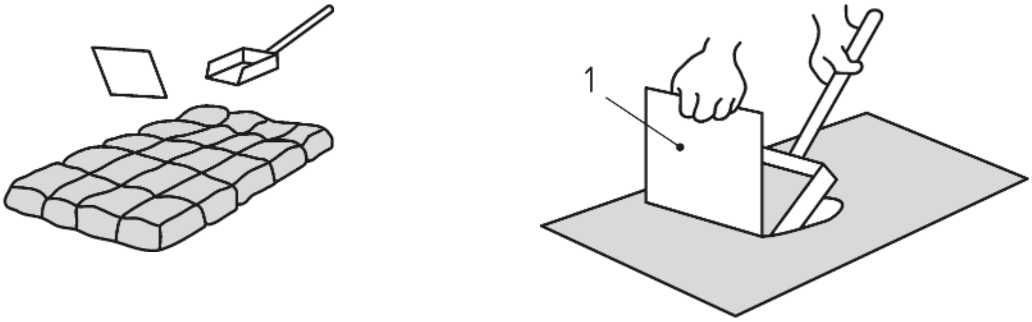
NOTE This method is regarded as a high-precision method in spite of the large division ratio; i.e. the ratio of the total sample mass to the retained sample mass. It is a recommended manual method for obtaining moisture samples.

a)

Spread the crushed gross sample into a rectangle with a thickness as specified in Table A.1.

b)

Arrange in 20 equal parts, e.g. into 5 equal parts length wise and 4 equal parts breadth wise.



c)

Take a scoopful of sample at random from each of the 20 parts by inserting the scoop to the bottom of the sample layer and combine the 20 scoopfuls of sample into a divided sample.

d)

Outline of taking an increment by using a bump plate shown in c).

Key

1 bump plate

NOTE Example for arranging into 20 parts.

Figure A.4 — Manual increment division

b) **Riffling:** A riffle is a sample divider which is used to divide the bulk material fed onto it into halves, one being retained and the other rejected. It operates by allowing the material to fall through a set of parallel slots of uniform width, adjacent slots feeding opposite containers.

A riffle is symmetrical (so that a part-sample can be taken from either side) and all surfaces on which bulk material can rest should be inclined at no more than 30° to the vertical. Fit receivers closely against the body of the riffle, to minimise loss of dust. It is essential that the riffle used is appropriate to the nominal top size of the material to be divided, as serious errors might be introduced if the slots are too small or if there are too few slots.

The slot width shall be at least twice the nominal top size of the bulk material. There shall be at least eight slots for each half of the riffle.

Carry out the riffling as follows:

1) Mix the material and place it in a feed container.

2) Spread the material in the feed container so as to spread it along the full length of the container.

3) Tip the container to feed the material uniformly into the feed chute, so as to pass the material through the riffle and collect it in two canisters.

   If the material chokes the riffle, clear it before the operation is continued. In this case, air-drying might be necessary.

4) Retain the sample from one of the receiving canisters, chosen at random.

   If further subdivision is required, the retained sample may be passed through the riffle again. For successive passes, the retained sample should be taken from alternate sides.

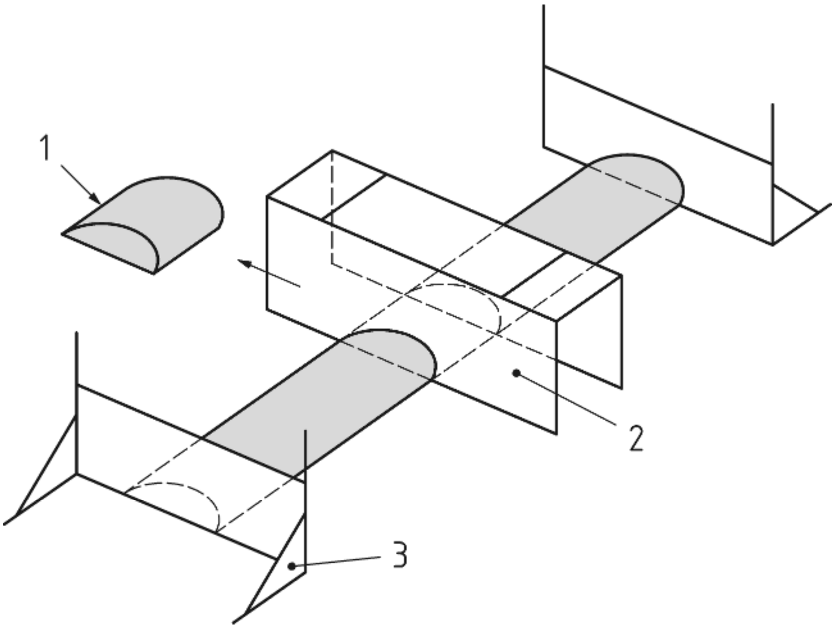
   Take care to minimise loss of moisture from a sample when carrying out division using a riffle. Use closed riffles for dividing moisture samples, or for dry bulk material to prevent loss of dust. Use gated riffles when dividing small sample masses.

c) **Strip mixing and splitting:** Strip mixing and splitting simulates the sampling of bulk material from a conveyor belt. Carry out strip mixing and splitting as follows.

   1) Form the material from a pile into a strip by careful distribution of the material from a shovel along the length of the strip as evenly as possible, working from end to end of the strip and from both sides of the strip. The length-to-width ratio of the strip shall be no less than 10:1. The general appearance of a completed strip is given in Figure A.5. The end plates ensure that size segregation only occurs laterally.

   2) Take the sub-sample by inserting a suitable sampling frame across the strip and removing the material from between the frame using a scoop, ensuring that all fine particles are included in the increment. Take the number of increments required (minimum 20).

   This procedure is not recommended for obtaining a moisture sample.



Key

1 increment

2 sampling frame

3 end-plates ("book-ends")

Figure A.5 — Completed strip

d) **Fractional shovelling** (see Figure A.6): The procedure for division by fractional shovelling is as follows.

1) Mix the material and form a heap on a smooth clean surface.

2) Using an appropriate shovel take successive shovelfuls from the base of the heap, working around the base.

3) Place each successive shovelful on separate successive heaps, the number of heaps being determined by the division ratio. For example, if a 1 in 5 division ratio is required, five heaps, N_1 to N_5 are formed as shown in Figure A.6. Ensure that at least 20 shovelfuls are placed on each heap.

4) Select at random the heap to be retained.

This division process may be used for bulk material having a nominal top size up to 45 mm.

This method is not recommended for obtaining a moisture sample.

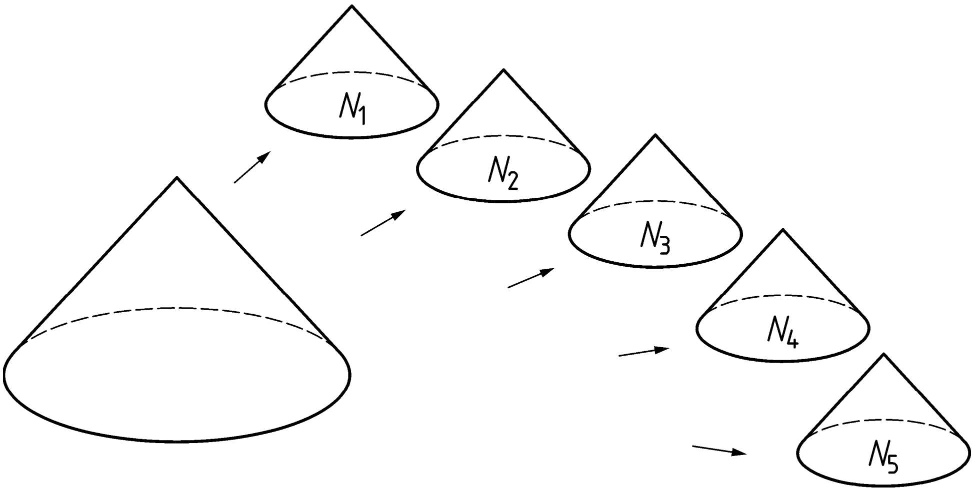
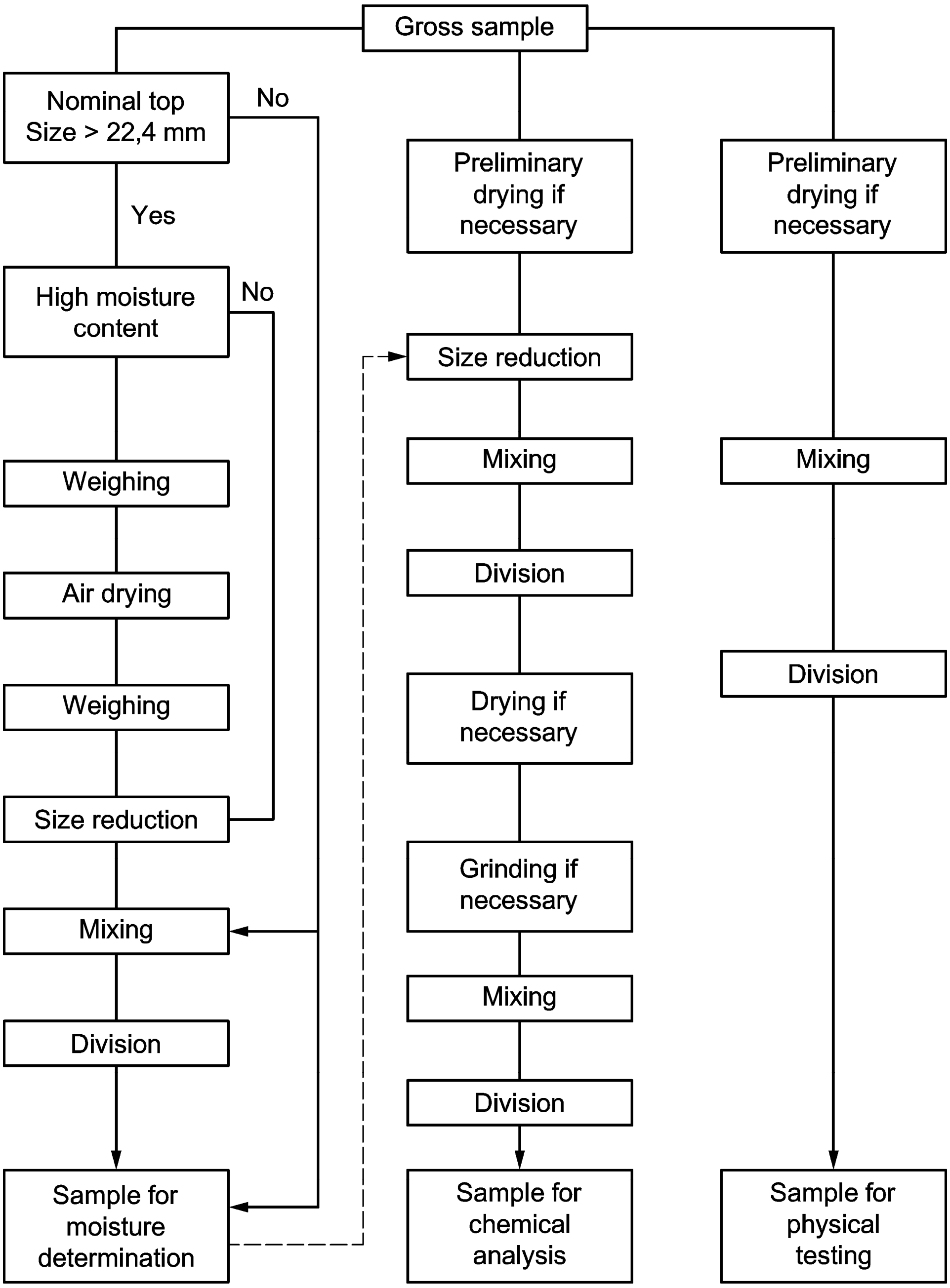


Figure A.6 — Sampling by fractional shovelling

Annex B
(informative)

Sample preparation schemes

Schemes for sample preparation are given in Figures B.1 and B.2.



Sample used for moisture determination may be used for chemical analysis if desired.

Figure B.1 — Sample preparation from a gross sample

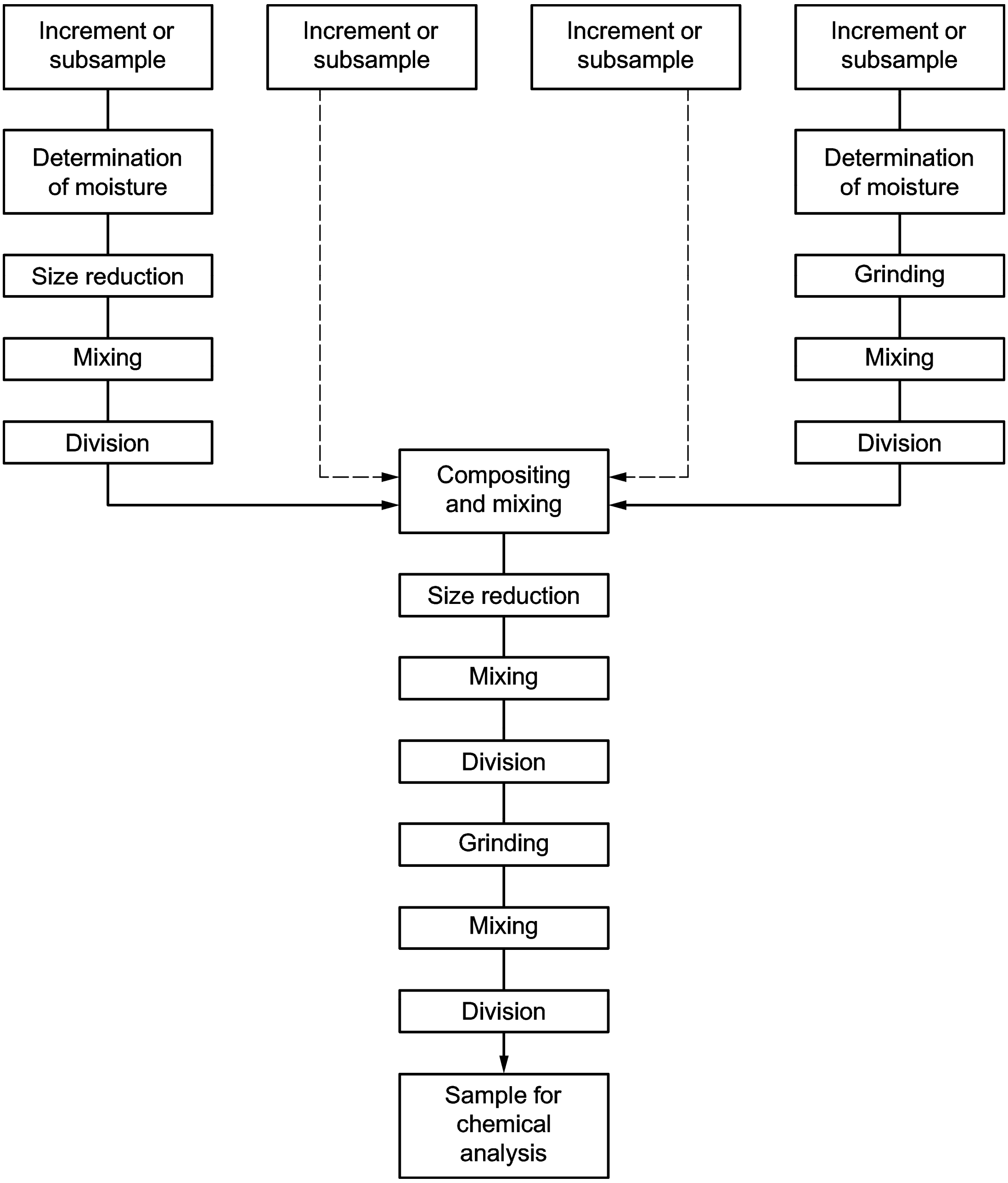


Figure B.2 — Sample preparation from increments or sub-lot samples

Bibliography

[1] EN 15342, *Plastics — Recycled Plastics — Characterization of polystyrene (PS) recyclates*

[2] EN 15343, *Plastics — Recycled Plastics — Plastics recycling traceability and assessment of conformity and recycled content*

[3] EN 15344, *Plastics — Recycled Plastics — Characterisation of Polyethylene (PE) recyclates*

[4] EN 15345, *Plastics — Recycled Plastics — Characterisation of Polypropylene (PP) recyclates*

[5] EN 15346, *Plastics — Recycled plastics — Characterisation of poly(vinyl chloride) (PVC) recyclates*

[6] EN 15347, *Plastics — Recycled Plastics — Characterisation of plastics wastes*

[7] EN 15348, *Plastics — Recycled plastics — Characterization of poly(ethylene terephthalate) (PET) recyclates*

[8] ISO/IEC Guide 98, *Guide to the expression of uncertainty in measurement (GUM)*

[9] ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

[10] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[11] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[12] ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

[13] ISO 5725-4, *Accuracy (trueness and precision) of measurement methods and results — Part 4: Basic methods for the determination of the trueness of a standard measurement method*

[14] ISO 5725-6, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

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