

Methods for

Sampling chemical products —

Part 4: Sampling of solids

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- British Tar Industry Association*
- Chemical Industries Association*
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- Cement and Concrete Association
- Department of Trade and Industry, Warren Spring Laboratory
- Process Plant Association
- Individual experts

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 43 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Amendments issued since publication

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Contents

	Page
Co-operating organizations	Inside front cover
Section 1. General	
1 Scope	1
2 References	1
3 General considerations	1
Section 2. Objectives of sampling	
4 General	1
Section 3. Sampling plan	
5 Introduction	1
6 Estimation of the mean value of a characteristic	1
7 Estimation of the variability of a characteristic	4
8 Estimation of what proportion of material is defective	5
Section 4. Sampling techniques	
9 Introduction	5
10 Sampling of powder, granules and small crystals	6
11 Sampling of coarse or lumpy materials	9
12 Sampling of massive pieces	10
13 Sampling of plastic materials	10
Section 5. Sample quantity reduction	
14 Introduction	11
15 Preparing the sample reduction plan	11
16 Particle size reduction techniques	12
17 Mixing techniques	13
18 Sampling subdivision techniques	13
Section 6. Sampling solids that require special handling	
19 Introduction	15
20 Solids that react with the environment	16
21 Reactive or unstable solids	16
Appendix A Sampling spears	17
Appendix B Sampling augers	24
Appendix C Pneumatic and vacuum probes	25
Appendix D Sampling drills	25
Appendix E Automatic samplers	28
Appendix F Representative sampling devices and sample division	32
Appendix G Miscellaneous equipment	40
Figure 1 — Cross-sectional samples of a falling stream	7
Figure 2 — Sampling plate	12
Figure 3 — Punnets	13
Figure 4 — Diagram showing coning and quartering process	14
Figure 5 — Open-end sampling spear (typical dimensions)	18
Figure 6 — Closed-end sampling spear (typical dimensions)	19
Figure 7 — Open-end sampling spears for use with plastic solids	19
Figure 8 — Further designs of open-end sampling spears	20
Figure 9 — Closable sampling spear	21
Figure 10 — Shuttered sampling spear	22
Figure 11 — Tubular sampling spear	23
Figure 12 — Typical grain sampling spear	23
Figure 13 — Simple auger	24

	Page
Figure 14 — Shuttered sampling auger	24
Figure 15 — Typical pneumatic probe sampler	26
Figure 16 — Typical suction sampling device	27
Figure 17 — Section through typical spear for suction sampling device	27
Figure 18 — Cutting bits for sampling drill	28
Figure 19 — Intermittent automatic sampler	29
Figure 20 — Continuous automatic samplers	30
Figure 21 — Automatic sampler for belt conveyor	31
Figure 22 — Granular material sampler	35
Figure 23 — Typical riffle sample divider	36
Figure 24 — Rotary sample dividers	37
Figure 25 — Double cone sample divider	38
Figure 26 — Schematic drawing of moving flap sample divider	39
Figure 27 — Grid type sample divider	40
Figure 28 — Grid type sample divider: arrangement of grid	41
Figure 29 — Grid type sample divider: views of grid	42
Figure 30 — Diagram of light probe and associated equipment	43
Table 1 — Relation of sample quantity to particle size	14

Section 1. General

1 Scope

This Part of this British Standard specifies methods for the sampling of solid chemical products in the form of particles varying from powders to coarse lumps, massive pieces and plastic solids.

It does not cover solids conveyed by gases, nor the sampling of slurries which is dealt with in Part 3.

2 References

The titles of the British Standards referred to in this standard are listed on the inside back cover.

3 General considerations

The development of a sampling procedure follows from the discussion in section 4 of Part 1:1976. The four main stages are as follows.

- a) Define the objectives of sampling.
- b) Prepare the sampling plan.
- c) Take the sample using the appropriate technique.
- d) Reduce the quantity of sample.

Section 2. Objectives of sampling

4 General

There are several possible objectives of sampling which can be grouped under three main headings as follows.

- a) To estimate the mean value of a characteristic.
- b) To estimate the variability of a characteristic.
- c) To estimate what proportion of material is defective.

Section 3. Sampling plan

5 Introduction

The sampling plan is designed to provide test samples from which some characteristic of the solid may be determined, with known and controlled errors of sampling, and to provide this control at the lowest possible cost.

The main factors which determine the sampling plan are the defined objective of sampling, the nature of the variability and the acceptable error. At the same time the method of containing or transporting the solid, i.e. single or multiple units or streams, should be taken into account.

Sometimes it may not be possible to use the ideal plan because the material to be sampled requires special handling. Thus a greater sampling error than desired may have to be accepted in order to have a workable plan.

6 Estimation of the mean value of a characteristic

The sample required to meet this objective is a representative sample that has been produced in such a way as to conform to a specified sampling error risk. Before such a sample can be produced from a bulk of solid the nature of the variability of the bulk has to be found or assumed.

6.1 Deciding the optimum sample quantity.

The optimum sample quantity is the smallest sample capable of giving the necessary information about the material it represents. The general considerations applying to the selection of the optimum sample quantity for any lot are given in clause 15 of Part 1:1976 of this standard.

Many test techniques require that the tester takes a finely divided portion of the sample for examination. It is necessary that optimum sample quantity considerations are applied to the portion used for testing. Since the particle size of this portion is generally specified in the test, this results in a requirement that the sample preparation step shall lead to the whole of the sample being reduced to particles of less than a certain size.

The efficacy of the preparation step should be measured by performing duplicate determinations on a number of prepared samples and comparing the duplicate results.

In all cases the optimum sample quantity should be such that sufficient material is taken to meet the needs of the intended tests. Unless there are other adequate reasons for restricting the quantity of samples the optimum sample quantity should provide sufficient material for efficient reduction of particle size and blending and to produce the final sample.

6.2 Material contained in a single item. With the exception of 6.2.2 the following recommendations are for the cases where, because the appropriate conditions (see clause 14 of Part 1:1976) cannot be fulfilled, a representative sample cannot be taken directly.

6.2.1 To find the nature of the variability. Examine the material for any obvious signs of variability, e.g. large variations in particle size, difference in colour, etc. (see **G.2**). Consider the history of the material, i.e. how it was made, how it was handled, etc. Confirm any apparent or suspected variability by testing spot samples of optimum sample quantity (see clause **15** of Part 1:1976) taken from the appropriate parts of the bulk.

For a material that is being regularly received it is probably only necessary to investigate the nature of the variability of the first few consignments. Thereafter a brief inspection of each consignment to detect whether there is anything abnormal should suffice. It also helps if it is known when any changes are made in the production or handling methods. A check can then be carried out to confirm that the nature of the variability has not altered.

The different kinds of variability that may be met with are described in clause **13** of Part 1:1976. Means of obtaining a representative sample, once the type of variability has been identified, are indicated in **6.2.2** to **6.2.6**.

6.2.2 Homogeneous material. Take a spot sample of at least optimum quantity from anywhere in the bulk that is convenient. This is representative of the whole (see **13.2** of Part 1:1976).

6.2.3 Random heterogeneous material. Take a number of spot samples of equal and at least optimum quantity from convenient places in the whole. Combine all these spot samples to give a representative sample (see **13.3** of Part 1:1976).

The number of spot samples to be taken depends on the acceptable sampling error risk (see clause **19** of Part 1:1976). It may be convenient to take several spot samples in one sampling operation by taking a directional sample.

6.2.4 Directional and cyclic non-random heterogeneous material. If convenient to do so mix the material so that it exhibits random heterogeneity and then sample as described in **6.2.3**. Otherwise take a directional sample of at least optimum quantity along the entire length of the axis which exhibits the non-random heterogeneity. The amount of sample at various points along the axis should be proportional to the amount of material in the strata at these points.

If there is a big difference between material at one end of the axis of variability compared with the material along the remainder of the axis, take a spot sample of optimum quantity from the stratum which is extremely different and test this sample separately. Take a directional sample of at least optimum quantity along the remainder of the axis of variability and test this sample. Calculate a weighted mean value for the characteristics being measured according to the respective amounts of material in the strata from which the two samples were taken.

In some cases there may be axes of non-random heterogeneity in several different directions, e.g. when the solid is liquid or plastic at the time of filling into a container and takes up the shape of the container. Usually the best sample to take in these circumstances is a geometrical sample of at least optimum quantity and which embraces the multidirectional, non-random heterogeneity.

6.2.5 Mixed population non-random heterogeneous material. Generally sample as in **6.2.4**. However, this type of variability (see **13.3.3.3** of Part 1) sometimes manifests itself in a single item in the form of two or more distinct homogeneous strata. In such cases take a spot sample of greater than optimum quantity from each stratum and combine all these spot samples (in proportion to the amount of material they represent) to give a representative sample. The smallest portion taken from any spot sample shall be of at least optimum sample quantity.

6.2.6 Reduction of spot samples. If, in obtaining a representative sample (see **6.2.2** to **6.2.5**), it is necessary to combine spot samples it may be convenient to first reduce the total quantity of spot samples before combining. This operation is simply a repetition of obtaining a representative sample of at least optimum quantity from a single item; the single unit in this case being the spot sample. However, if the spot sample is of optimum quantity or only slightly greater, in order to take a small amount of material from the spot sample, the particle size should be reduced in order not to increase the sampling error risk (see clause **19** of Part 1:1976). If the particle size cannot be reduced combine the spot samples in the correct proportions, the smallest portion being of at least optimum quantity, well mix the combined samples and take a representative sample of at least optimum quantity from the mixed material to give the final sample.

NOTE The most satisfactory way of sampling large amounts of heterogeneous material is to sample from the material in motion as the item is filled or discharged (see **6.4**).

6.3 Material contained in multiple items. With the exception of **6.3.2** the following recommendations are for the cases where, because the appropriate conditions (see clause **14** of Part 1:1976) cannot be fulfilled, a representative sample cannot be taken directly.

6.3.1 To find the nature of the variability.

Examine the items for any obvious differences, e.g. different batch numbers, order of filling as indicated by a running number, etc. Examine the material in all the items for any obvious signs of variability both within and between units, e.g. large variations in particle size, differences in colour, etc. Consider the history of the material, i.e. how it was made, how it was handled, etc.

Confirm any apparent or suspected variability by testing spot samples of greater than optimum sample quantity taken from the appropriate parts of the items.

For a material that is being regularly received it is probably only necessary to investigate the nature of the variability of the first few consignments. Thereafter a brief inspection of each consignment to detect whether there is anything abnormal should suffice. It also helps if it is known when any changes are made in production or handling methods. A check can then be carried out to confirm that the nature of the variability has not altered.

The different kinds of variability that may be met with are described in clause **13** of Part 1:1976. Means of obtaining a representative sample, once the type of variability has been identified, are indicated in **6.3.2** to **6.3.7**.

6.3.2 Homogenous material throughout the consignment. Take a spot sample of at least optimum quantity from anywhere in any item that is convenient. This is representative of the whole.

6.3.3 Homogeneous material within items, but random heterogeneous variability between items. Take a spot sample of greater than optimum quantity from each of a number of items which is convenient to handle. Combine the spot samples in amounts proportional to the amount of material in the items from which they were taken. The smallest portion shall be of at least optimum sample quantity.

The number of items to be sampled depends on the acceptable sampling error risk.

6.3.4 Homogeneous material within items, but directional non-random heterogeneous variability between items. This type of variability is unusual because it depends upon knowing how the items should be arranged to show non-random variability. However, if this is known it helps greatly in taking a representative sample.

Take spot samples of at least optimum quantity from items spaced at random unequal intervals in the order that gives the non-random variability. Combine the spot samples in portions proportional to the amount of material in the items from which they were taken. The smallest portion shall be of at least optimum sample quantity.

The number of items to be sampled depends on the acceptable sampling error risk.

6.3.5 Homogeneous material within items, but mixed population non-random heterogeneous variability between items. Generally sample as in **6.3.4**. However, it may be possible to arrange the items into two or more groups such that the material is homogeneous throughout any one group. In such cases sample each group as in **6.3.2** and combine all the spot samples so taken in proportion to the amount of material in the groups from which they were taken.

6.3.6 Heterogeneous material within items and between items. The various types of heterogeneity between items can obviously be combined with the same types of heterogeneity within an item. Thus instead of taking spot samples from items representative samples should be taken from an item as described in **6.2.3** to **6.2.5**.

6.3.7 Reduction of spot samples. Proceed as described in **6.2.6**.

NOTE The most satisfactory way of sampling large amounts of heterogeneous material is to sample from the material in motion as the item is filled or discharged (see **6.4**).

6.4 Material in motion. The amount of material for which the mean value of a characteristic is required should be defined either by a container or containers that are to be emptied or in terms of rate of flow and time.

With the exception of **6.4.2** the following recommendations are for the cases where, because the appropriate conditions (see clause **14** of Part 1:1976) cannot be fulfilled, a representative sample cannot be taken directly.

6.4.1 To find the nature of the variability. Material in motion can be considered to vary in two ways, i.e. across the direction of flow and along the direction of flow. In **6.4.2** to **6.4.5** only the variability along the direction of flow is considered. Variability across the direction of flow should have been eliminated by the technique used to take the sample, i.e. by always taking the full cross section of the stream for the sample.

It is impossible to make a preliminary examination of material actually in motion in order to decide the nature of the variability before taking a representative sample. Thus, for material of unknown history the samples taken to determine the nature of the variability are also used for determining the mean value of the required characteristic.

In some cases it may be possible to examine the material whilst stationary. However, it should be remembered that the method of subsequently moving the material may create a different variability from that shown by the material when it is stationary.

6.4.2 Homogeneous material. Take a cross-sectional sample of at least optimum quantity from anywhere in the stream that is convenient. This is representative of the whole of the material (see **13.2** of Part 1:1976).

6.4.3 Random heterogeneous material. Take a number of cross-sectional samples of equal and at least optimum quantity from a convenient place in the stream and at convenient intervals of time. Combine all these cross-sectional samples to give a representative sample (see **13.3** of Part 1:1976).

The number of cross-sectional samples to be taken depends on the acceptable sampling error risk.

6.4.4 Directional and mixed population non-random heterogeneous material. Take cross-sectional samples of greater than optimum sample quantity from a convenient place in the stream and at equal intervals of time. Note the rate of flow of material past the sampling point at the time of sampling. Combine all the cross-sectional samples in proportion to the respective rates of flow to give a representative sample, the smallest portion being of at least optimum sample quantity.

The time interval between each cross-sectional sample depends on the acceptable sampling error risk.

6.4.5 Cyclic non-random heterogeneous material. Take cross-sectional samples of greater than optimum sample quantity from a convenient place in the stream and at random intervals of time. Note the rate of flow of material past the sampling point at the time of sampling. Combine all the cross-sectional samples in proportion to the respective rates of flow to give a representative sample, the smallest portion being of at least optimum sample quantity.

The number of cross-sectional samples to be taken depends on the acceptable sampling error risk.

6.4.6 Reduction of cross-sectional samples. Proceed as described in **6.2.6**.

7 Estimation of the variability of a characteristic

It is not necessary to know the exact nature of the variability in order to plan a sampling method. It is essential however, to decide upon and specify the sub-items between which variability is to be measured (see clause **11** of Part 1:1976).

The number of sub-items to be sampled and tested depends on the degree of confidence required in the assessment of variability and the cost of sampling and testing (see clause **11** of Part 1:1976).

7.1 Material contained in a single item. If the specified sub-item is small enough to be taken as a spot sample divide the item hypothetically into sub-items and take a number of these sub-items as spot samples, each of which is then treated as a laboratory sample.

If the sub-items are too large to be taken as spot samples take a representative sample from each of the selected sub-items. Treat each representative sample as a laboratory sample.

The sub-items for sampling should generally be selected at random in order to detect variability due to non-random heterogeneity (see **13.3.3** of Part 1:1976). If, however, the nature of the variability is known or suspected the sub-items should be selected in such a way as to detect the maximum variability.

7.2 Material contained in multiple items. If the specified sub-item is smaller in quantity than one of the multiple items the procedure described in **7.1** should be carried out by treating each of the multiple items as a single item.

If the specified sub-item is greater than one of the multiple items then single items forming the consignment should be hypothetically divided into batches approximately equal to, but no greater than, the specified sub-item. Select a number of batches and take a representative sample from each item of the selected batches (see **6.2**). Combine these item representative samples to produce a representative sample for each selected batch. Treat each representative sample taken as a laboratory sample.

The single items should generally be divided randomly into batches. If, however, there are indications of a particular type of non-random heterogeneity (e.g. batch numbers on sacks) the single items should be batched in such a way as to produce the maximum variability between batches.

7.3 Material in motion. Material in motion can be considered to vary in two ways, i.e. across the direction of flow and along the direction of flow. In this subclause only the variability along the direction of flow is considered. Variability across the direction of flow should have been eliminated by the technique used to take the sample, i.e. by always taking the full cross section of the stream for the sample; thus the sub-items between which variability is to be measured are not less than the smallest cross-sectional sample that can be taken.

If the nature of the variability is not known take cross-sectional samples, each equal in size to the specified sub-item, at random points in time. If the nature of the variability is known or suspected the times of taking samples should be selected so that samples will detect the maximum variability.

Treat each cross-sectional sample taken as a laboratory sample.

8 Estimation of what proportion of material is defective

In general the sampling plan considerations described in clauses 6 and 7 apply to the procedure for the estimation of the proportion of defective solid present.

Special consideration has to be given to a definition of defective material appropriate to a practicable sampling plan and to the total errors which may be encountered in sampling (and testing).

8.1 Definition of defective material. Defective material is defined by reference to an existing specification or one implied by the name of a material. The significance of specification limits and the possible sampling errors have to be considered in drawing up a practical definition (see 9.3 and 9.4 of Part 1:1976).

If a definition of defective material is not available the sampling plan required is that needed to establish the variability of a characteristic (see clause 11 of Part 1:1976).

8.2 Errors and the sampling plan. The sampling plan should be related to the importance of detecting defective material. If, for example, a solid is unsafe or unsatisfactory in other ways when a characteristic falls outside certain levels then the sampling (and testing) plan(s) should be designed to give the maximum practical chance of detecting defective solid. The possible error incurred in sampling and testing should be allowed for, in the sampling plan, in relation to the importance of detecting defective material.

Section 4. Sampling techniques

9 Introduction

This section is a guide to the known available techniques of sampling. There may be situations where it is possible to design special sampling equipment and this section is not meant to discourage such enterprise. However, it is emphasized that any such specialized equipment, particularly automatic sampling devices should be tested for bias (see clause 12 of Part 1:1976).

It is emphasized that care has to be taken to use sampling techniques that will not affect any of the properties of the material which are to be evaluated. Where the test to be applied depends upon the sample being received in the same condition as the material from which it was taken, then the sampling procedure should allow for this. Thus it may be necessary to submit a large sample to the laboratory for measurement of particle size but permissible to submit a much smaller one for chemical assay because it has been possible to reduce the size of the larger particles and therefore further divide the sample.

As has been seen from section 3 there are five types of sample that require separate techniques:

- spot sample;
- directional sample;
- representative sample;
- cross-sectional sample, applicable mainly to material in motion;
- geometrical sample, only applicable to a non-particulate mass.

The main factors which determine the type of equipment to be used are the particle size, the quantity of material involved and whether it is static or in motion. The sampling techniques have therefore been classified firstly under particle size:

- powder, granules and small crystals;
- coarse or lumpy materials;
- massive pieces;
- plastic materials.

and secondly:

- small amount of material;
- large amount of material;
- material in motion.

Supplementary sampling precautions for solids that require special handling are described in section 6.

General safety precautions to be followed by the person taking the sample are dealt with in section 6 of Part 1:1976. However, some of these precautions are repeated in this section where they are particularly applicable to the techniques being used.

10 Sampling of powder, granules and small crystals

The solids to which the following sampling methods apply are those in which the particle size for all practical purposes does not affect the sample size.

10.1 Small amount (bag, sack, keg, drum, tote bin or similar sized heap).

10.1.1 Spot sample. Dip a scoop of suitable size into the material at the required spot, withdraw, and level off the material so that there is none above the sides of the scoop.

If the required spot is not readily accessible push a suitable sampling spear (see Appendix A) through the material so that the bottom portion of the spear takes a sample from the required spot.

10.1.2 Directional sample. Push a suitable sampling spear or tube through the material in the required direction so that the full cross section is traversed. Take the whole of the material in the spear as the directional sample.

10.1.3 Representative sample. Use one of the techniques described in clause 18.

10.2 Large amount (hopper, silo or heap). If it is economical to do so a material may be rearranged so that it is the equivalent of a number of small heaps and sampled as such (see 10.1). Otherwise it is considered that only a small layer of material at the top surface is accessible for the techniques described in 10.1 and therefore other techniques have to be used.

10.2.1 Spot sample. Push a vacuum probe (see Appendix C) or suitable sampling auger (see Appendix B) through the material to the spot from where the sample is required and collect the material from this spot.

10.2.2 Directional sample. Push a vacuum probe or suitable sampling auger through the material in the required direction. As the probe passes through the material take a series of spot samples until the traverse is complete. Combine the spot samples to give the directional sample.

10.3 Material in motion. In general the best place to take a sample from material in motion in where the material is falling freely in a stream. However, as this is not always convenient methods are given for sampling from all the common means of moving this class of solids.

CAUTION. Never sample manually from a moving conveyor.

10.3.1 Sampling from a falling stream

10.3.1.1 Spot sample. Take a scoop of sufficient size to hold the required quantity of sample without spilling over. Put the scoop upside down in the desired part of the stream, turn right side up and collect the spot sample. Quickly remove the scoop in the direction which brings it soonest out of the stream.

Alternatively, use a scoop with a cover which can be removed when the scoop is in the required spot and can be replaced when sufficient sample has been collected.

Automatic devices to take similar spot samples are also available.

10.3.1.2 Cross-sectional sample. The method of taking such a sample depends on the size of the stream and the density of the material. The various cases that can arise and the technique of sampling are illustrated in Figure 1. For each case:

W is the width of the stream;

D is the depth of stream from front to rear;

w is the width of the scoop;

d is the length of the scoop;

V is the average rate of volume flow;

t is the time for a single traverse of the scoop in cases 2 and 3.

Thus $W \times D$ describes the cross-sectional area of the stream and $w \times d$ describes the cross-sectional area of the opening of the sampling scoop.

Case 1. W and D both small; w greater than W ; d greater than D .

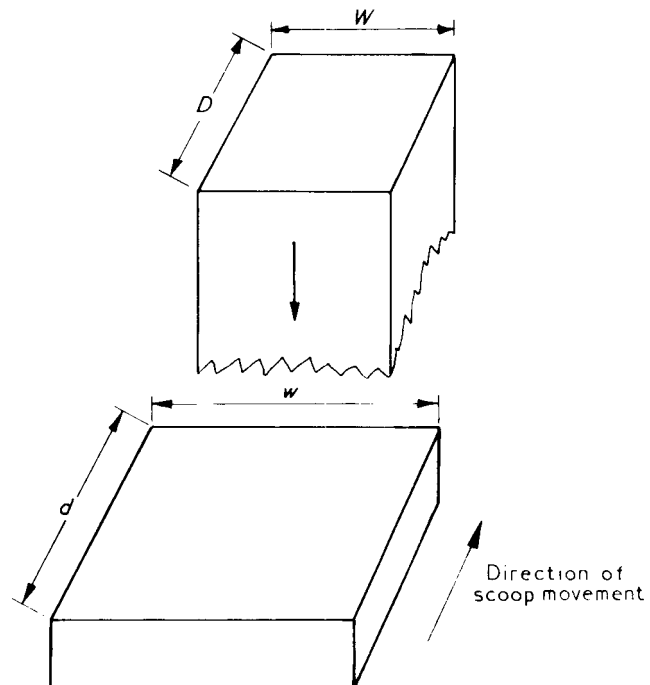
Position the scoop quickly in the stream so that it collects the full cross section. When the required amount of sample has been collected, quickly remove the scoop, continuing in the original direction of motion.

Case 2. W large, D small; d greater than D ; w at least 6 times the effective diameter of the largest particle.

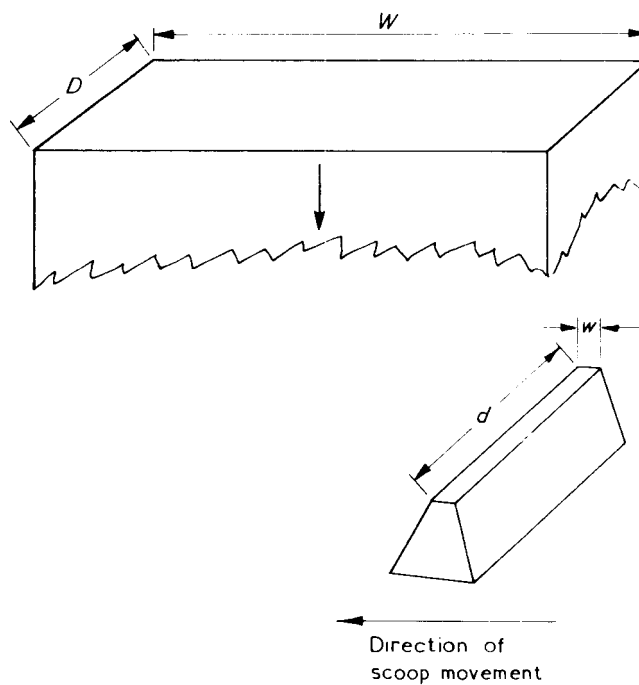
Move the scoop across the width of the stream at a uniform rate such that the required amount of material is collected.

The average volume of material entering the scoop is given by $\frac{Vtw}{W}$ and therefore the capacity of the scoop should be $2 \frac{Vtw}{W}$

The sample represents a volume Vt of the material.

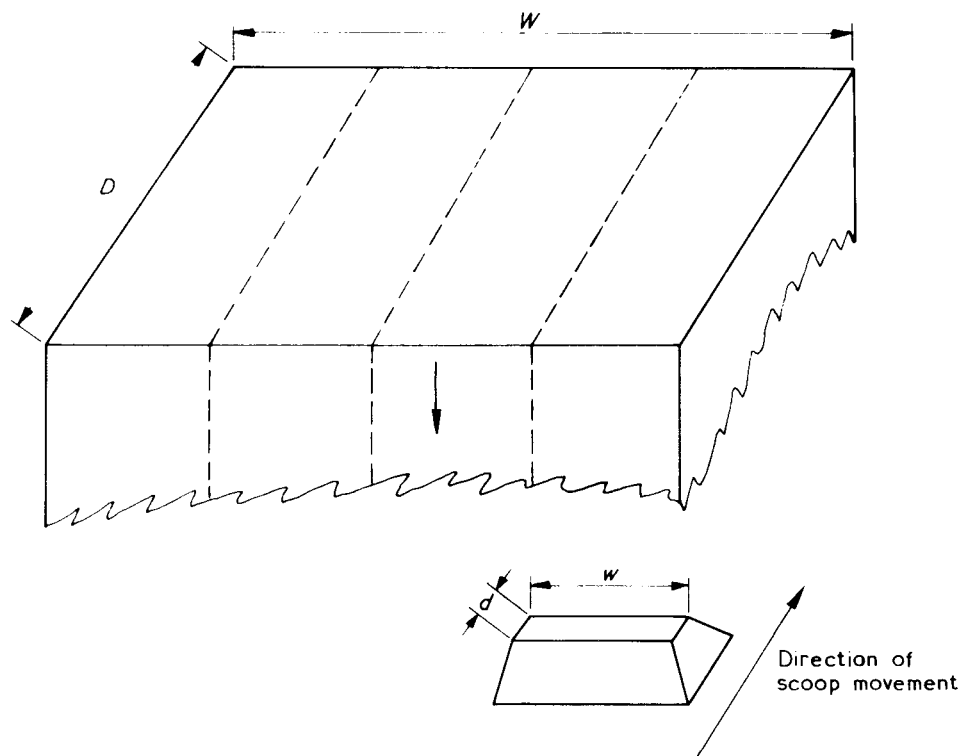


Case 1



Case 2

Figure 1 — Cross-sectional samples of a falling stream



Case 3

Figure 1 — Cross-sectional samples of a falling stream (concluded)

Case 3. W large, D large; $w = W/n$, where n is the number of imaginary sections of the stream; d at least 6 times the effective diameter of the largest particle.

Hypothetically divide the width or the depth of the stream into n sections. Move the scoop from front to rear of the stream (or vice versa) or from side to side for each section. Combine the samples of the sections to give the cross-sectional sample. The rate of movement of the scoop through the stream should be uniform through any section of the stream and should be the same for each section. The whole operation should be carried out as quickly as possible so that variation of the material with time is not included in the cross-sectional samples. The average volume¹⁾ of material entering the scoop is given by $\frac{Vtd}{nD}$ and therefore the capacity¹⁾ of the scoop should be $\frac{2Vtd}{nD}$. The average volume¹⁾ of the combined samples is given by $\frac{Vtd}{D}$ and this represents a volume Vt of the material.

It may be more convenient to reduce the quantity of section samples before combining (see section 5). It may be possible to use the technique employed in case 1 for cases 2 and 3 by mounting a large scoop of cross section greater than that of the stream on a suitable gantry. The scoop can then be swung into the stream to collect the full cross section as in case 1.

A example of an automatic sampler for taking cross-sectional samples is given in E.1.

10.3.1.3 Representative sample. Pass the whole of the stream through a device which will split off a small portion of the required size. The device should be such that every particle in the stream has an equal chance of being split off.

10.3.2 Sampling from an open or closed chute

10.3.2.1 Spot sample. Using a suitable scoop remove the required amount of material from the required spot. Obviously this cannot apply to a closed chute.

¹⁾ If the scoop is moved from side to side instead of from front to rear D will be replaced by W in these expressions.

10.3.2.2 Cross-sectional sample. Make a suitable closable aperture across the full width of the base of the chute and provide means of completely blocking and unblocking the full cross section of the chute. A convenient device is to make part of the base of the chute a hinged flap which can be moved upwards so as to block the cross section.

To take the sample place a receptacle under the aperture and open the aperture for a sufficient length of time to give the required amount of material.

10.3.2.3 Representative sample. Various devices can be fitted into a chute so that the whole flow of material passes through and a small portion is continuously split off and collected. The selected device should be such that every particle has an equal chance of being split off.

10.3.3 Sampling from a band conveyor

10.3.3.1 Spot sample. Stop the conveyor and, using a suitable scoop, take the required amount of material from the required spot.

10.3.3.2 Cross-sectional sample. Stop the conveyor and take a complete cross section of the material on the band by sweeping into the container the whole of the material lying between the sides of a suitable rectangular frame placed across the band. The frame should be inserted in the material until it is in contact with the band across its full width.

Alternatively, use an automatic sampler which sweeps material from the moving band into a container. The setting of such a device should be carefully adjusted to ensure that the whole thickness of the stream is taken, and that there is no loss of the material displaced by the cutting blade.

If many cross-sectional samples are required it may be convenient to install an automatic device which takes a cut across the band at preset time intervals. Such a device should be tested for bias (see clause 12 of Part 1:1976).

10.3.4 Troughed belt conveyor. Sample as described in 10.3.3 except that an automatic sampler cannot be used to obtain a cross-sectional sample across the width of the conveyor.

10.3.5 Screw conveyor

10.3.5.1 Cross-sectional sample. Make a suitable removable section in the base of the conveyor which is large enough for the full cross section of material to drop through when the section is removed. **Fit a chute of sufficient length so that the sampler cannot reach the moving screen with his hand.**

10.3.6 Fluidized conveyor. Shut off the air and treat as for a band conveyor (see 10.3.3).

10.3.7 Vibratory conveyor. Stop the vibration and treat as for a band conveyor (see 10.3.3).

11 Sampling of coarse or lumpy materials

The solids to which the following sampling methods apply are those in which the maximum effective diameter of any particle does not exceed approximately 100 mm. The size of particle affects the optimum sample quantity.

11.1 Small amount (bag, sack, keg, drum, tote bin or similar sized heap).

11.1.1 Spot sample. Dip a scoop of suitable size into the material at the required spot.

11.1.2 Directional sample. Carefully empty the material onto a clean surface. Using a straightedge board or piece of metal make a cut into the material in the required direction. Move the material on one side of the board away from the rest of the material. Make a parallel cut into the material that has not been moved and move all the material between two cuts away from the remainder to form the directional sample.

11.1.3 Representative sample. If the original state of the material does not have to be preserved reduce the particle size so that all the material can be thoroughly mixed and passed through a device which splits off a representative portion of the required amount (see section 5).

If the material contains a fairly large portion of particles which could be classified as granules, it may be preferred to separate this material, using a 4.75 mm sieve (complying with the requirements of BS 410) to reduce the particle size of the material retained on the sieve so that it all passes the same sieve, and then to re-mix all the material thoroughly before passing it through a sample reducing device.

11.2 Large amount (hopper, silo or heap)

11.2.1 Spot sample. Only the top surface is normally accessible and a suitable scoop can be used to take the sample from the required spot on the surface.

A sample from the interior may be obtained if a suitable scoop is suspended in the container at the required spot before filling. A suitable scoop can be made by cutting a hole in a tube of appropriate diameter and making a compartment by sealing off the tube below the hole. If a number of spot samples are required in one direction several such compartments can be made in the tube. The use of such a device is limited by the shape of the container and the position and size of the filling opening.

11.2.2 Directional sample. The device described in 11.2.1 for taking several spot samples should be used within the limits indicated and the several spot samples combined to give a directional sample.

11.3 Material in motion. It may be possible to use the same sampling techniques as for fine particles (see 10.3) if suitable apparatus can be mounted to handle the larger and heavier sample.

12 Sampling of massive pieces

12.1 Small amount. This may consist of a few massive pieces or a continuous mass occupying a small volume.

12.1.1 Spot sample. Take a lump from the required spot in the container and chip material from the required spot on the lump using a suitable tool.

If the lump cannot be chipped, drill or saw into it at the required spot and collect all the drillings or sawings as the sample. Avoid contamination due to wearing of the substance of which the drill or saw is made. If the material is heat sensitive, cool the drill or the saw with a suitable inert coolant.

If the material consists of a continuous mass treat it as described for a single lump.

12.1.2 Directional sample. This type of sample does not apply to a small container of several lumps but only to a single lump or a continuous mass.

Break the lump into two parts along the required direction using a hammer and chisel. Chip the two pieces so exposed and collect all the chippings as the directional sample.

If the mass cannot be chipped, drill or saw in the required direction and take all the drillings or sawings as the sample (see 12.1.1 for precautions to be observed).

12.1.3 Geometric sample. This type of sample only applies to a single lump or a continuous mass. Cut out the required shape and amount of material, using a hammer and chisel for material that breaks easily and a saw for material that does not. (See 12.1.1 for precautions to be observed.)

12.1.4 Representative sample. If the original state of the material does not have to be preserved crush all the massive pieces to a particle size such that all the crushed material can be thoroughly mixed and passed through a device which splits off a representative sample (see section 5).

If the material is to be dissolved in a solvent for use and its solution does not affect the tests to be carried out, sample the solution by the appropriate method described in Part 3.

12.2 Large amount. This may consist of a large number of massive pieces or a continuous mass occupying a large volume.

12.2.1 Spot sample. For a continuous mass occupying a large volume the same methods apply as for a small amount.

For a large number of massive particles only those near the surface of the bulk are readily accessible. Having selected a particle at the required spot near the surface take a spot sample by one of the methods already indicated for small amounts.

12.2.2 Directional sample. For a continuous mass, using a suitable chipping tool, chip a channel of required depth and width and in the required direction. Collect all the chippings as the directional sample.

If the mass cannot be chipped use the methods described in 12.1.2.

12.2.3 Geometrical sample. Proceed as described in 12.1.3.

12.3 Material in motion. It may be possible to use the same sampling techniques as for fine particles (see 10.3) if suitable apparatus can be mounted to handle the large and invariably heavy sample.

13 Sampling of plastic materials

The solids to which the following sampling methods apply are those which consist of a continuous soft mass. Sometimes the melting point of these solids may be near to or within the range of expected ambient temperatures and therefore they should be stored or transported in containers that are suitable for liquids. These materials should not be confused with the class of materials known collectively as “plastics materials” which are polymers, usually in the form of powders, granules or, less often, coarse lumps. For a plastic solid it is generally better to heat it until it is completely liquid, provided that heating does not affect the characteristics which are to be measured, and then to sample it as a liquid as described in Part 3 of this standard.

13.1 Small amount. This may consist of material in a keg or drum, or as a block, up to about 500 kg.

13.1.1 Spot sample. Dip a scoop of suitable size into the material at the required spot.

If the desired spot is not readily accessible push a suitable sampling spear of convenient size through the desired spot. A “stick” of plastic material is thus obtained from which the portion corresponding to the desired spot can be cut.

13.1.2 Directional sample. Proceed as described in 10.1.2.

13.1.3 Geometric sample. Cut out the required shape and amount of material using a suitable knife or cutting wire.

13.2 Large amount. Large amounts of this type of material are not usually encountered for sampling purposes as a solid since a large amount is usually handled as a liquid. However, if large amounts of a plastic solid have to be sampled as a solid the various types of sample should be taken as described in 13.1.1 to 13.1.3.

13.3 Material in motion. Sampling in motion is unlikely to be required for any plastic solid that is a finished product. However, during manufacture this type of solid is frequently extruded in the form of a long bar. In addition, a band conveyor may be used to cool a liquid so that it forms a moving slab of plastic solid.

CAUTION. Never sample manually from a moving conveyor.

13.3.1 *Extruded bar*

13.3.1.1 Spot sample. Cut or scoop out the required amount of material from the desired spot.

13.3.1.2 Cross-sectional sample. Cut out a piece of the extruded bar of desired length by cutting at right angles through the bar at two places with a suitable knife or cutting wire.

13.3.2 *Band conveyor*

13.3.2.1 Spot sample. Stop the band if the process permits and cut or scoop out the required amount of material at the desired spot.

13.3.2.2 Cross-sectional sample. Stop the band if the process permits and cut out the desired amount of material by cutting at right angles through the slab at two places with a suitable knife.

Section 5. Sample quantity reduction

14 Introduction

Invariably the quantity of primary sample obtained from the bulk of material is too great to use for testing and has to be reduced. That is, a representative sample has to be taken from the primary sample.

When taking a representative sample, there is an optimum sample quantity of sample for a given sampling error risk (see clause 15 in Part 1). This optimum quantity is related to the size of the individual particles. Therefore, in reducing the quantity of the primary sample it may be necessary to reduce the size of the individual particles in order to avoid an increase in the sampling error risk.

In the method used to reduce the primary sample all parts of it should ideally have the same chance of being included in the sub-sample. However, many samples are taken in the field and because of their bulk, it is impractical to effect reduction other than at the sampling location. This may impose limitations on the equipment which can be used and some of the techniques mentioned may not fulfil the above-mentioned ideal, e.g. coning and quartering is known to be very susceptible to bias.

Reduction of the primary sample may therefore involve the three operations of:

- a) particle size reduction;
- b) mixing;
- c) subdivision.

These three operations may need to be carried out successively several times until the desired quantity of laboratory sample is obtained.

15 Preparing the sample reduction plan

The sampling error risk should have already been specified for the taking of the primary sample (this risk should not be exceeded at any stage when reducing the sample) and the size of the sample required for test purposes should also be known. Thus the maximum allowable particle size in the final sample can be calculated.

Knowing the final particle size required, the final quantity of the sample, the quantity of the primary sample, the size of the particles in the primary sample and the nature of the solid, decide the number of reduction stages required and select the techniques for each reduction stage.