

Methods for

# Determination of particle size distribution —

Part 1: Guide to powder sampling

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# Committees responsible for this British Standard

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 British Ceramic Society  
 British Laboratory Ware Association  
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## Foreword

This Part of BS 3406, prepared under the direction of the General Mechanical Engineering Standards Committee, supersedes BS 3406-1:1961 which is withdrawn. This revision is Part 1 of a series in which the other Parts describe recommended methods for determining particle size distribution. This Part of BS 3406 is based on sampling methods which have come into more widespread use since BS 3406-1 was published in 1961. Current and proposed Parts of BS 3406 are as follows:

- *Part 1: Guide to powder sampling;*
- *Part 2: Recommendations for gravitational liquid sedimentation methods for powders and suspensions;*
- *Part 3: Air elutriation methods;*
- *Part 4: Optical microscope method;*
- *Part 5: Recommendations for electrical sensing zone method (the Coulter principle);*
- *Part 6: Recommendations for centrifugal liquid sedimentation methods for powders and suspensions;*
- *Part 7: Recommendations for single particle light interaction methods<sup>1)</sup>.*

Differences between this revision and the original BS 3406-1:1961 include:

- a) the introduction of:
  - 1) the scoop sampling method;
  - 2) the chute riffing method;
  - 3) further rotary sampling methods;
  - 4) methods for preparing small (0.5 mL) test portions;
  - 5) more comprehensive guidelines on collection of the gross sample;
  - 6) additional comments on precautions;
  - 7) clauses on preliminary examination and selection of methods;
  - 8) sampling efficiency data;
- b) the deletion of:
  - 1) the halving method;
  - 2) the turntable type method;
  - 3) the oscillating hopper type method;
  - 4) the grid type method.

Representative sampling of particulate material is an essential prerequisite for the determination of size distribution. The procedures described in this Part of BS 3406 have been selected to provide a test portion as representative of the laboratory sample as is possible. Guidelines on procedures for obtaining the gross and laboratory samples are also included.

This Part of BS 3406 is intended to be a practical guide to the representative subdivision of the laboratory sample. Other related British Standards, some of which include more theoretical detail, are BS 1017, BS 5309, BS 5660, BS 5661, BS 5662, BS 6000, BS 6001 and BS 6002.

**NOTE OF CAUTION** Some of these standards include a particle size reduction step in the sampling process, e.g. in the preparation of the sample for general analysis and for total moisture determination in BS 1017 and in BS 5309. This step should obviously be omitted when preparing samples for particle size distribution analysis.

BS 3406-1 is also of relevance to BS 1796 and to BS 4359.

<sup>1)</sup> In preparation

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

**Compliance with a British Standard does not of itself confer immunity from legal obligations.**

#### **Summary of pages**

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 26, an inside back cover 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.

# Section 1. General

## 0 Introduction

There are a number of stages in the application of sampling techniques to powders.

A gross sample is taken from a defined quantity of material. This sample is either one in a series of spot samples required to measure, for example, variability of the material through its bulk or is meant to be fully representative of the bulk material sampled.

In some cases the gross sample is inconveniently large to send to the laboratory for testing and has to be reduced in quantity. This sample reduction should produce a laboratory sample which is fully representative of the gross sample. When this reduction is unnecessary, the gross sample is the laboratory sample.

The laboratory sample is often required for a number of different tests of which particle size distribution analysis is only one, and it is therefore sometimes necessary to reduce the quantity of the laboratory sample to provide a test sample which is fully representative of the laboratory sample.

Finally, the quantity of sample which is used in its entirety for the appropriate test or observation, the test portion, has to be obtained by reduction of the test sample and should be fully representative of the test sample and thus of the original gross sample. In some cases the test portion is obtained directly from the laboratory sample.

In many cases the analyst making the particle size distribution measurement is presented with a laboratory sample from which the test portion(s) will eventually be obtained, and has no control over the taking of the gross or laboratory samples. Whilst this Part of BS 3406 is primarily written to recommend methods for the reduction of laboratory samples to a quantity suitable for a particle size distribution analysis, the analyst should be aware of the possible errors caused by inadequate gross and laboratory samples. Some guidelines and strategy for the collection of gross and laboratory samples are given in section 2 and they should be considered when assessing the adequacy of such samples.

Methods of sample subdivision described in this Part of BS 3406 range from the relatively simple and inexpensive scoop sampling through coning and quartering, and chute riffing, to divided stream and whole stream rotary sampling.

## 1 Scope

This Part of BS 3406 provides guidance on powder sampling. It recommends methods for the subdivision of gross or laboratory powder samples into test portions suitable for analysis by methods for the determination of the particle size distribution described in other Parts of BS 3406 and for particle characterization analyses described in other British Standards. Ways of obtaining very small test portions are described.

This Part of BS 3406 indicates points to look for in a preliminary examination of the sample to assist in the selection of the most appropriate method to subdivide the powder in question. Factors affecting this selection are considered and recommendations made to ensure that the integrity of the sample is maintained throughout the subdivision process.

Appendix A gives the results of investigations by several workers into the relative efficiencies of the sample dividing methods.

**NOTE** The titles of the publications referred to in this standard are listed on the inside back cover.

## 2 Definitions

For the purpose of this Part of BS 3406, the definitions given in BS 2955 apply together with the following.

### 2.1 gross sample

the sample obtained or prepared from the bulk material under the sampling plan from which subdivision for testing, reference or storage can be made

### 2.2 laboratory sample

the sample delivered to the laboratory

### 2.3 spot sample

a sample of specified quantity taken from a specified place in the bulk material, gross sample or laboratory sample, or at a specified place and time in a stream and representative of its own local environment

### 2.4 test sample

the sample prepared from the laboratory sample from which the test portions are withdrawn

**2.5**

**test portion**

the portion withdrawn from the test sample and entirely used in the test or observation

**2.6**

**heterogeneous material**

material in which a spot sample will have a significantly different value of the characteristic under consideration from the mean value of the characteristic for the whole material

**2.7**

**homogeneous material**

material in which a spot sample will not have a significantly different value of the characteristic under consideration from the mean value of the characteristic for the whole material



## Section 2. Collection of gross and laboratory samples

### 3 Guidelines

The purpose of sampling is to obtain a sample or samples of the bulk material which is representative of that material in some particular property. To ensure accuracy, the sampling operation has to be free of systematic errors; for precision, the random errors should have small variation about the mean. A suitable theoretical model for the sampling operation is that in which the particles forming the sample have been chosen one by one and at random from a homogeneously distributed bulk. A well-designed sampling procedure is one in which both theoretical and practical errors arising from differences between the sampling operation and the model have been minimized.

Generally the size of sample to be taken will be given in a specification or determined by the requirements of any subsequent analysis. However, there is a minimum size of sample which needs to be taken, due to the discrete nature of the material, even if all other errors are zero. This arises because even in a well-mixed material the various particle sizes will not be uniformly distributed throughout the bulk. The coarsest size class will, generally, contain the fewest particles and consequently will have the greatest coefficient of variation. A sample mass which gives the required precision for the coarsest size class will then satisfy the requirements of the other classes. See Appendix B for a graphical relationship between the minimum sample mass and the mass fraction of the coarsest size class and the required precision. For example, for powders with a largest size class of about 1 mm and material density of 2.5 g/cm<sup>3</sup>, a minimum sample of 4.4 g would be required if the largest size class formed 3 % ( $m/m$ )<sup>2)</sup> of the bulk and was to be determined with a coefficient of variation of 0.1. The corresponding figure for a largest size class of 100 µm would be 4.4 mg. For finer powders any practical sample mass would be adequate.

Since the bulk material will rarely be homogeneous in the characteristics to be tested, it is often required that a measurement of this variability be made. It is then necessary to take several spot samples, the number and size of which will depend on the quantity and particle characteristics of the material. The variability can be assessed by the variance or by the range of the characteristic, derived from these spot samples. The mean value of the characteristic is an estimate of the mean value of the bulk, with a precision defined by the variability.

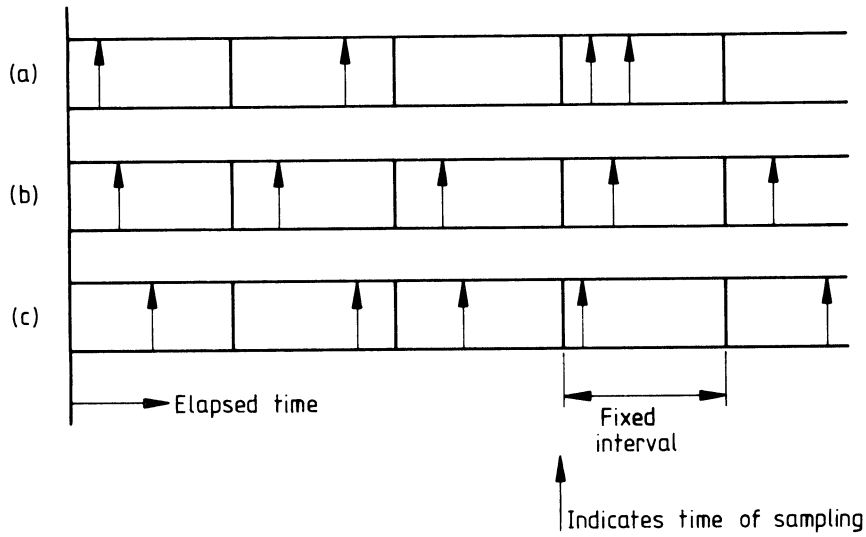
The best practical method to obtain a gross or laboratory sample which is representative of the bulk material is to sample a flowing stream of the material. The sampling errors can be minimized by maintaining a constant rate of flow and by taking a large number of increments to form the sample. The increments have to be taken over the full cross section of the stream. Care should be taken that no mechanism is in operation which preferentially accepts or rejects particles of certain sizes, and that no loss of sample or gain of adventitious material occurs.

### 4 Sampling strategy

If the material is known by experience to be homogeneously distributed in the bulk then simple random sampling [see Figure 1(a)] is sufficient. In many cases, the spatial distribution of the material particles will not be homogeneous and systematic sampling [see Figure 1(b)] is to be preferred unless there are cyclic variations of bulk quality. Such variations can lead to imprecise results. To guard against this possibility a random stratified sampling strategy [see Figure 1(c)] is recommended. Using this strategy, in a continuous delivery operation a sample increment is taken at a random time within each of successive fixed time intervals (strata) spanning the whole process. For static bulks the strata could consist of wagon-loads, bags or a number of bags. Some suggestions for sampling procedures have, for example, been described in BS 1017 and BS 5309-4.

The strategies are illustrated in Figure 1, drawn for a continuous delivery process.

<sup>2)</sup> Percentage by mass.



- (a) Random sampling in which the times of sampling are determined by chance
- (b) Systematic sampling in which the interval between samples is equal to the fixed interval or "stratum"
- (c) Random stratified sampling in which a sample is taken within each interval, but the timing within that interval is determined only by chance

**Figure 1 — Sampling strategies**

## Section 3. Selection of a sampling method for subdivision of the laboratory sample

### 5 General

The objective in the selection of a specific sampling technique is to obtain from the laboratory sample a test sample or test portion that represents as closely as possible the laboratory sample. Sampling is based on the premise that there is a theoretical possibility of dividing a sample into smaller samples of portions without the introduction of biased errors. The collection of data from every sample or position in a sample is expected to embrace the mean value of a characteristic of the sample. It also has to permit an estimation to be made of any variation of the measured characteristic throughout the sample.

Four methods for subdividing powders are described in section 4 (special methods for producing very small test portions are described in section 5). The choice of method will depend mainly on:

- a) the homogeneity or tendency to segregate of the powder (see clause 6);
- b) the flow characteristics of the powder (see clause 7);
- c) the acceptable accuracy of subdivision.

A number of other considerations have to be taken into account; for example, the properties of the powder to be sampled may require that special material are used to construct any apparatus (see 8.3).

In many cases previous experience will be a sufficient guide to selecting an appropriate method but in other cases all the factors in a) to c) should be considered. It is recommended that the efficiency of the chosen sampling method is checked.

### 6 Homogeneity

#### 6.1 General

In any homogeneous sample, any aliquot or subdivision of the sample will precisely represent the sample as long as the sampling technique does not impart any systematic bias to the subdivision procedure.

When a heterogeneous mix occurs on subdivision, a concentration of certain sizes or characteristics can occur in local positions. The sampling technique should therefore prevent or overcome these local concentrations.

#### 6.2 Homogeneous material

When a laboratory sample is known to be homogeneous, any practical volume selected (see clauses 3 and 9) will constitute a perfect sample. With such a homogeneously mixed material the technique of scoop sampling could be used. To ascertain if the laboratory sample is homogeneously mixed it has to be established that the value of the characteristic for each subdivision agrees within close limits with the mean value of the characteristic for all subdivisions.

To verify homogeneity a number of samples or portions should be chosen at random and tested. In reality no material can be deemed to be homogeneously mixed unless sampled and verified.

To assume a homogeneous mix prior to sampling and then to use the method of scoop sampling can only be justified if from past experience the assumption of a homogeneous material is correct and scoop sampling (or coning and quartering) gives results comparable with more rigorous sampling methods.

#### 6.3 Heterogeneous material

It is advisable to assume that all laboratory samples, unless known to the contrary, are heterogeneous. It is therefore necessary either to render the laboratory sample homogeneous by mixing before further subdivision as in 6.2 or to use a sampling technique which produces subdivisions all of which are representative of the laboratory sample.

### 7 Flow characteristics

#### 7.1 General

The flow properties of a powder may be judged by its flow through a 60° funnel having a 10 mm diameter orifice. A free flowing powder will run completely from the funnel in a relatively short time, leaving no residue adhering to the sides of the funnel.

A non-free flowing powder will not flow readily from the funnel unless assisted by, for example, vibration or it may flow slowly but leave a residue adhering to the sides of the funnel.

Some free flowing powders may become non-free flowing or even non-flowing when, for example, damp.

#### 7.2 Free flowing powders

Free flowing powders show a greater tendency to segregate than non-free flowing powders. Rotary sampling (see clause 14) is the method of first choice with chute riffing (see clause 13) as second choice.

Coning and quartering (see clause 12) gives sufficiently accurate subdivision for some purposes, but scoop sampling (see clause 11) should be used only if the powder is known to be homogeneous.

### 7.3 Non-free flowing powders

Coning and quartering (see clause 12) is the method of first choice. If the powder can be thoroughly mixed so that it is homogeneous, scoop sampling (see clause 11) will also give accurate subdivisions.

Rotary sampling may be used if the powder can be induced to flow by, for example, applying vibration but the possibility of biased sampling by the retention of the finer particles of the powder adhering to the apparatus should be considered.

### 7.4 Non-flowing powders

Coning and quartering (see clause 12) is the method of first choice. If the powder can be mixed so that it is homogeneous, scoop sampling (see clause 11) will also give accurate subdivision. Rotary sampling and chute riffing cannot be used.

## 8 Preliminary examination and precautions

### 8.1 General

Prior to selection of a sampling technique, a preliminary examination of the physical and chemical nature of the material to be sampled is essential. This will indicate whether certain methods of sample division will be suitable.

**NOTE** Ideally, preliminary tests should be carried out on a separate sample. Where this is not possible, the amounts taken from the laboratory sample should not significantly affect its integrity.

The precautions given in this clause are to ensure that, as far as possible, the sample and subdivided portions are not changed by the subdivision procedure, when applying the techniques described in section 4.

The properties of the material and aspects detailed in 8.2 to 8.15 should be considered.

### 8.2 Hazards

Precautions are necessary when sub-sampling hazardous powders. The relevant statutory requirements should be followed (e.g. Health and Safety at Work etc. Act, 1974) and reference should be made to any toxicology data (e.g. reference [1]<sup>3)</sup>) to protect the operator.

### 8.3 Chemical reactivity and environmental contamination

A knowledge of the chemical nature of the powder is necessary to assess its possible reaction with the environment and working surfaces.

Powders that react with, absorb or may be affected in some manner by normal atmospheric constituents such as water, oxygen or carbon dioxide, have to be subdivided in a way to prevent their contact with these substances or to reduce contact to a minimum. For example, subdivision can be carried out in an inert atmosphere or in a suitable non-reacting liquid. Subsequent handling, storage and testing should be carried out under the same conditions. With powders that react only slowly with these atmospheric substances, the subsampling should be carried out without undue delay but it should be realized that this may entail some loss of sampling accuracy. The effects of other possible gaseous atmospheric pollutants and dusts can, if essential, be minimized by carrying out the subsampling process in a clean-air environment, e.g. in a clean-air cabinet. If it is necessary to prevent bacteriological or fungal contamination, the sample subdivision process should be performed under aseptic conditions that encompass the sample containers, apparatus, operator and the immediate environment.

Certain powders may react with the material of the apparatus. Only materials that are compatible with such powders should be used in sampling containers, tools and the apparatus, or be permitted to come into contact with the powders.

The material used should also be impermeable to the samples being subdivided.

### 8.4 Foreign particles

Examination for the presence of contaminants, e.g. fibres, paper, wood, should be carried out, and their possible influence on sample division assessed. It may be necessary to remove them before subdivision, in which case this should be reported.

<sup>3)</sup> The numbers in square brackets used throughout the text of this standard refer to the bibliographic references given in Appendix C.

### 8.5 Particle size

The maximum particle size should be assessed. This, by definition of a powder, will not normally exceed 1 000  $\mu\text{m}$ , but the use of the methods in this Part of BS 3406 is not precluded by the presence of larger particles, provided that they do not affect the subdivision method. The loss of fine particles should be prevented by carrying out subdivision in an enclosed space, free from draughts or air currents. Sample dividers with moving hoppers or blades may themselves generate sufficient draught to cause segregation of particle sizes. The appearance of dust on parts of the apparatus outside the collection zone indicates that loss of fine particles is occurring.

### 8.6 Uniformity and density

The material should be examined to determine whether it contains particles of widely differing density or size, and an assessment made of its likely behaviour during sample division.

Powders of low density, especially organic materials which tend to form loose agglomerates, will be particularly susceptible to loss of fines by air currents. The magnitude of the effect is illustrated by the fact that a horizontal air current moving at only 15 cm/s will carry a 10  $\mu\text{m}$  particle of density 4 g/cm<sup>3</sup> fifteen times as far sideways as the height through which it falls.

### 8.7 Friability

Some materials can be liable to particle size reduction during sample subdivision owing to their friable nature and this property of the powder should be taken into consideration when selecting the most appropriate subsampling method. The effect of pressure and handling on the material should be assessed in the preliminary examination.

### 8.8 Abrasiveness

Abrasive powders can damage the sampling apparatus and thereby introduce the possibility of bias in subsequent sampling.

### 8.9 Moisture

The presence of high levels of moisture should be determined. This might be detected by a tendency to coalesce under pressure. A moisture determination may be of value where hygroscopic or efflorescent materials are concerned. Precautions should be taken to prevent changes in moisture content occurring during the subdivision process.

### 8.10 Electrostatic properties

The behaviour of the powder should be observed when subjected to vigorous movement. Signs of agglomeration, adhesion to working surfaces and unusual movement of individual particles should be noted.

Some powders may become charged with static electricity during the subdivision process and adhere to the apparatus. Parts of the apparatus in contact with the powder may require to be made of a suitable antistatic material.

### 8.11 Magnetic properties

Magnetic properties of materials may affect the sub-sampling process because of the attraction of particles to each other and to the apparatus.

### 8.12 Light

Powders affected by light should be subdivided in the absence of detrimental light.

### 8.13 Temperature

Powders, the composition of which might be affected by change of temperature, should be subdivided at the same temperature as that at which they are normally stored and handled.

### 8.14 Solubility (suspension sampling)

In preparing test portions of less than about 0.5 mL by making a suspension of the powder in a liquid, it is essential that the particulate matter is highly insoluble, i.e. less than 0.01 % dissolves in the liquid and it does not react chemically with it, swell or contract. It may be necessary to dilute pastes carefully in a controlled manner in order to prevent "dilution shock", i.e. flocculation of the particles, and the dispersion efficiency should be checked, e.g. by microscopic examination. The possibility of the occurrence of temperature cycling during sample preparation and its effect on such aspects as solubility should be considered.

### 8.15 Storage

Samples should be stored in such a manner that they are not liable to be changed or contaminated in any way during the storage period.

## 9 Quantity of material to be sampled

For a test sample or test portion to be defined as homogeneous, the size of the sample or portion has to be specified because homogeneity implies that the variability between samples or portions is either negligible or too small to be measured. This should not be taken to imply that homogeneity persists as material is further subdivided. An optimum sample or portion volume therefore has to be used which is capable of giving the necessary information about the material it represents. Sampling techniques used to obtain one volume of material may not therefore be applicable to obtain a representative smaller volume of material. Sampling techniques can be broadly categorized in terms of those giving test samples or test portions greater or less than about 0.5 mL in volume.

The chute riffler and rotary divider can easily be used to subdivide 10 g to 100 g laboratory samples of free flowing and, in some cases, non-free flowing particles. With many analytical methods, including microscopic examination and electrical sensing zone (ESZ) measurement, the test portion from the laboratory sample can be as small as a few milligrams. Samples smaller than about 0.5 mL of non-free flowing powder and at times even free flowing powder are difficult to subdivide and prone to errors with a dry sampling technique. A wet sampling technique may therefore be advisable (see section 5).

Difficulties may arise with heterogeneous materials when representative test portions with an exact predetermined mass are required. A number of operations of the sampling method(s) may be required, the feed to each sampling operation being the contents of one or more of the receivers, selected at random, filled during the previous operation.

## 10 Sampling efficiency

The attainment of a truly representative test portion will depend upon the efficiency of the sampling method (see Appendix A).

It is essential that methods of sample subdivision used to obtain test portions do not introduce bias. Any measurement of sampling efficiency will include a contribution from testing error. Only when the precision of the test is known can sampling efficiency be determined absolutely.

To measure sampling efficiency a number of separate test samples are produced by identical operation of the sub-sampling regime under consideration using standard materials with certified properties. From each test sample, two test portions are taken and tested by the method subsequently to be used. The overall variability and bias of the results is due to a combination of sampling and testing errors and these may be evaluated by statistical procedures.

Although known synthetic mixes, e.g. sand and sodium chloride, may be put through the subsampling regime, the results obtained will not necessarily be applicable to other materials.

Appendix A gives a comparison of the relative efficiencies, in terms of coefficients of variation, of the various sample subdivision methods described in sections 4 and 5 with a range of free flowing and poorly flowing particulate materials.

## Section 4. Methods for subdivision of the laboratory sample

### 11 Scoop sampling

#### 11.1 General

Scoop sampling is suitable only for materials that are homogeneous (or can be made so by mixing) within the limits set by the quantity of material taken by the scoop. It may be used for non-free flowing or damp materials where instrumental methods are inappropriate, and is also sometimes used to obtain test portions from a test sample produced by another sampling method.

If there are any doubts about the homogeneity of the material being sampled, scoop sampling is inappropriate.

This method is more suitable for less-free flowing powders; for powders with poor flow characteristics the method may be capable of subdividing samples with an accuracy and precision equal to that of instrumental methods (see Table 4).

#### 11.2 Apparatus

**11.2.1** *Sheets of impermeable material*, e.g. polished steel or glazed paper; the material chosen should not react with the sample.

**11.2.2** *Flat-bottomed scoops*, of a suitable material, e.g. stainless steel or suitable plastics, with a chamfered edge to collect the sample from the surface of the sheet. The size of the scoop should be such that it is capable of holding an amount of sample sufficiently large to be representative.

**11.2.3** *Containers*, of such a size that the material being sampled occupies between one-third and two-thirds of the volume, with airtight closures.

**11.2.4** *Spoons or spatulas*, which can be introduced through the neck of the test sample container.

#### 11.3 Procedure to obtain the test sample

**11.3.1** Mix the laboratory sample and empty it on to the impermeable sheet. With the back of the scoop or with a spatula flatten the heap and examine for evidence of particulate segregation. If this is apparent, remix the sample thoroughly and re-examine. If segregation persists use another sampling method, e.g. rotary sampling.

If the flattened heap appears to be homogeneous, take a number of scoopsful of sample at random and combine them. Ensure that each scoop takes the full depth of material, including that in contact with the surface of the sheet. It is recommended that a minimum of 10 scoopsful should be taken.

**11.3.2** Mix the combined scoopsful and repeat the procedure in **11.3.1**, if necessary, with a smaller scoop. However the scoop should not be so small that it introduces bias into the sample (see clause **9**).

Repeat the procedure of mixing and taking scoopsful of material until the required test sample is produced. In some cases the test portion is obtained directly from the laboratory sample and this procedure is not necessary. The procedure given in **11.4** may then be used directly to obtain the test portion.

**NOTE** There may be advantages in mixing the sample with a liquid to produce a cohesive mass or even a paste before the operation of subdivision. The liquid chosen should neither react with the sample nor be incompatible with any subsequent testing. It should also be possible to recover the sample in its original, unchanged form if dry material is required for testing.

#### 11.4 Procedure to obtain the test portion(s)

When the test sample is dry, place it in a clean, dry container of such a size that the test sample occupies between one-third and two-thirds of the volume of the container. Close with an airtight closure and thoroughly mix the test sample. The method of mixing chosen will depend upon the material and for each new material the tests for sampling efficiency described in clause **10** should be applied. Remove the closure and take the required test portion(s) with the spoon or spatula.

When the test sample is damp, or if preferred, use the procedure given in **11.3** to obtain the test portion(s).

## 12 Coning and quartering

### 12.1 General

Coning and quartering is more suitable for the less-free flowing powders; for powders with poor flow characteristics the method may be capable of subdividing samples with an accuracy and precision equal to that of instrumental methods (see Table 4).

### 12.2 Apparatus

**12.2.1** *Sheets of impermeable material*, e.g. polished steel or glazed paper; the material chosen should not react with the sample.

**12.2.2** *Flat-bottomed scoops*, of various sizes made of suitable material, e.g. stainless steel or suitable plastics.

**12.2.3** *Quartering device*, e.g. a thin metal or wooden cross, or crosses of different sizes, if necessary, consisting of four blades, fixed in the centre at right angles, of equal radius and at least as deep as the cone of powder.

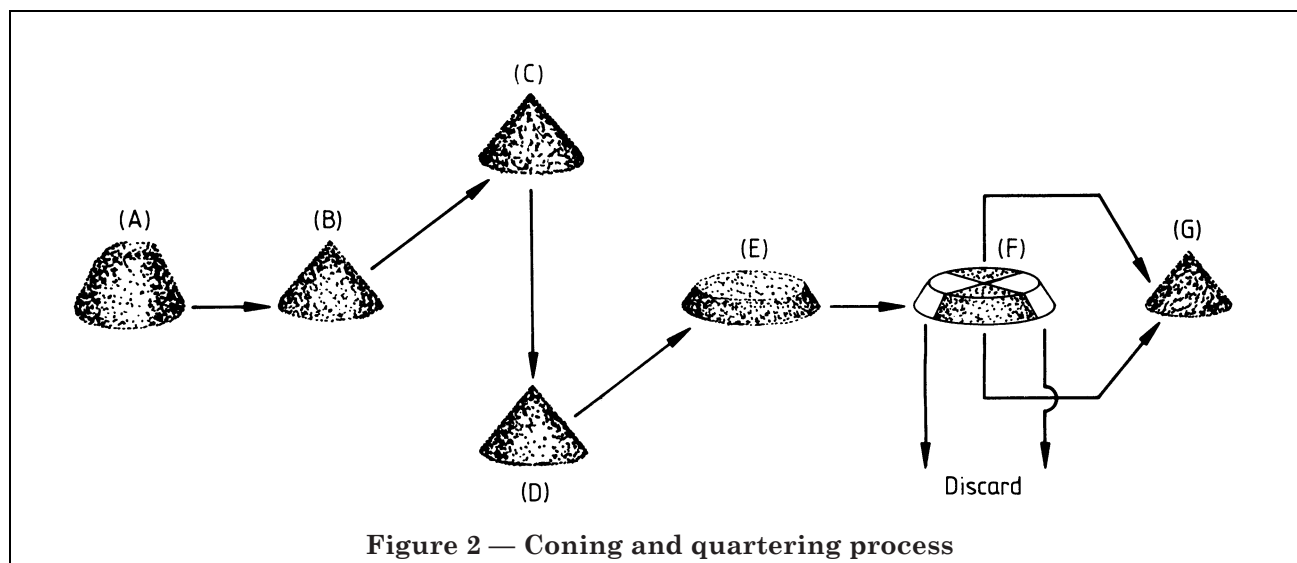


Figure 2 — Coning and quartering process

### 12.3 Procedure (see Figure 2)

**12.3.1** Place the powder as received in a heap (A) on the sheet of impermeable material large enough to enable the formation of a cone of powder of the same size beside it. Form a cone of powder (B) of the whole of the heap by tipping scoopsful on to the centre each time, so that it runs evenly down the sides of the cone. Repeat the process twice, i.e. forming cone (C) and then (D).

Flatten cone number three (D) with the back of the scoop. The flattened heap, (E), has to be of uniform thickness and diameter with its centre the same as that of the original cone (D). Since the larger particles tend to roll down the side of the cone and collect at the base there is likely to be segregation of particles from the top to the bottom of the cone. Care is therefore necessary when building up and flattening the cone.

Push the quartering device gently through the centre of the flattened heap (F).

Discard two of the opposing quarters and brush clean the empty segments while the cross is in position.

Mix the two remaining segments, form three further cones (G), etc. and repeat the procedure until the required size of test portion is obtained.

For free flowing powders it may be convenient as the heaps get smaller to use a short-stemmed funnel to form the cone. It is essential that it is placed low over the apex so that the material runs gently and evenly out of the stem.

**12.3.2** When using the method for pastes, press the material into shape when forming the cones. A conical mould in two halves could be used.

## 13 Chute riffing

### 13.1 General

Chute riffing is suitable neither for damp materials nor for powders which will not flow through the apparatus.

The method is generally used to produce samples of a minimum volume of 5 mL.

The method gives better results than scoop sampling or coning and quartering for free flowing powders (see Table 4).

### 13.2 Apparatus (see Figure 3)

**13.2.1** *Metal hopper*, fitted with an even number of parallel compartments of equal width. These are shaped and arranged so that every other compartment directs material fed to it to one side of the hopper, the other compartments directing material to the opposite side.

The width of each compartment (dimension A in Figure 3) should generally be not less than four times the maximum particle size in the powder. Widths of between 5 mm and 15 mm will be found suitable for most powders. The angle of slope should be such as to allow free fall under gravity of the powder through the apparatus.

The materials of construction should not corrode or react with the powders being divided.

**NOTE** Efficiency of sample division is improved by increasing the number of compartments in a given size of apparatus. However, there is a limit to the number of compartments that can be used before reduction of their width results eventually in blocking of the divider.



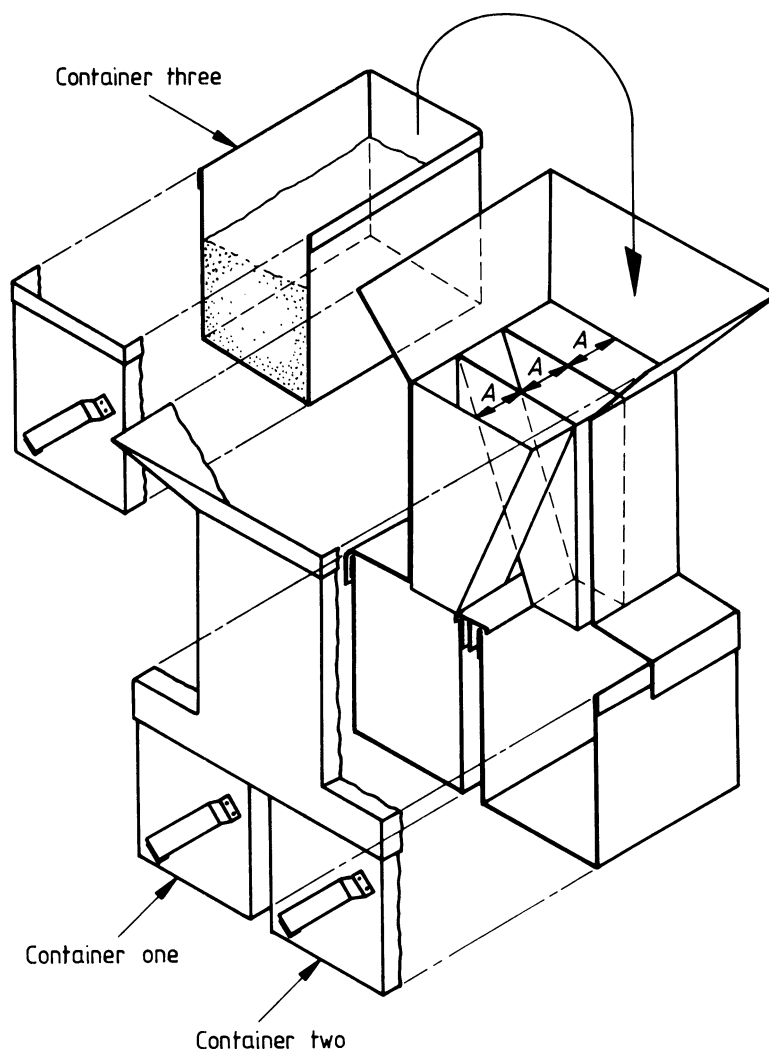


Figure 3 — Chute riffler

**13.2.2 Containers**, into which the material so directed falls and which may be covered to prevent loss of dust from the sample. The materials of construction should not corrode or react with the powders being divided.

**13.2.3 Third container or scoop**, of the same width as the top of the hopper which contains the powder sample to be divided.

Figure 3 shows a design which has proved to be satisfactory but alternative designs may be employed provided the essential requirements are fulfilled.

The materials of construction should not corrode or react with the powders being divided.

### 13.3 Procedure

**13.3.1** With containers one and two positioned on the apparatus, charge the third container or scoop with the well mixed powder, distributing it evenly across the width. Raise this container and hold it with its front edge on top of the long rim of the hopper and then slowly tilt it so that the powder flows in a uniform stream into all the compartments and thence into the two lower containers (one and two), one-half of the sample being collected in each of the latter.

**NOTE** It is important that the third container or scoop is the same width as the top of the hopper and that horizontal segregation is not introduced into the powder while filling the third container or during transfer to the hopper.

**13.3.2** Discard the material in container one and mix that in container two and repeat the procedure in **13.3.1** until the sample has been reduced to the required quantity.

Brush off powder adhering to the surface of a compartment so that it is collected in the appropriate container after each operation of the procedure.

## 14 Rotary sampling

### 14.1 Whole stream sampling

**14.1.1 General.** In sampling a particulate material the best results will be obtained if a stream of the material is sampled systematically, i.e. at regular intervals throughout the flow, and these increments combined to form the sample representing the bulk. There should be no periodicity in the variation of the material characteristic throughout the stream (a condition which is usually met in laboratory samples) and the flow of material should be as uniform as possible.

In practice, the material is delivered from a hopper by a small vibratory feeder and falls on to a succession of receivers mounted on a rotating table as shown schematically in Figure 4.

By this means the entire feed bulk is divided uniformly between the receivers. The greater the number of increments in each receiver, the smaller will be the variation between the collected subsamples. It has been shown that it is preferable to increase the number of increments by using low rates of flow of the powder rather than by increasing the rate of rotation.

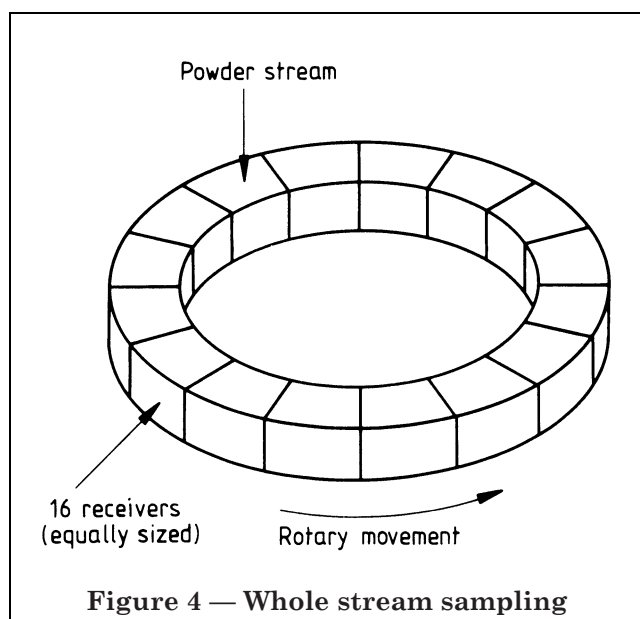


Figure 4 — Whole stream sampling

Other conditions for satisfactory operation require that the table holding the receivers should rotate at a uniform speed and that the receiver openings on to which the powder stream falls should be identical and of a uniform sector angle. Protection from draughts and adventitious materials should be provided.

**14.1.2 Whole stream rotary sample divider type 1** (see Figure 5). The laboratory sample is contained in a mass flow hopper which feeds material on to a vibratory chute and thence peripherally on to a rotating head. The head contains a number of identical downward angled ports which have sharp edged partitions between them. Receivers, one underneath each port, collect the divided sample. Typically the hopper is of 1 L capacity and the head contains 16 ports. The whole apparatus should be covered to prevent particle loss.

The material feed rate is controlled by variation in the gap between hopper and vibratory feeder and the degree of electromechanical vibration. Typically the speed of rotation is between 10 r/min and 20 r/min.

Alternative designs have vibratory hoppers rather than chutes and may have higher rates of rotation, e.g. 100 r/min, in order to assist the transfer of the material from the container ports by centrifugal force into receivers set at an angle to the rotating head.

**14.1.3 Whole stream rotary sample divider type 2** (see Figure 6). The laboratory sample is contained in a conical hopper and flows through an orifice on to a rotating table which is slightly conical.

A radial channel is cut in this table, extending from the centre to the periphery. The intention is that the material falling on to the table should be projected along the channel as the table rotates and be discharged into a number of ports, set into a stationary head. In order to assist the flow of the powder along the channel it is necessary that the table should rotate at a fairly high speed, e.g. 150 r/min. Sample receivers are held against the head below the ports by a platform which can be raised or lowered. The feed hopper is closed by a lid so that the apparatus is dust tight in operation. Typically the hopper is of 0.5 L capacity and the head contains 10 ports and 10 receivers.

The orifice of the hopper should be adjustable to maintain control of the flow rate. A vibrator of fixed frequency and amplitude can be used to assist the flow of difficult materials.

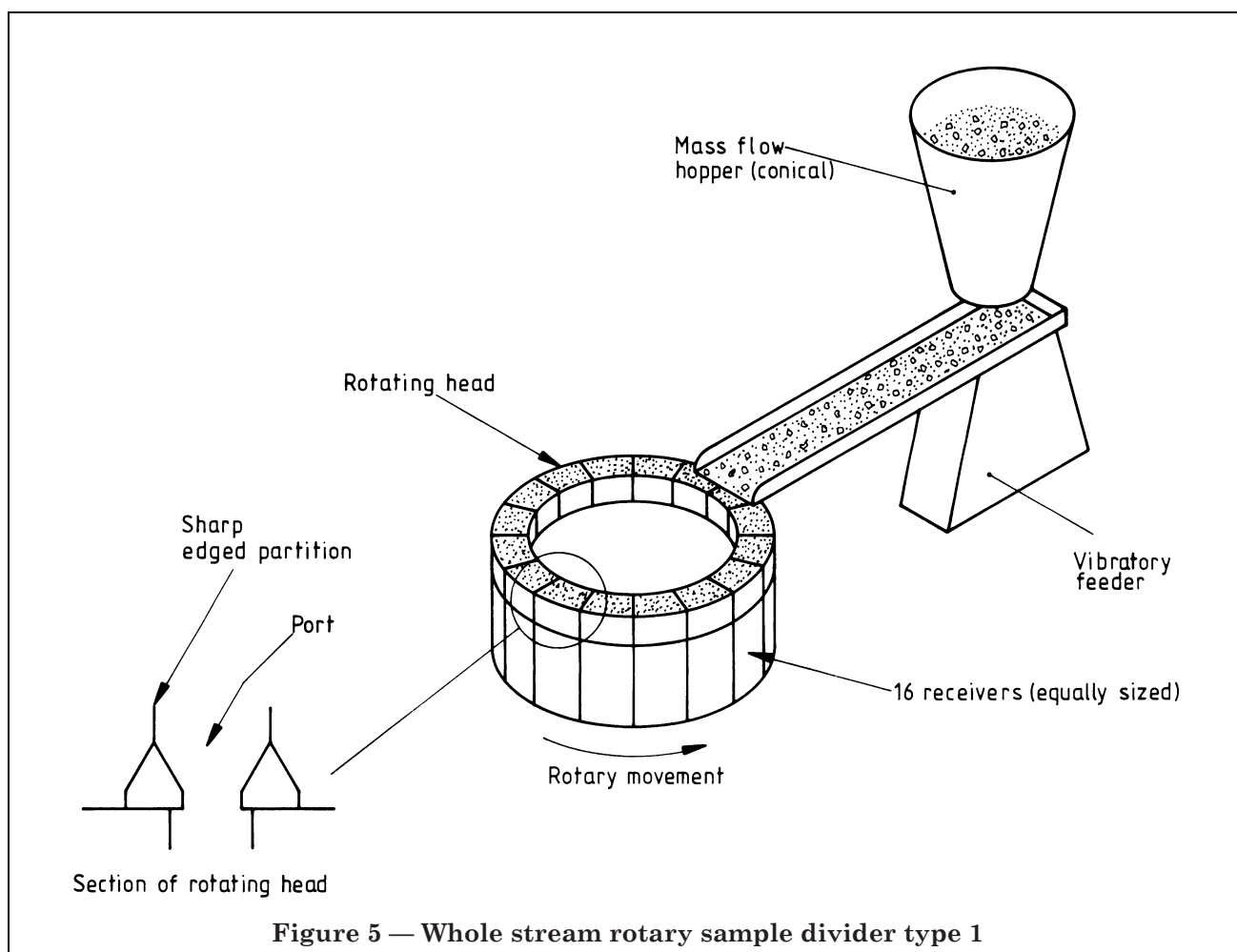


Figure 5 — Whole stream rotary sample divider type 1

## 14.2 Divided stream sampling

**14.2.1 General.** As with whole stream samplers, the principle of divided stream samplers is to sample a falling stream of material with a succession of receivers into which the material falls. However, the sample stream is one of a number of substreams into which the main stream can be considered to be divided by a distributor. This device is usually a cone with the intention that the material should be distributed uniformly round the periphery, and fall as a continuous curtain of particles (see Figure 7).

The sample collected in a particular receiver is composed of increments obtained by systematic sampling of a large number of streams (substreams) obtained by division of the main stream.

The substreams should be of equal rates of flow and uniform composition. The receivers should be identical and of a small sector angle so that a large number of small increments would be taken in each receiver. The conical distributor should distribute the powder stream equally in all directions. It should be vertical and coaxial with the falling stream, particularly if it is stationary. It should not be damaged or displaced, otherwise the relationship between it and the receivers could result in systematic variation between the samples. The rate of flow of the powder stream should be controllable, so that the distributor does not become overloaded and fail to distribute the powder uniformly. As for whole stream sampling (see 14.1), the operation should be protected from draughts and adventitious material.

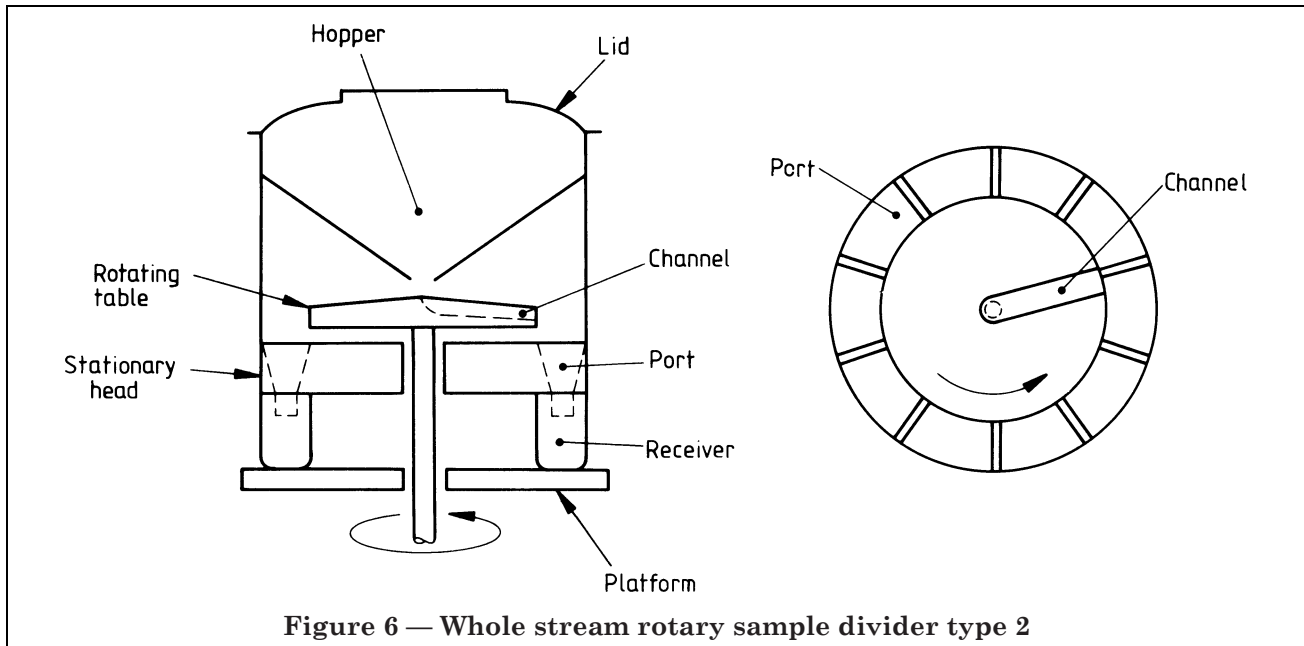


Figure 6 — Whole stream rotary sample divider type 2

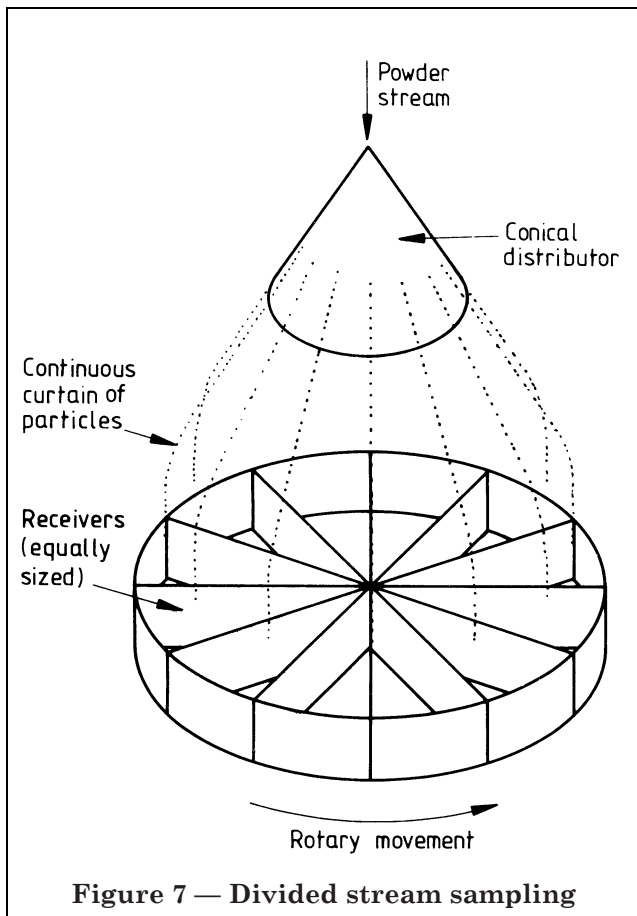


Figure 7 — Divided stream sampling

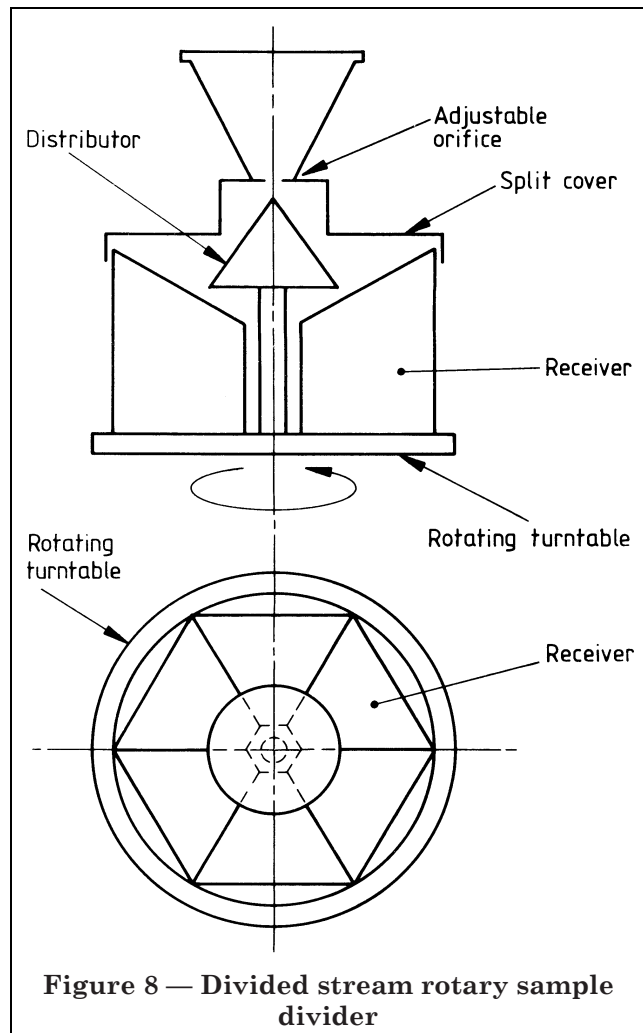


Figure 8 — Divided stream rotary sample divider

#### **14.2.2** *Divided stream rotary sample divider*

(see Figure 8). The laboratory sample, contained in a conical hopper, is fed through an adjustable orifice on to a conical distributor. The material falling from the distributor is intercepted by a number of truncated sector-shaped receivers, mounted on a rotating turntable. Figure 8 illustrates an arrangement in which the distributor rotates at the same speed as the receivers. Typically the hopper is of 2 L capacity and the turntable carries six receivers. A typical speed of rotation is 50 r/min.

To obtain an even distribution of material over the distributor the cone should be coaxial with the rotation axis and undamaged and the rate of flow should be low. This evenness of distribution becomes more difficult to achieve, the finer and less-free flowing the powder.

#### **14.3** **General procedure for rotary sampling**

**14.3.1** A preliminary trial to find the flow control conditions to give a low consistent rate of flow is advisable, the material being returned to the hopper before sample subdivision takes place.

Ensure that all surfaces of the equipment likely to come in contact with the material are clean and dry, particularly the hopper, feeder, ports and receivers. With the clean receivers in position commence the sampling process. At the end of the sample division weigh the collected material in each receptacle and compare the total masses with the initial mass of the powder. The discrepancy should be not more than 0.25 %. If greater, it should be investigated and the cause eliminated if possible.

Make a minimum of 30 complete revolutions of the sampling head during the sampling.

**14.3.2** If the sampling equipment will not accept the whole of the laboratory sample, mix it by any convenient means and divide it into two or more parts which can each be accepted by the sampling equipment. Proceed as in **14.3.1** with one of the parts and reserve the contents of one or more of the receivers. Repeat with each of the other parts separately, reserving in each case the contents of the same number of receivers. When all the laboratory sample has been so treated, combine all the reserved powder as the test sample which may be further subdivided if required.

## Section 5. Methods for the preparation of very small test portions

### 15 General

This section describes methods intended for the preparation of subsamples of about 0.5 mL or less which are not conveniently prepared by the methods described in section 4. This does not preclude their use for the preparation of samples of larger volume. The starting samples for the methods described in this section are usually the samples divided using the techniques described in section 4.

### 16 Suspension sampling

#### 16.1 General

The suspension sampling methods described in 16.2 and 16.3 can be used to obtain test portions as small as 0.2 mL by successive dilution and subdivision of one or more of the individual suspension samples. The test portion (in suspension) can be used directly in the analysis method, if necessary after further dilution with suspension liquid, or after centrifuging and drying. The method of drying chosen should not change the physical or chemical nature of the particles.

#### 16.2 Suspension sampling by multiple capillary tubes

**16.2.1 Apparatus** (see Figure 9). A glass pipe with a capacity of about 150 mL is provided with a stirrer and a base plate. Around the periphery of the base plate are drilled a number of equi-spaced holes into which are fixed capillary tubes which are flush with the upper face of the base plate and extend below it. Each tube protrudes into a graduated glass receiver standing in a plinth which also supports the base plate. The pipe is covered with a second stainless steel plate with a central hole containing a bearing through which passes a stirrer shaft. This is rotated by means of a suitable motor (not shown). Also in the cover plate is a sealable inlet through which is admitted the powder suspension and a gas orifice in the form of a threaded union which enables the gas pressure in the vessel to be increased or decreased. In a typical instrument [2] there are 10 equi-spaced holes, the capillary tubes are 0.7 mm bore stainless steel and the graduated receivers are of 10 mL capacity.

**16.2.2 Procedure.** The procedure should be as follows.

- a) Admit to the vessel a volume (typically 100 mL) of a suspension, e.g. prepared from a test sample produced by one of the subdivision methods described in section 4, while simultaneously rotating the stirrer.

- b) Apply a slight suction to the vessel while the suspension is being stirred. The capillary tubes are thus kept free of suspension and free from the possibility of blockage until the next step c).

- c) After 10 s to 15 s and while still being stirred, subject the suspension to a positive pressure of 20 kPa to 35 kPa<sup>4)</sup> so that it is blown out through the capillary tubes into the receivers.

- d) Divide the suspension into aliquots of substantially 10 mL each. If required, dilute one or more of the 10 mL aliquots and subdivide in the same way.

- e) Test the apparatus before and after use with clean liquid: the volumes delivered to the receivers will thus provide an indication as to whether the capillary tubes are free from blockage. Blockage of the capillary tubes is the most probable cause of error. This is most likely to occur when suspensions of powders containing larger particles or agglomerates of particles are being subdivided although the use of suction during stirring and the application of pressure during discharge minimizes this problem. It is recommended that the apparatus is not used for powders whose maximum size exceeds about one-third of the capillary tube bore; for the apparatus depicted in Figure 9 this would mean an upper limit of about 200 µm.

#### 16.3 Suspension sampling by pipette (or dropping tube)

**16.3.1 General.** Experience shows that suspension sampling (or dropping tube) is more suitable for powders with a maximum particle size of about 20 µm which are known to have a narrow size distribution.

**16.3.2 Procedure.** Place a suspension of the test sample in a cylindrical vessel fitted with a stirrer and baffles which are positioned to minimize symmetrical flow patterns. Agitate the suspension thoroughly.

While the suspension is being agitated, withdraw aliquots containing the required amounts of test portions, using a pipette or dropping tube of an appropriate volume and orifice diameter.

### 17 Paste sampling

#### 17.1 General

Paste sampling has also been found suitable for obtaining the test portions for analysis methods requiring a powder suspension but is highly operator dependent.

<sup>4)</sup> Approximately 3 lbf/in<sup>2</sup> to 5 lbf/in<sup>2</sup> in imperial units.

### 17.2 Procedure

Mix into a paste with a few drops of a suitable dispersant using a flexible spatula test samples greater than 0.5 mL obtained by one of the methods described in section 4. Work and knead the paste gently with a spatula in order to disperse agglomerates but not to crush primary particles. When the paste appears to be thoroughly smooth and homogeneous take, with the spatula, test portions of the required amount. These can then either be diluted with and dispersed in a suitable liquid for analysis, or dried by some suitable means.

### 18 Sampling for microscopic analysis

The technique of microscopic analysis is highly specialized and BS 3406-4 and appropriate text books should be consulted.

The amount of energy used in dispersing a sample in the laboratory during its preparation for analysis should be as equivalent as possible to the energy of dispersion in product use. This is particularly important with regard to agglomerates, which may behave in use as single particles, and to fragile particles.

It is important to observe under the microscope the addition of the proposed liquid dispersion medium to some of the dry sample prior to preparing it for microscopic analysis in order to ascertain if any reaction, e.g. complex formation or breakdown which can be instantaneous, occurs. It is also important to ensure no dissolution occurs.

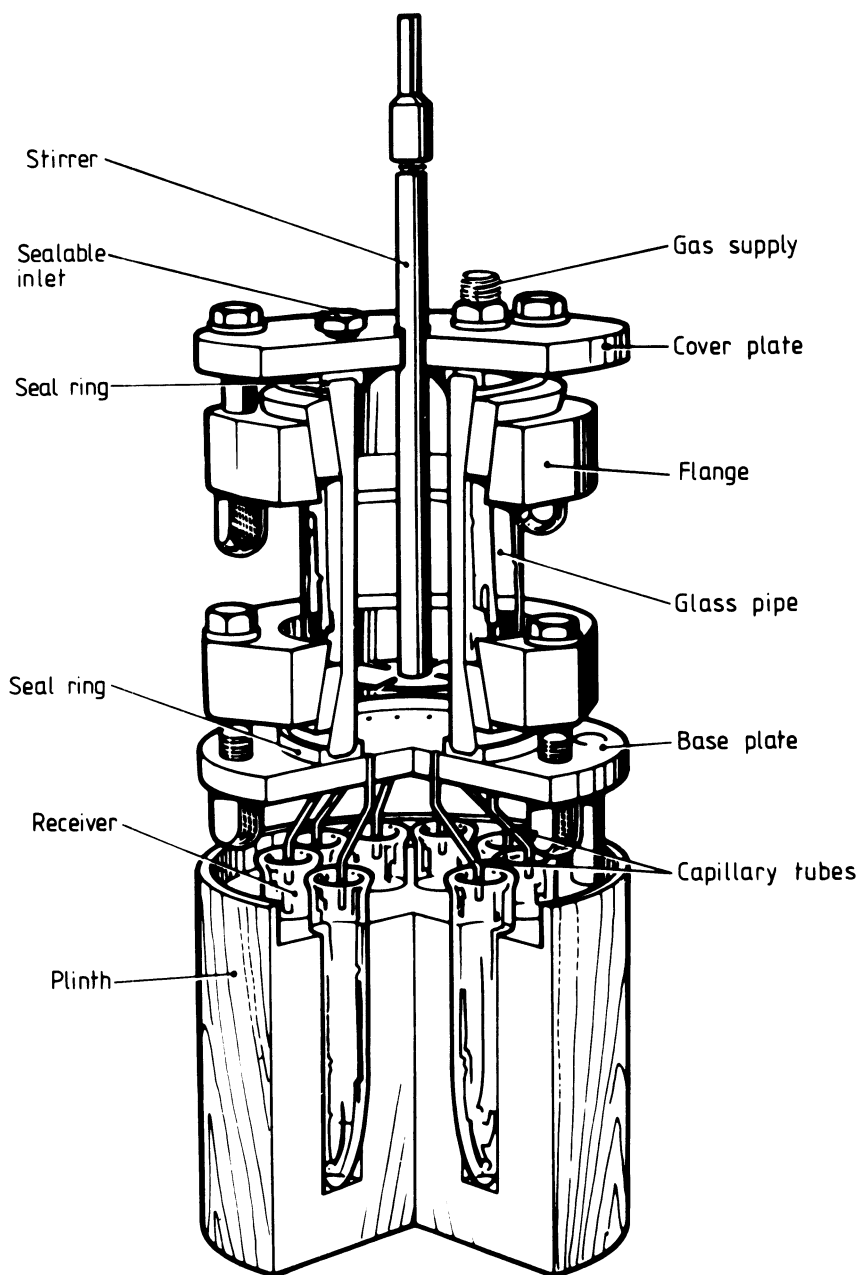


Figure 9 — Suspension sampler



## Appendix A Comparative sampling efficiencies

### A.1 Methods of dry powder sampling

#### A.1.1 General

Investigations into the relative efficiencies of the sample dividers described in section 4 have been carried out by several workers in powder technology [3], [4] and [5]. The studies have been designed to evaluate the effects that differing physical characteristics, such as particle size, shape, density, flow properties and the influence of operator bias, may have on the procurement of a representative test portion.

#### A.1.2 Free flowing powders

Table 1 gives the relative efficiencies of the laboratory sample subdivision methods described in section 4 as found by three groups of workers using free flowing materials of differing particle size, shape and density. Table 2 gives the ranges of coefficients of variation of sub-sample mass. This is appropriate to sampling to arrive at a test portion close to a desired mass. Division of an *unmixed* sample using the rotary sampler gave coefficients of variation from 1.4 to 3.1, the latter resulting when the fine component was placed on top of the coarse component in the hopper, i.e. better results than obtained with the other methods when subdividing well-mixed samples.

The results given in Table 1 and Table 2 show that for free flowing material, rotary sample dividers:

- a) give the most representative sample;
- b) are relatively operator independent;
- c) give the lowest inter sub-sample mass variance.

Chute riffling is the second best method.

#### A.1.3 Powders with different flow properties

The effect of differing flow properties on the laboratory sample subdivision methods described in section 4 has been investigated using the following four feed materials.

A. Free flowing powder	sand limestone	200 $\mu\text{m}$ to 300 $\mu\text{m}$ 45 $\mu\text{m}$ to 150 $\mu\text{m}$	30 % 70 %
B. Less-free flowing powder	sand AC fine dust	200 $\mu\text{m}$ to 300 $\mu\text{m}$ 0 $\mu\text{m}$ to 80 $\mu\text{m}$	80 % 20 %
C. Poorly flowing powder	sand AC fine dust	200 $\mu\text{m}$ to 300 $\mu\text{m}$ 0 $\mu\text{m}$ to 80 $\mu\text{m}$	20 % 80 %
D. Very-poorly flowing powder	fine chalk (majority < 10 $\mu\text{m}$ )		100 %

NOTE The fractions of material in samples A, B and C were mixed together only by shaking in a bottle. The degree of flowability of the feed materials was assessed only subjectively.

After division of the weighed feeds, the compositions of the subsamples were determined (see Table 3) and their masses obtained (see Table 4).

Table 3 shows that for the whole stream sampler type 1 (WS1) the tendency was for variability of composition to increase with decreasing flowability and also with increasing rate of flow. For the other methods, the most freely flowing material, in general, yielded samples with the largest variability. The chute riffler appeared to be at least as good as the divided stream sampler (DS), in this respect. In general, the scoop sampling and coning and quartering methods subdivided the less freely flowing powders as effectively as the more mechanical methods.

Table 4 shows that, overall, diminishing ability to flow was associated with an increase in variability of sample mass. A rate-of-flow effect was seen with the free flowing sample A and WS1 although, contrary to expectations, the variability was increased at a very low rate of flow. This suggests that for this apparatus and material there may be an optimum range of rate of flow. With whole stream sampler type 2 (WS2), the rate of flow is uncontrolled and varies with flowability. Considering the relative magnitude of these rates of flow, WS2 seemed to perform as well as WS1 with respect to variability of subsample mass, apart from the curiously high value of the coefficient of variation for sample B. The DS was plainly worse for variability and in general was bettered by the chute riffler.

In summary, based upon these results, for free flowing materials WS1 would seem to be best method for obtaining representative samples. As flowability decreases, the technique of subdividing becomes less important, unless a number of samples of nearly equal mass are required, in which case WS1 or WS2 would be preferred.

#### **A.2 Methods of wet powder sampling**

The precision and efficiency of the suspension sampler have been compared with other sampling methods to obtain small test portions [5].

Table 5 shows that the suspension sampler and the paste method have similar precisions for the largest samples and are both better than the pipette method. As the sample size is decreased the superiority of the suspension sampler becomes evident.

The possible large contribution of sampling error to the precision of analytical methods using very small samples is also indicated.

**Table 1 — Relative efficiencies of sampling methods for free flowing powders**

Workers	Feed material	Coefficient of variation of sample composition <sup>a</sup>				
		Scoop sampling	Coning and quartering	Chute riffing	Rotary sampling (whole stream)	Random variation <sup>b</sup>
[3]	Bronze spheres A 44 % > 125 µm	—	2.0	0.74	0.32	—
	Bronze spheres B 80 % > 125 µm	4.4	—	1.8	—	—
[4]	Coarse sand 420 µm to 500 µm. Density 2.65 g/cm <sup>3</sup>					
	Fine sand 150 µm to 250 µm. Density 2.65 g/cm <sup>3</sup>	8.6	11.4	1.7	0.21	0.13
	Fine sand 150 µm to 250 µm. Density 2.65 g/cm <sup>3</sup>					
	Sugar 420 µm to 500 µm. Density 1.64 g/cm <sup>3</sup>	10.5	9.6	1.8	0.45	0.16
[5]	Sodium chloride 0 to 300 µm. Density 2.2 g/cm <sup>3</sup> Polypropylene pellets approximately 3 000 µm. Density 0.9 g/cm <sup>3</sup>					
Operator	1 No previous experience in any of the methods	15.1	11.7	6.4	1.8	—
	2 Limited experience in all methods	14.2	8.1	3.8	1.4	—
	3 Some experience in all but rotary sampler	11.6	7.7	3.2	1.2 to 1.8 (6 results)	—
	4 Very experienced in all methods	6.6	3.6	2.8	1.6	—

<sup>a</sup> Coefficient of variation. The samples obtained by each method were analysed for composition and the mean value and standard deviation of each group of test results calculated.  
Coefficient of variation =  $\frac{\text{Standard deviation}}{\text{Mean value}} \times 100$ .

<sup>b</sup> Random variation is the theoretical minimum variation to be expected under ideal conditions.

**Table 2 — Powder sampling methods: variation of subsample masses**  
(obtained on 16 subsamples by operators 2, 3 and 4 [5] (see Table 1))

Method	Range of coefficient of variation (percentage of mean mass) obtained by three operators
Scoop sampling	7.2 to 17.7
Coning and quartering	5.3 to 9.3
Chute riffing	5.9 to 9.0
Rotary sampling (whole stream)	2.0 to 2.5

Table 3 — Relative efficiencies of powder sampling methods: subsample composition

Feed	Nominal percentage of major component	Scoop sampling		Coning and quartering		Chute riffling		Rotary sampling								
		$\bar{C}$	$V_c$	$\bar{C}$	$V_c$	$\bar{C}$	$V_c$	Whole stream sampler type 1 (WS1)			Whole stream sampler type 2 (WS2)			Divided stream sampler (DS)		
								$R$	$\bar{C}$	$V_c$	$R$	$\bar{C}$	$V_c$	$R$	$\bar{C}$	$V_c$
A Free flowing powder	70	71.0	4.0	72.3	2.3	69.8	0.9	g/s 0.17 0.98 4.30	% 70.0 70.0 70.0	0.2 0.3 0.4	g/s 11.1	% 70.1	1.1	g/s 13.3	% 70.1	3.9
B Less-free flowing powder	80	81.8	0.6	80.6	0.3	80.4	0.6	0.17	81.1	0.5	3.60	80.7	2.3	0.60	81.0	0.6
C Poorly flowing powder	80	78.6	0.5	79.7	0.3	79.7	0.4	0.17 0.56	79.8 79.7	0.5 1.4	1.38	79.5	0.2	0.96 4.00	79.8 80.4	0.4 3.1
Number of sub-samples		6		6		8		16			10			6		

**Key:**

$R$  is the rate of flow.

$\bar{C}$  is the mean mass percentage of major component.

$V_c$  is the coefficient of variation of mass percentage of major component =  $\frac{\text{Standard deviation of mass percentages of major component}}{\text{Mean mass percentage of major component}} \times 100$ .

NOTE Results for feed material D are not included in Table 3. This is because the rapid sieve analysis method used for the other three feed materials was not appropriate. A limited number of size distribution analyses were carried out using a light scattering method. Although the results were not subjected to rigorous statistical analysis no obvious differences were observed between the results obtained. This is not entirely unexpected because of the probable homogeneity of this cohesive material. However, not all cohesive materials will necessarily exhibit the same degree of homogeneity.

Table 4 — Relative efficiencies of powder sampling methods: subsample mass

Feed	Chute riffling		Rotary sampling								
			Whole stream sampler type 1 (WS1)			Whole stream sampler type 2 (WS2)			Divided stream sampler (DS)		
	$\bar{W}$	$V_w$	$R$	$\bar{W}$	$V_w$	$R$	$\bar{W}$	$V_w$	$R$	$\bar{W}$	$V_w$
A. Free flowing powder	g 12.5	2.7	g/s 0.17 0.98 4.3 0.027	g 25.0 25.0 25.0 1.0	1.0 0.8 2.9 3.3	g/s 11.1	g 20.0	2.0	g/s 13.3	g 33.2	10.0
B. Less flowing powder	12.4	6.7	0.17	12.5	1.9	3.60	20.0	11.7	0.60	34.0	6.4
C. Poorly flowing powder	12.4	4.9	0.17 0.56	12.5 12.6	4.4 4.4	1.38	20.0	3.3	0.96 4.00	33.2 31.7	9.0 12.5
D. Very poorly flowing powder	12.6 12.4	10.4 4.0	0.42 0.29	6.3 6.3	9.7 4.3	— 0.45	10.2 10.0	9.1 2.3	— 0.26	33.2 16.6	6.4 8.2
Number of sub-samples	8		16			10			6		

**Key:**  
 $R$  is the rate of flow.  
 $\bar{W}$  is the mean sample mass.  
 $V_w$  is the coefficient of variation of sample mass =  $\frac{\text{Standard deviation of sample masses}}{\text{Mean sample mass}} \times 100$ .

NOTE Data for coning and quartering and for scoop sampling are not given because these techniques are not designed to produce multiple subsamples of equal mass.

Table 5 — Comparison of sampling methods to obtain small test portions

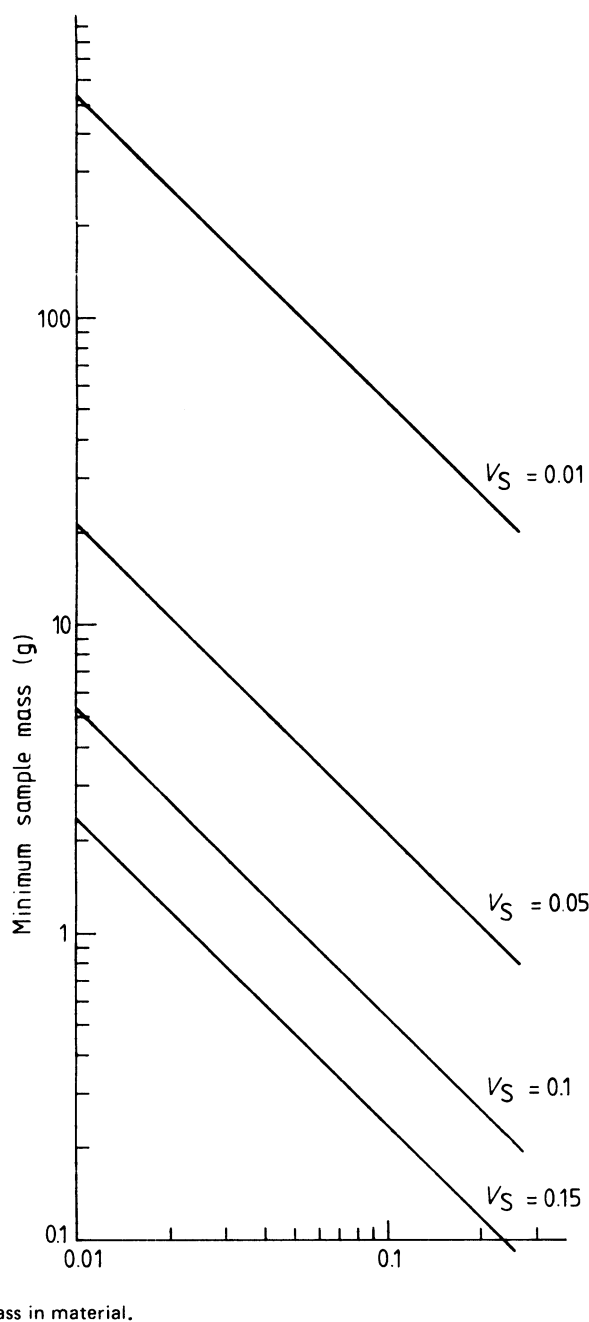
Feed material: Mixture of 90 % fine alumina powder and 10 % coarse copper oxide powder					
Method	$W_t$	$w$	Copper oxide content by mass %		
			$\bar{M}$	$\sigma$	$V_M$
Suspension sampler	mg	mg	%	%	%
	2 000	200	10.05	0.148	1.5
	200	20	9.94	0.338	3.4
Paste coning and quartering	200	2	9.87	0.839	8.5
	2 000	200	10.02	0.140	1.4
Pipette	200	20	9.97	0.568	5.7
	2 000	200	10.03	0.351	3.5

**Key:**  
 $W_t$  is the total solids in bulk sample.  
 $w$  is the approximate total solids in analysis sample.  
 $\sigma$  is the standard deviation.  
 $V_M$  is the coefficient of variation.  
 $\bar{M}$  is the average copper oxide content by mass.

## Appendix B Relationship between minimum sample mass and mass fraction of coarsest size

The relationship between minimum sample mass and the mass fraction of the coarsest size is shown in Figure 10. The graphs are calculated for material of density  $1 \text{ g/cm}^3$  and mean class size  $1 \text{ mm}$ . For density  $\rho$  (in  $\text{g/cm}^3$ ) and size  $s$  (in  $\text{mm}$ ), multiply indicated sample mass by  $\rho s^3$ .

$V_s$  is the coefficient of variation.



**Figure 10 — Minimum sample mass as function of mass-fraction of largest-size class in a well-mixed bulk material and required precision**

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- BS 3406-2, *Recommendations for gravitational liquid sedimentation methods for powders and suspensions*<sup>5)</sup>.
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- BS 3406-4, *Optical microscope method*.
- BS 3406-5, *Recommendations for electrical sensing zone method (the Coulter Principle)*<sup>5)</sup>.
- BS 3406-6, *Recommendations for centrifugal liquid sedimentation methods for powders and suspensions*<sup>5)</sup>.
- BS 3406-7, *Recommendations for single particle light interaction methods*<sup>5)6)</sup>.
- BS 4359, *Determination of the specific surface area of powders*<sup>5)</sup>.
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<sup>5)</sup> Referred to in the foreword only.

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