

Methods for

Determination of particle size distribution —

Part 1: Guide to powder sampling

UDC 620.168.36:620.113-492.2

NO COPYING WITHOUT BSI PERMISSION EXCEPT AS PERMITTED BY COPYRIGHT LAW

BSI
Documents

AVA

Document Sharing Hub

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the General Mechanical Engineering Standards Committee (GME/-) to Technical Committee GME/29 upon which the following bodies were represented:

- BCIRA
- British Aggregate Construction Materials Industries
- British Ceramic Society
- British Laboratory Ware Association
- Cement and Concrete Association
- Cement Makers' Federation
- Coated Abrasives Manufacturers' Association
- Department of Trade and Industry (Warren Spring Laboratory)
- Department of Transport (Highways)
- Guild of Metal Perforators
- Incorporated National Association of British and Irish Millers
- Institution of Chemical Engineers
- Institution of Mining and Metallurgy
- Mechanical Handling Engineers' Association
- Ministry of Defence
- National Coal Board
- Society of Chemical Industry
- Society of Cosmetic Scientists
- Woven Wire Association

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

- AERE Harwell
- Aluminium Federation
- British Steel Industry (Wire Section)
- China Clay Association
- Department of Trade and Industry (National Physical Laboratory)
- Pharmaceutical Society of Great Britain
- Royal Society of Chemistry
- Sunderland Polytechnic (Magnet Centre)
- University of Bradford
- University of Technology, Loughborough
- Wellcome Federation

This British Standard, having been prepared under the direction of the General Mechanical Engineering Standards Committee, was published under the authority of the Board of BSI and comes into effect on 31 October 1986

© BSI 09-1999

The following BSI references relate to the work on this standard:
Committee reference GME/29
Draft for comment 83/79599 DC

ISBN 0 580 15305 3

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

	Page
Committees responsible	Inside front cover
Foreword	iii
Section 1. General	
0 Introduction	1
1 Scope	1
2 Definitions	1
Section 2. Collection of gross and laboratory samples	
3 Guidelines	3
4 Sampling strategy	3
Section 3. Selection of a sampling method for subdivision of the laboratory sample	
5 General	5
6 Homogeneity	5
7 Flow characteristics	5
8 Preliminary examination and precautions	6
9 Quantity of material to be sampled	7
10 Sampling efficiency	8
Section 4. Methods for subdivision of the laboratory sample	
11 Scoop sampling	9
12 Coning and quartering	9
13 Chute riffing	10
14 Rotary sampling	12
Section 5. Methods for the preparation of very small test portions	
15 General	16
16 Suspension sampling	16
17 Paste sampling	16
18 Sampling for microscopic analysis	17
Appendix A Comparative sampling efficiencies	19
Appendix B Relationship between minimum sample mass and mass fraction of coarsest size	24
Appendix C Bibliography	25
Figure 1 — Sampling strategies	4
Figure 2 — Coning and quartering process	10
Figure 3 — Chute riffler	11
Figure 4 — Whole stream sampling	12
Figure 5 — Whole stream rotary sample divider type 1	13
Figure 6 — Whole stream rotary sample divider type 2	14
Figure 7 — Divided stream sampling	14
Figure 8 — Divided stream rotary sample divider	14
Figure 9 — Suspension sampler	18
Figure 10 — Minimum sample mass as function of mass-fraction of largest-size class in a well-mixed bulk material and required precision	24
Table 1 — Relative efficiencies of sampling methods for free flowing powders	21
Table 2 — Powder sampling methods: variation of subsample masses	21
Table 3 — Relative efficiencies of powder sampling methods: subsample composition	22

	Page
Table 4 — Relative efficiencies of powder sampling methods: subsample mass	23
Table 5 — Comparison of sampling methods to obtain small test portions	23
Publications referred to	Inside back cover

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 methods¹⁾.*

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.

¹⁾ 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/cm³, a minimum sample of 4.4 g would be required if the largest size class formed 3 % (m/m)²⁾ 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.

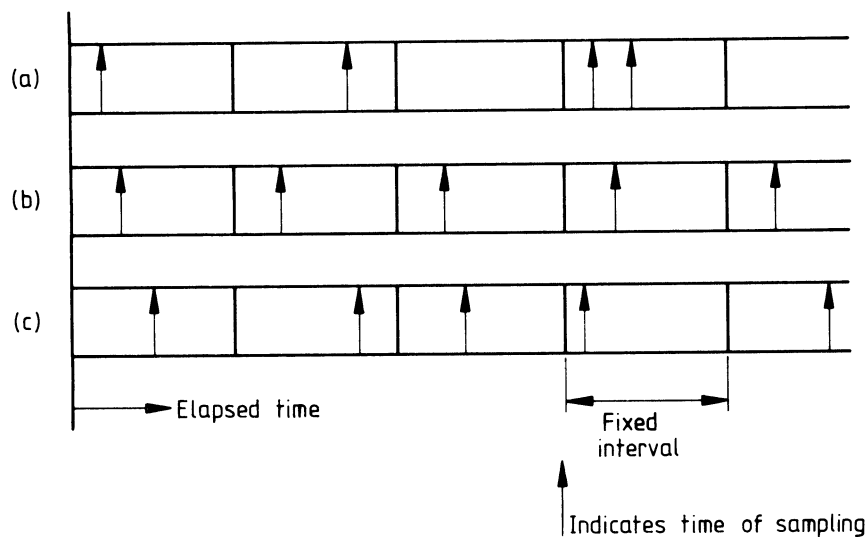
The best practical method to obtain a gross or laboratory sample which is representative of the bulk material is to sample a flowing stream of the material. The sampling errors can be minimized by maintaining a constant rate of flow and by taking a large number of increments to form the sample. The increments 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.

²⁾ 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.