

BRITISH STANDARD 3406 : Part 3 : 1963

CONFIRMED DECEMBER 1983

METHODS FOR DETERMINATION OF PARTICLE SIZE DISTRIBUTION

Part 3. Air elutriation methods

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THIS BRITISH STANDARD, having been approved by the Chemical Engineering Industry Standards Committee and endorsed by the Chairman of the Engineering Divisional Council was published under the authority of the General Council on 20th June, 1963.

The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

In order to keep abreast of progress in the industries concerned, British Standards are subject to periodical review. Suggestions for improvements will be recorded and in due course brought to the notice of the committees charged with the revision of the standards to which they refer.

A complete list of British Standards, numbering over 9,000, fully indexed and with a note of the contents of each, will be found in the BSI Catalogue which may be purchased from BSI Sales Department. The Catalogue may be consulted in many public libraries and similar institutions.

This standard makes reference to the following British Standards:

- B.S. 410. Test sieves.
- B.S. 1377. Methods of testing soils for civil engineering purposes.
- B.S. 1796. Methods for the use of British Standard fine-mesh test sieves.
- B.S. 2955. Glossary of terms relating to powders.
- B.S. 3406 : Methods for determination of particle size distribution
- B.S. 3483. Methods for testing pigments for paints.

British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. It is important that users of British Standards should ascertain that they are in possession of the latest amendments or editions.

The following B.S.I. references relate to the work on this standard:
Committee reference CHE/50, CHE/50/1
Draft for comment CZ(CHE)3221

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CO-OPERATING ORGANIZATIONS

The Chemical Engineering Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

- *Association of British Chemical Manufacturers
- Association of Consulting Engineers (Incorporated)
- Board of Trade
- *British Chemical Plant Manufacturers' Association
- Coke Oven Managers' Association
- Engineering Equipment Users' Association
- Gas Council
- Glass Manufacturers' Federation
- Institute of Petroleum
- *Institution of Chemical Engineers
- Institution of Gas Engineers
- Institution of Structural Engineers
- Ministry of Transport
- *Society of Chemical Industry (Chemical Engineering Group)
- United Kingdom Atomic Energy Authority

The scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

Aluminium Federation
Borough Polytechnic
British Cast Iron Research Association
British Coal Utilisation Research Association
British Colour Makers' Association
British Diamond Die Federation
British Electrical and Allied Manufacturers' Association
British Hard Metal Association
British Pharmacopœia Commission
Cement and Concrete Association
Cement Makers' Federation
D.S.I.R.-Road Research Laboratory
D.S.I.R.-Warren Spring Laboratory
Imperial College of Science and Technology
Incorporated National Association of British and Irish Millers Ltd.
Institute of Physics and The Physical Society
Institution of Mining and Metallurgy
Ministry of Aviation
Ministry of Power
National Physical Laboratory (D.S.I.R.)
Oil and Colour Chemists' Association
Permanent Magnet Association
Pharmaceutical Society of Great Britain
Radio and Electronic Component Manufacturers' Federation
Research Association of British Paint, Colour and Varnish Manufacturers
Society of Cosmetic Chemists of Great Britain
Toilet Preparations' Federation Ltd.
University of London
War Office
Zinc Pigment Development Association
Individual firms

BRITISH STANDARD METHODS FOR
DETERMINATION OF
PARTICLE SIZE DISTRIBUTION
Part 3. Air elutriation methods

FOREWORD

A conference on powders was convened by the British Standards Institution in 1952. This standard is the outcome of one of the projects then decided. It is one of a series which describes methods of determining the size distribution of particles in those fractions of powders which pass through a 75 micron British Standard test sieve*. The size distribution of particles retained on the sieve may be determined by sieving†.

Present standards in this series are as follows:

- B.S. 3406 : Part 1. Sub-division of gross sample down to 0.2 ml.
- 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).

Further standards will be compiled as necessary.

Accurate sampling of particulate material is an essential prerequisite for the determination of size distribution, especially when using methods in which the test portion may be relatively small in amount. The specified procedures described in Part 1 have been selected to prevent any alteration in the size grading of the gross sample, and to avoid segregation of different sized particles when taking the increments.

No single method of size analysis can be specified to cover the many different types of powder encountered, but it is possible to formulate recommended procedures which may be applied to the majority of cases. The purpose of these standards is to obtain uniformity in procedure for any one of the methods selected, in order to facilitate comparisons between size analyses made in different laboratories.

Alternative methods of size analysis are specified because of the varying properties of the powders and the differing circumstances of usage. The conditions of test should be co-ordinated with the conditions of use. A powder used in a wet process should be tested by liquid sedimentation. A dry powder may be tested by liquid sedimentation methods, but if used in an air dispersed

* B.S. 410, 'Test sieves'.

† B.S. 1796, 'Methods for the use of British Standard fine-mesh test sieves'.

condition it may be better to analyse it by air elutriation. Such co-ordination is not always necessary or possible, but samples which are collected in the wet state must not be dried before analysis unless it can be proved that all aggregates formed on drying will be dispersed completely without any attrition of the particles.

The choice of the most suitable method will depend on the following considerations:

- the purpose for which the analysis is required, e.g. quality control, research, specification requirements;
- the size range and density of the particles;
- important properties of the powder, e.g. solubility in water, stickiness and difficulty of dispersion, agglomerating characteristics;
- the amount of material available for analysis;
- the method by which the gross sample has been collected.

The optical microscope is the only method in which direct observation is made of the size of particles. Even this method has a tendency to measure the largest dimensions unless the particles are intentionally dispersed with random orientations. The sedimentation and elutriation methods measure the free-falling velocities of the particles in a fluid. These depend not only upon the particle size, but are affected by particle shape and other properties (see Appendix D). Consequently, size analyses made on identical powders by the various methods may not show complete agreement in the size distributions, and the divergence will increase with irregularity of particle shape. It is therefore essential when reporting an analysis that the method employed be identified by its letter and number.

The results of a series of comparative tests on silica powder by different methods are given in Appendix B.

Good reproducibility does not preclude bias caused by a combination of procedure and the properties of the particles. When determinations of size distribution are made for the first time with an unfamiliar powder, the evaluation method of Appendix C should be operated if an accurate and unbiased result is required.

The apertures of B.S. test sieves are in a $\sqrt[4]{2}$ progression and it is convenient to continue this series by expressing size analyses of smaller particles in microns as cumulative percentages below sizes of a consecutive $\sqrt{2}$ series, namely:

75, 53, 37, 27, 19, 13, 9.4, 6.6, 4.7, 3.3, 2.3, 1.7.

Alternatively the international (ISO) series may be continued. This is a $\sqrt{2}$ series occupying values intermediate between those of the series quoted above, and is included in some national sieve standards, including B.S. 410.

63, 45, 31, 22, 16, 11, 7.8, 5.5, 3.9, 2.8, 2.0, 1.4.

The size distribution is expressed on a weight basis.

INTRODUCTION

Terminology. For the purpose of this standard the Terminology of B.S. 2955* applies with the addition of the following terms.

Laboratory sample. The portion of the gross sample which is delivered to the laboratory for determination of particle size distribution.

Analysis sample. The portion of the laboratory sample which is used in the size analysis apparatus.

Air or gas supply. The methods refer to air as the elutriating medium, although other gases may be used if the powders react in any way with air, provided that the values of viscosity and density appropriate to the chosen gas at the operating temperature and pressure are inserted in equation (1).

All elutriators need to be supplied with clean, dry gas at inlet (gauge) pressures usually between 5 and 15 lb/in² (0.3 — 1.0 kg/cm²). Water vapour is removed by a conventional drying train, and oil mist by filtration through an inch layer of superfine glass wool.

Basic principle. Elutriation methods are based on the measurement of the proportion of the powder which is carried off by an upward flow of gas in a vertical column at a known velocity. The largest size of particle elutriated by a given gas velocity is calculated from Stokes's law:

$$v = \frac{gd^2(\sigma - \rho)}{18\eta} \times 10^{-8} \quad (1)$$

where d = Stokes diameter† of the particle in microns, i.e. diameter of a sphere which has the same density and the same free-falling velocity in a given fluid as the particle within the range of Stokes's law;

η = absolute viscosity of gas (poises)

ρ = density of gas (g/cm³)

σ = apparent density of particle (g/cm³)

g = acceleration due to gravity (cm/s²)

v = free falling velocity of particle (cm/s)

Equation (1) is valid only in the region of viscous flow determined by the magnitude of the Reynolds number, a dimensionless quantity defined by:

$$\frac{v d \rho}{\eta} \times 10^{-4}$$

which should not exceed 0.2 if the error is not to exceed 5 per cent (See Appendix A.).

* B.S. 2955, 'Glossary of terms relating to powders'.

† See B.S. 2955 for definitions of these terms.

The minimum value of the linear gas velocity necessary to elutriate a particle of diameter d is its free falling speed v . The corresponding volume rate of gas flow F is therefore:

$$F = \frac{\pi}{4} D^2 v \quad \text{cm}^3/\text{s} \quad (2)$$

where D is the diameter of the elutriator tube (cm)

In a determination of size distribution, the velocity in the column is adjusted to carry off particles smaller than the upper limit of the smallest size fraction. The residue is weighed, and the velocity increased to carry off the next smallest fraction of the particles remaining in the residue. This process is repeated until the full size range has been covered.

Preparation of the analysis sample. The analysis sample is prepared as follows.

Air-dry the sample.

Carefully break down large aggregates.

Sieve on a 75 micron B.S. test sieve* and record the percentage weight remaining on the sieve.

Sub-divide the portion which has passed through the sieve as necessary to provide the analysis sample, using the methods of Part 1†.

Determine the density of the particles by the method described in B.S. 1377 or B.S. 3483‡.

Dry the sample at 110°C (if permissible).

Cool in a dessicator.

Disaggregate again if necessary.

Weigh the elutriator powder container.

Put the analysis sample into the container.

Re-weigh.

Dispersion. For accurate measurement of particle size distribution it is essential that the particles should be freely dispersed. The main difficulty is to break down or prevent the formation of aggregates without fracturing the individual particles. This can be achieved to some extent by the gas/solid distributor systems of the elutriators. Nevertheless, the powder must be as finely dispersed as possible before it is fed to the distributor. A method which is applicable to most, but not all, powders is to extract them successively with a dehydrated liquid, followed by highly volatile liquid, e.g. alcohol followed by ether. Mechanical dispersion can be effected by such methods as gentle rolling under a glass rod, or brushing with a short camel-hair brush. The suitability of a method for any particular powder can be ascertained only by trial experiments.

* B.S. 410, 'Test sieves', B.S. 1796, 'Methods for the use of British Standard fine mesh test sieves'.

† B.S. 3406 : Part 1, 'Subdivision of gross sample down to 0.2 ml'.

‡ B.S. 1377, 'Methods of testing soils for civil engineering purposes', B.S. 3483, 'Methods for testing pigments for paints'.

METHODS

METHOD E1. GONELL

E1.1 Principle

The Gonell elutriator* is suitable for readily dispersed particles in the size range 5–75 microns. An analysis sample of 1 to 3 ml is required.

Three tubes of different diameters are operated independently, the sizes being such that for the same volume rate of air flow passing through each, the maximum diameters of particles carried off are in the geometric ratio of 1, 2, 4. The apparatus described differs from the original Gonell apparatus in the design of the inlet injector.

E.1.2 Apparatus (See Figs. 1, 2, 3, 4)

Elutriator. The elutriator consists of three tubes 42 in (107 cm) long, the internal diameter of the tubes being $5\frac{1}{2}$ in (140 mm), $2\frac{3}{4}$ in (70 mm) and $1\frac{3}{8}$ in (35 mm) respectively. The tubes are made in brass and nickel or chromium plated internally. The inside surface shall be free from ripples. At the base of each tube is a small container which holds the analysis sample of powder to be examined. The two larger tubes are connected to the container by conical extensions.

The three tubes are set truly vertical. This is essential in order to minimize the amount of dust which may deposit on the walls of the tubes. A vibrator of robust construction shall be attached to the tube in use to assist in dislodging or preventing particles from adhering to the walls.

Air supply. A supply of clean dry air at 5 lb/in² (0.3 kg/cm²) is sufficient for most purposes.

Flowmeters. Two flowmeters covering the ranges 50–500 and 400–4000 l/h, are required.

Weighing. The balance should be sensitive to 1 mg when loaded with 100 g.

* See H. W. GONELL, *Zeit. Ver. D. Ing.* 72, 1928, p.945; and 'Richtlinien für die Bestimmung der Zusammensetzung von Stäuben nach Korngrösse und Fallgeschwindigkeit' V.D.I. Berlin, 1936.

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|---------------------|-------------------|
| A. Elutriator tube | E. Top plate |
| B. Conical adaptor | F. Deflector cone |
| C. Powder container | G. Bell jar |
| D. Screwed coupling | H. Ring |

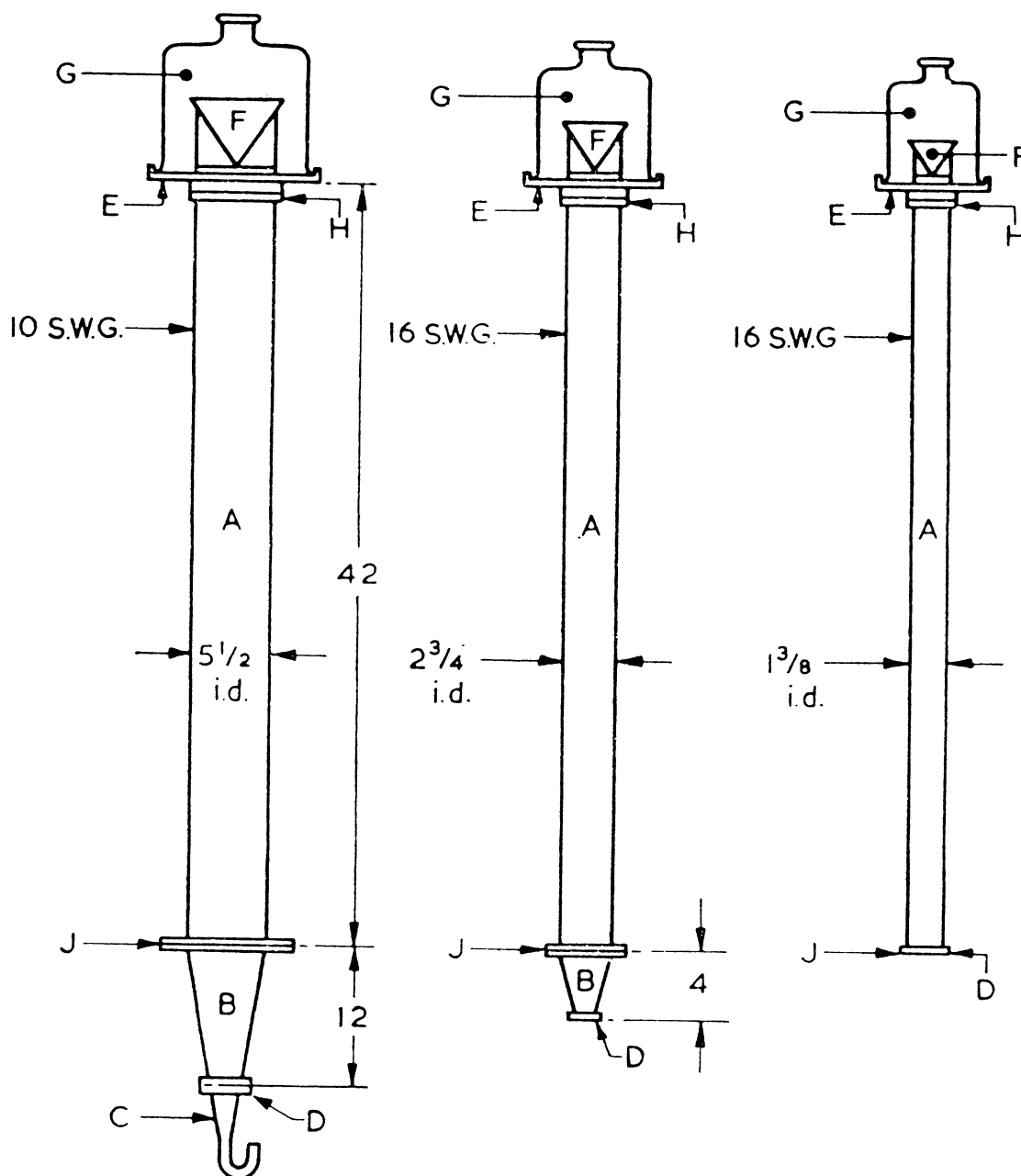


Fig. 1. Gonell elutriator assembly
Dimensions in inches.