

Methods for

Determination of particle size distribution —

**Part 5: Recommendations for
electrical sensing zone method
(the Coulter principle)**

UDC 620.168.36:66 – 492.2

Cooperating organizations

The Chemical and Petroleum Engineering Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following:

Association of Consulting Engineers	Department of Trade (Marine Division)
British Chemical Engineering Contractors' Association	Energy Industries Council
British Gas Corporation	Engineering Equipment Users' Association
British Rubber Manufacturers' Association	Glass Manufacturers' Federation
British Steel Industry	Health and Safety Executive
Chemical Industries Association	Institution of Chemical Engineers*
Coke Oven Managers' Association	Institution of Gas Engineers
Department of Energy [Petroleum (Oil)]	Institution of Structural Engineers
Department of Industry	Oil Companies Materials Association
	Process Plant Association*

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

AERE Harwell	Incorporated National Association of British and Irish Millers
Bcira	Institution of Mining and Metallurgy
British Ceramic Society	Mechanical Handling Engineers' Association
British Laboratory Ware Association	Ministry of Defence
British Quarrying and Slag Federation	National Coal Board
Brunel University	Pharmaceutical Society of Great Britain
Cement and Concrete Association	Royal Society of Chemistry
Cement Makers' Federation	Society of Chemical Industry
China Clay Association	Society of Cosmetic Scientists
Coated Abrasives Manufacturers' Association	Sunderland Polytechnic (Magnet Centre)
Department of Industry (National Physical Laboratory)	University of Bradford
Department of Industry (Warren Spring Laboratory)	University of Salford
Department of Transport	University of Technology
Guild of Metal Perforators	Wellcome Foundation
	Woven Wire Association

This British Standard, having been prepared under the direction of the Chemical and Petroleum Engineering Standards Committee, was published under the authority of the Board of BSI and comes into effect on 30 September 1983

© BSI 12-1999

The following BSI references relate to the work on this standard:
Committee reference CPE/10
Draft for comment
81/74136 DC

ISBN 0 580 13244 7

Amendments issued since publication

Amd. No.	Date of issue	Comments
4535	April 1984	
7976	February 1994	Indicated by a sideline in the margin

Contents

	Page
Cooperating organizations	Inside front cover
Foreword	iii
0 Introduction	1
1 Scope	1
2 Definitions	1
3 Symbols and abbreviations	2
4 Health and safety	3
5 Principle	3
5.1 General operation	3
5.2 Response theory	4
5.3 Effect of particle shape and orientation	4
5.4 Effect of coincident particle passage	6
5.5 Pulse shape	6
5.6 Size limits	7
6 Operational procedures	7
6.1 Instrument location	7
6.2 Choice of electrolyte solution	7
6.3 Electrolyte solution preparation	8
6.4 Sample preparation and dispersion	8
6.5 Choice of orifice(s) and manometer setting(s)	9
6.6 Stability of dispersion	9
6.7 Clearing an orifice blockage	10
6.8 Analytical procedure	10
6.9 Background counts	12
6.10 Calibration	12
6.11 Multiple orifice method for wide size range samples	13
6.12 Calculation of results	14
6.13 Extrapolation	17
6.14 Determination of mass percentage detected	18
7 Reporting of results	22
Appendix A Comparison with other methods	23
Appendix B List of suitable electrolyte solutions	23
Appendix C Instrument checks	31
Appendix D Measurement of the immersed density of particles of powder when dispersed in electrolyte solution	32
Appendix E Example of calibration by mass integration	33
Appendix F Example of determination of percentage mass analysed	34
Appendix G Large orifice technique (orifice diameter $\geq 560 \mu\text{m}$)	34
Appendix H Bibliography	35
Figure 1 — Schematic drawing of a simple form of analyser	5
Figure 2 — Pulses	7
Figure 3 — Current density at orifice	7
Figure 4 — Pulses resulting from axial and non-axial passage of particles	8
Figure 5 — 2-orifice overlap	18
Figure 6 — Example of constant vacuum supply	35
Table 1 — Background counts	9
Table 2 — Suggested manometer settings	9
Table 3 — Stability of suspension	9

	Page
Table 4 — Typical coincidence correction chart	11
Table 5 — Recommended number of counts	11
Table 6 — Suitable orifice diameter combinations	13
Table 7 — Data sheet: normal calculation	15
Table 8 — Data sheet: worked example without corrections	16
Table 9 — Data sheet: large orifice analysis	19
Table 10 — Data sheet: small orifice analysis	20
Table 11 — Worked example (for a 2-orifice analysis)	21
Table 12 — Recommended electrolyte solutions	25
Table 13 — Density of pure water, ρ_w	32
Table 14 — Data for calibration by mass integration	33
Publications referred to	Inside back cover

Foreword

This new Part 5 of BS 3406 was prepared under the direction of the Chemical and Petroleum Engineering Standards Committee and is one of a series that describes methods for determining the size distributions of particles. 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 (obsolescent);*
- *Part 4: Guide to microscope and image analysis methods;*
- *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.*

The particle size distribution of a powder is an important basic property that can distinguish it from other samples of the same composition. Vast quantities of powdered materials are sold or processed and specifications often include average particle size and/or particle size distribution. Even when other bulk properties are specified, compliance with the specification may be achieved by varying the particle size distribution. Particle size analysis is also used extensively to monitor environmental conditions and in numerous areas of scientific research. In all these contexts the reproducibility and comparability of results is of paramount importance and the use of standardized procedures is essential.

Representative sampling and correct dispersion of particulate material are prerequisites for the determination of size distribution. The procedures described in BS 3406-1 have been selected to give test portions representative of the bulk.

The techniques used to measure particle size distribution are many and are varied both in principle and in their degree of sophistication and automation. In principle, any property that depends on particle size can be used to define particle size. In practice, the methods that are widely used are both experimentally convenient and based upon a physical principle having a well defined relationship to particle size.

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

- a) the purpose for which the analysis is required, e.g. quality control, research, specification requirements;
- b) the size range and other properties of the particles, e.g. density, solubility, refractive index;
- c) aggregation and dispersion characteristics of the powder;
- d) the amount of material available for analysis;
- e) the method by which the sample has been collected;
- f) the conditions under which the powder will be used, for example the state of dispersion in the test should be related to the state of dispersion in the application;
- g) resources available.

The optical microscope method is the only method in which direct observation is made of the size and shape of the particles. Most other methods of particle size measurement ascribe a single dimension to each particle, usually expressed as the diameter of the spherical particle equivalent to some aspect of its properties, such as volume, surface, settling velocity or light scattering phenomena. Physical properties of the particles, such as solubility, friability, refractive index and conductivity, preclude the use of some methods. Nevertheless the same powder can be analysed using a range of techniques and if different size parameters are determined different size distributions will result. The intercomparison of results can only be reliable if each laboratory is using a representative sample and is measuring the same parameter by reliable equipment and procedures. Standardization between laboratories is best established by the use of certified reference materials, for example those of the Community Bureau of Reference (BCR) (see Appendix A).

The electrical sensing zone method is a widely used method of particle size analysis as large numbers of particles can be counted and sized at high speed with precision and accuracy. The external volume of each particle is measured independently of the density and refractive index of the particle. Response is affected only to a small extent by the shape and resistivity of the particle, and these effects can be minimized by suitable calibration. A limitation to the method is that the particles have to be suspended in an electrically conducting solution.

It has been assumed in the drafting of this British Standard that the execution of its provisions is entrusted to appropriately qualified and experienced people, for whose guidance it has been prepared.

WARNING. This British Standard calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. Attention is drawn to the specific hazards described in clause 4.

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 36, 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.

0 Introduction

The electrical sensing zone method of particle size analysis [1, 2, 3]¹⁾ is based on the following principle.

Particles, suspended at low concentration in an electrolyte solution, are detected upon their passage through a small orifice in an insulating wall by the modulation of an electrical current passing through the orifice. This electrical modulation is sensed as a series of voltage and/or current pulses which are then amplified and processed by appropriate circuitry for single size-band counting and/or multi-channel pulse height (size distribution) analysis.

Pulse amplitude is essentially proportional to the volume of the particle, is independent of particle density and, to a great extent, independent of particle conductivity or shape. The only basic limitation is that the particles to be measured have to be suspended in an electrolyte solution. In practice, most materials having particles below approximately 250 µm in diameter can be measured using electrolytes in water or organic solvents giving solutions of suitable viscosity. (See also 5.6.)

Practical considerations are:

- a) the orifice diameter should be at least twice that of the largest particles to reduce blockages and non-linear size response;
- b) particle concentration should be low to reduce coincident passage through the orifice;
- c) uniform particle dispersion should be maintained.

The approximate size range covered by each orifice is restricted to about 2 % to about 60 % of the diameter of the orifice, smaller particles being lost in electronic noise generated within the orifice and the electronic circuitry, and larger particles increasing the chance of blockage of the orifice. Some typical materials require two or more orifices to cover the wide range of sizes present, analogous to a microscope analysis where two or more magnifications are necessary.

The electrical sensing zone technique is widely used for sizing particles from 0.6 µm diameter upwards but smaller particles can be measured if care is taken. Typical repeatability of analysis of the same sample dispersion will be within an average of ± 1 % by mass (1 standard deviation) at any point on the size distribution.

The technique can be applied to a wide variety of powders, suspensions, emulsions, airborne environmental pollutants and particulate contaminants in clean fluids. It is a traceable method and also facilitates comparison between size analyses made in different laboratories. It can also be applied to haematology and cell biology, which are not covered at present by other Parts of BS 3406; however, these specialized techniques are outside the scope of this Part of this standard.

Because of the great variety of instrument designs, it is not possible to cover all of the operating procedures, and the examples given in this standard are not comprehensive. Manufacturers' manuals should be consulted for specific details.

1 Scope

This Part of this British Standard gives recommendations on the electrical sensing zone method for the determination of the particle size of powders, suspensions and emulsions, and facilitates comparison between size analyses made in different laboratories.

Specialized techniques applicable to haematology, cell biology, environmental pollution and determination of particulate contaminant in clean fluids are outside the scope of this Part of this standard.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover. Bibliographic references are listed in Appendix H.

2 Definitions

For the purposes of this standard, the following definitions, together with those given in BS 2955, apply.

2.1

spatulation

the gentle kneading with a flexible spatula of material to which a few drops of dispersant have been added

2.2

immersed density

the mass of powder per unit volume of suspension medium it displaces

2.3

envelope volume

external volume of a particle such as would be obtained by tightly shrinking a film to contain it

¹⁾ The numbers in square brackets used throughout the text of this standard refer to the bibliographic references given in Appendix H.

3 Symbols and abbreviations

3.1 Symbols. For the purposes of this standard, the following symbols apply.

Symbol	Quantity	Unit	Symbol	Quantity	Unit
A	attenuation control reading	dimensionless	ΔN	number of particles in a size interval	—
A_o	cross-sectional area of orifice	μm^2	N_1	number of particles corresponding to d_1	—
a	cross-sectional area of particle	μm^2	N_2	number of particles corresponding to d_2	—
$\bar{a}$	average cross-sectional area of particle	μm^2	n	observed particle count	—
B	background count	dimensionless	$\bar{n}$	average number of particles above each size level	—
b_i	a series of constants for equation 4	dimensionless	n'	number of particles not counted owing to coincident particle passage	—
D	diameter of orifice	μm	p	coincidence factor	dimensionless
d	diameter of a spherical particle	μm	p_1	orifice factor	dimensionless
$\bar{d}$	$(d_1 + d_2)/2$	μm	ΔR	change in resistance of orifice due to the presence of a particle	Ω
d_1	a value of d_v as in Table 7, Table 8, Table 9 and Table 10	μm	S_m	mass specific surface area of a powder	m^2/kg
d_2	the next smaller value of d_v in Table 7, Table 8, Table 9 and Table 10	μm	T	electrolyte solution temperature	$^{\circ}\text{C}$
$d_{\max}$	equivalent volume diameter of largest particle measurable with any given orifice	μm	t	lower threshold control reading	—
$d_{\min}$	equivalent volume diameter of smallest particle measurable with any given orifice	μm	V	particle volume	dimensionless (arbitrary)
d_s	surface median diameter	μm	$\bar{V}$	arithmetic mean volume for a particular size interval	dimensionless
d_{sv}	surface volume mean diameter	μm	V_1	volume corresponding to d_1	dimensionless
d_v	equivalent volume diameter of particle	μm	V_2	volume corresponding to d_2	dimensionless
d_w	volume/mass median diameter	μm	ΔV_L	volume of particles in a size channel for a large orifice 2-channel analysis	cm^3
F	has the value of a series (see equation 4)	—	V_m	manometer volume	cm^3
I	current control reading	—	V_o	volume of orifice	cm^3
K_d	diameter calibration constant	dimensionless	V_s	volume of sample in metered volume V_m	cm^3
K_{dL}	diameter calibration constant for the larger orifice	dimensionless	V_T	total volume of electrolyte solution used	cm^3
K_{ds}	diameter calibration constant for the smaller orifice	dimensionless	V_z	sensing zone volume	cm^3
L	length of orifice	μm	W	total mass of sample used	g
δL	thickness of element of particle	μm	W_1	mass of pure water to fill a density bottle at the selected operating temperature	g
N	true particle count	—	W_2	mass of electrolyte/dispersant solution to fill a density bottle at the selected operating temperature	g
			W_3	W_2 plus the mass of the empty density bottle	g
			W_4	mass of sample used to determine the immersed density of a powder	g

Symbol	Quantity	Unit
W_5	W_4 plus the mass of the empty density bottle and the electrolyte/dispersant solution to fill the bottle at the selected operating temperature	g
W_A	calculated mass of sample in metered volume	g
W_M	actual mass of sample in metered volume	g
ρ	immersed density of sample as determined in electrolyte	kg/m ³ , g/cm ³
ρ_L	density of electrolyte/dispersant solution at the selected operating temperature	kg/m ³
ρ_w	density of pure water during the calibration of a density bottle at the selected operating temperature	kg/m ³
σ_g	geometric standard deviation	dimensionless
ϕ	resistivity of electrolyte solution	Ω m

3.2 Abbreviation. The following abbreviation used in this standard should not be confused with the use of V given in 3.1.

V/V volume per volume

4 Health and safety

4.1 Electrical. In electrical sensing zone particle size analysers, voltages of up to 110 V, and even up to 300 V d.c., can exist across the electrodes, although at low current. The outer electrode or beaker contents should not be touched while the mercury manometer is unbalanced and the current supply is on. The orifice current should be turned off when trying to clear an orifice blockage.

NOTE Some models have protection against this hazard.

4.2 Chemical. Electrolyte solutions used in the method may sometimes be harmful and normal laboratory procedures should be observed in their preparation and use [4], for instance, some organic solvents [e.g. electrolyte solution nn (see Appendix B)] should be handled only in a fume cupboard, into which the instrument and/or glassware assembly is fitted.

Do not mix strong oxidizing agents, such as chromium (VI) trioxide, nitric acid, chlorates, perchlorates or permanganates, with glycerol or acetone.

Do not mix dimethylformamide with chlorinated hydrocarbons.

Do not add sodium azide to acidic solutions, as harmful hydrazine gas will be liberated.

4.3 Mercury. The manometer contains mercury which, while contained, presents no hazard. Should spillage occur, however, it is important that proper precautions for the protection of laboratory personnel are taken. Attention is drawn to the relevant regulations and guidance documents, which include the Health and Safety Executive Guidance Note EH17, published by HMSO.

5 Principle

5.1 General operation. Figure 1 is a schematic diagram of a simple form of analyser. A dilute suspension of particles dispersed in an electrolyte solution is stirred to provide a homogeneous mixture and is drawn through a small orifice in an insulating wall via tap T open to a vacuum. At the same time the mercury manometer becomes unbalanced. Closing the tap T isolates the orifice from the external vacuum; suspension continues to be drawn through the orifice as the mercury returns to equilibrium. A current applied across the two electrodes enables the particles to be sensed by the momentary impedance changes in the electrical path across the orifice.

Start and stop contacts in the mercury column enable particles in a precise volume of suspension to be counted. The particle-generated pulses are amplified and compared with a scaling circuit such that pulses reaching or exceeding a pre-settable amplitude or threshold are recorded on a digital counter. By taking a number of measurements at different amplitudes, a distribution of a number of particles against volume or equivalent diameter is obtained. The size parameter is usually expressed as the diameter of a sphere of equal volume to that of the particle.

In single channel models, data are converted to mass distribution manually. In more complex models, various forms of pulse height analysis are used to accumulate results simultaneously at a number of size levels. Between 16 and 256 channels, or size intervals, are common and such analysers are called multi-channel models. They allow the accumulation of size distributions in a very short time, and to save further time it is usual to analyse samples at lower than normal concentration levels to simplify calculations by ignoring coincidence effects (see 5.4).

Some multi-channel analysers convert raw number/size data to mass/size by Simpson's rule of integration, others by accumulation of individual particle volumes in each size interval, which is more accurate for narrow size ranges or with unusual particle size distributions. The main advantages of multi-channel analysis are the saving of operator time by removing repetitive functions, and the convenience with which the results can be presented by using internal or external computers. Some models present the volume (mass) percentage size distribution directly and graphically.

Analysis time is typically between 2 min and 30 min, depending on the model and the size range of the particles being analysed.

5.2 Response theory. An assumption underlying the operation of electrical sensing zone analysers is that the response, i.e. the pulse generated when a particle passes through the orifice, is directly proportional to the particle volume.

Theoretically, the response depends on the resistivity of the particle but this is rarely observed in practice. This is probably due to oxide surface films, ionic inertia of the Helmholtz electrical double layer or associated solvent molecules at the particle surface making the apparent resistivity of almost all types of particles extremely large.

Several theoretical models have been proposed for the electrical sensing zone method [5, 6, 7, 8]. The following approximate theory is used for its ease of understanding.

Ignoring the resistivity of the particle, the change in resistance δ (ΔR) of an element of particle of thickness δL along the axis of the orifice is given by

$$\delta(\Delta R) = \phi a \delta L / A_o^2 \left(1 - \frac{a}{A_o} \right) \quad (1)$$

where

- a is the cross-sectional area of the particle in a plane parallel to the orifice;
- A_o is the cross-sectional area of the orifice;
- ϕ is the resistivity of the electrolyte solution.

The external resistance in the circuit is sufficiently high to ensure that the small change ΔR in the resistance of the orifice due to the presence of a particle will not affect the current I ; the voltage pulse generated is therefore $I\Delta R$.

The response is not directly proportional to the particle volume element but is modified by the term a/A_o , according to the theoretical model used above.

With rod shaped particles whose length is smaller than the orifice length this leads to an oversizing of about 6 % in terms of diameter at the upper limit of each orifice with distortion of the measured size distribution. The error decreases as a/A_o decreases.

For a spherical particle of diameter d in an orifice of diameter D the complete geometric solution is

$$\Delta R = - \frac{4\phi}{\pi D} \left\{ \frac{\sin^{-1}(d/D)}{1 - (d/D)^2} - \frac{d}{D} \right\} \quad (2)$$

This equation may be written

$$\Delta R = - \phi VF / A_o^2 \quad (3)$$

where

V is the particle volume;

F has the value of a series, such that

$$F = 1 + \sum_{i=1}^{i=j} b_i (d/D)^i \quad (4)$$

Various experimental and theoretical values have been proposed for the constants b_i .

However, practical experiments show that there is linearity of volumetric response, for the spherical latex test particles and instrument model used, at least up to some 80 % of the orifice diameter [11].

5.3 Effect of particle shape and orientation.

Much experimental evidence indicates that the response of electrical sensing zone analysers is essentially proportional to particle volume. However, it is known that particles of extreme shapes, e.g. flakes, are oversized significantly, and some very clean metal particles have been found to conduct at moderate applied orifice voltages (≈ 10 V), leading to undersizing. There is evidence that the size parameter measured is the envelope volume of the particle; for instance with porous particles the measured volume may be several times the skeletal volume, and the total volume of flocculated particles measured is greater than exists in reality.

Model experiments have been carried out by several investigators but no firm conclusions may be drawn from them since the models used differed widely from the commercial instruments.

Correlation with other techniques has been found to be good for spherical particles; for non-spherical particles the results may differ. For irregular particles, size distributions obtained by different techniques will inevitably differ unless the size parameter measured is the same (see Appendix A).

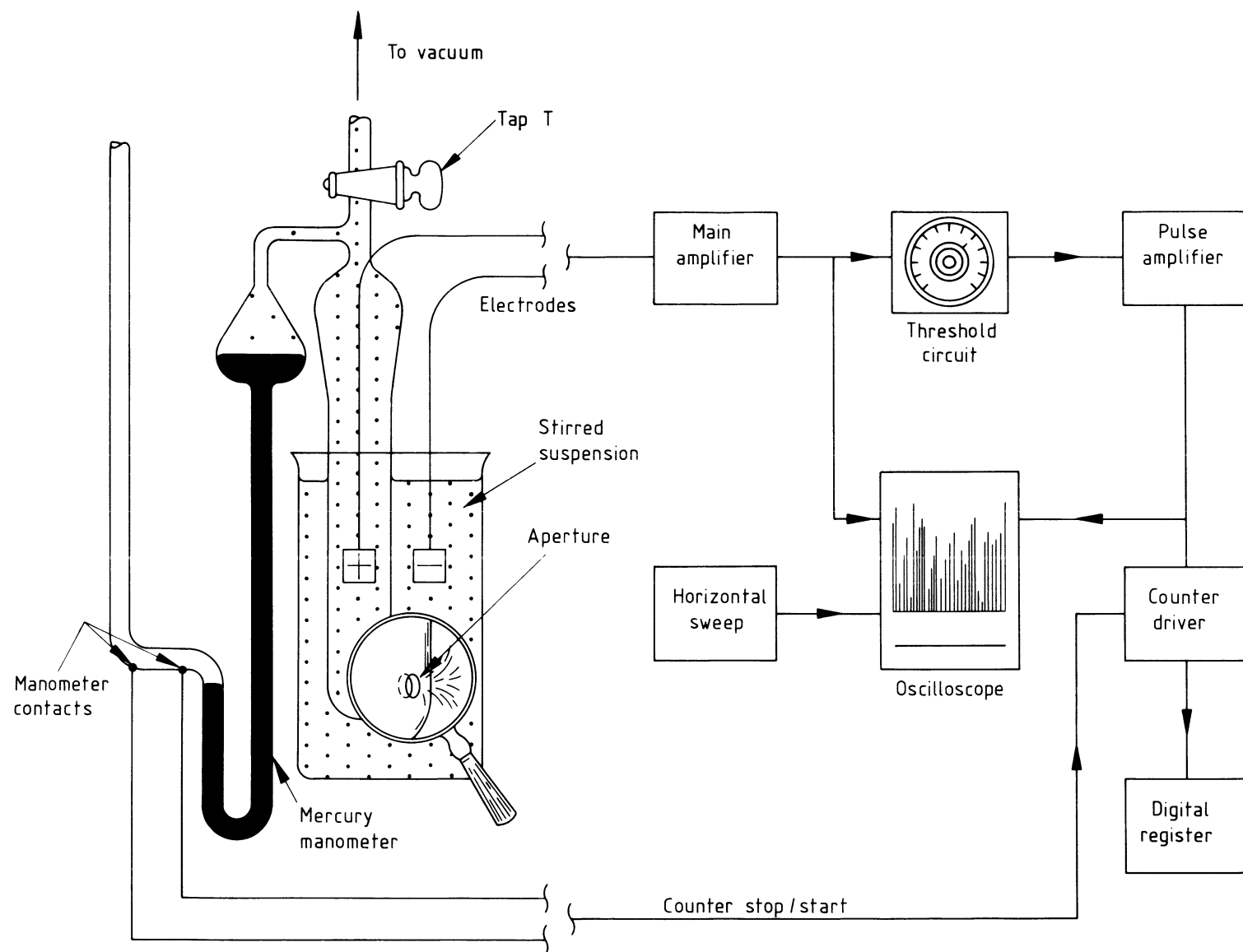


Figure 1 — Schematic drawing of a simple form of analyser

For these reasons it is recommended that, to ensure the maximum accuracy, the analysers should be self-calibrated once with the type of material under test. Standard uniform particles, such as latex, may then be given a reference size relative to the material under test and used for all subsequent calibration checks. It is also possible to calibrate for particle length or diameter as, for instance, in fibre analysis.

For fibre diameter, straight fibres are microtomed to constant length, and thus the instrument response becomes proportional to cross-sectional area, from which the fibre diameter (denier) distribution can be calculated. Fibre length may be measured by detecting the duration, not the amplitude, of the pulse produced on passage through the orifice.

5.4 Effect of coincident particle passage. If it is assumed that ideal data would result if particles traverse the orifice singly, two types of error result due to deviation from this condition.

- a) *Primary coincidence.* Two or more particles in the sensitive zone about the orifice give rise to two or more overlapping pulses. Depending on proximity and electronic resolution these pulses may not be resolved, resulting in loss of count.
- b) *Secondary coincidence.* Two or more very close particles, which individually give pulses below the threshold level, can collectively give a single pulse above the threshold level.

Both errors are minimized by using extremely dilute suspensions. The effect of primary coincidence can then be corrected for. If primary coincidence is kept low, secondary coincidence is less significant as it can occur only if the particles are of similar size and close to the threshold limit.

The correction for primary coincidence may be theoretically determined by assuming a Poisson probability of finding a number of particles concurrently in the sensing zone. This yields relationships between the true count N and observed count n

$$n = \frac{V_m}{V_z} \{1 - \exp(-V_z N/V_m)\} \quad (5)$$

$$N = n + (V_z N^2 / 2V_m) \quad (6)$$

where

V_m is the volume monitored for each count;

V_z is the sensing zone volume.

In practice, it is more convenient to base the correction on the observed count n and the equation commonly used is

$$N = n + p (n/1000)^2 \quad (7)$$

where p is the coincidence factor, which can be calculated from

$$\begin{aligned} p &= 2.5 (D/100)^3 (0.5/V_m) \\ &= 1.25 \times 10^{-6} D^3/V_m \end{aligned} \quad (8)$$

where D is the orifice diameter.

The factor 2.5 was determined experimentally using a 100 μm diameter orifice 75 μm long. If other length-to-diameter ratios are used, this sensing zone factor (p_1) should be calculated from the following equation based on Auerbach's electrical resistance of a tube of comparable length L and diameter D :

$$p_1 = \pi D^2 (L + 0.8D) / 4V_0 \quad (9)$$

where V_0 is the volume of the orifice.

NOTE For standard orifices of 70 μm to 400 μm , the L/D ratio is approximately 0.75; for smaller sizes it ranges from 1 to 4.

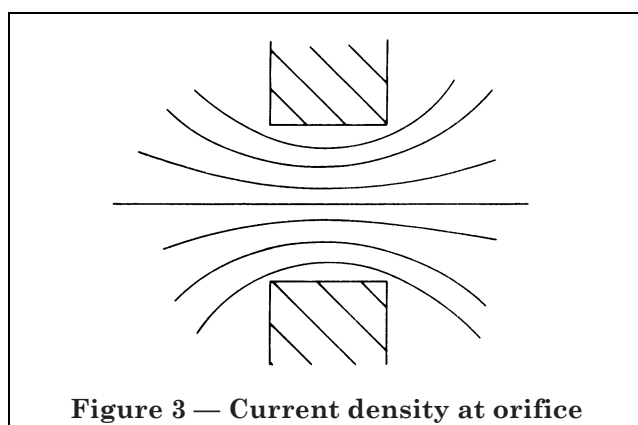
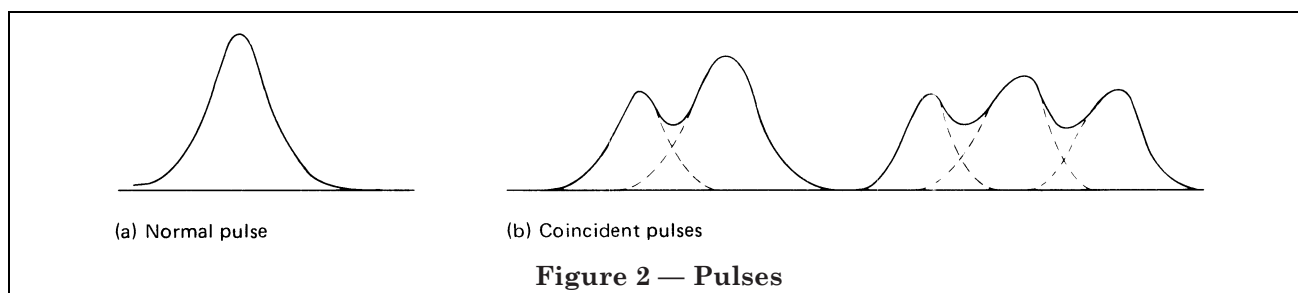
For larger sizes it is specified by the manufacturer.

5.5 Pulse shape. Pulses will deviate from the ideal, uniform, single modal shape when they are generated from coincident particles and/or from particles travelling non-axially through the orifice. Coincident pulses may take on a variety of shapes, always of longer duration and often having multiple peaks. Examples are shown in Figure 2.

If the particle passes non-axially through the orifice, it passes areas of higher than normal current density around the edges of the orifice producing a pulse larger than expected (see Figure 3 and Figure 4).

These artefactual pulses cause oversizing of those particles. This is seen as a skewing of a narrow size range particle distribution, such as latex or blood cells, to the coarse side. In wider size range materials, such as most industrial powders, the error in size distribution is not observable.

For particles of narrow size range ($\leq 3:1$ by diameter) there are several methods of improving accuracy. The pulses can be electronically "edited" (the most distorted ones being rejected from the size distribution), they can be electronically reshaped (to provide information from all pulses) or, most satisfactory of all, they can be produced in a hydrodynamically focussed (HDF) orifice. In an HDF orifice, nearly all particles are forced to flow centrally into and through the sensing zone so that the pulses are correctly formed.



5.6 Size limits. The lower size limit of the electrical sensing zone method is generally considered to be restricted only by thermal and electronic noise. It is normally stated to be about $0.6\ \mu\text{m}$, but under favourable conditions a lower limit is possible. There is no theoretical upper limit and for particles having a density similar to that of the electrolyte solution the largest orifice normally available ($2\ 000\ \mu\text{m}$) may be used.

When the particle density is high, the upper size limit is reached when the particles can no longer be kept in suspension. In this case, the electrolyte solution has to be greatly increased in viscosity and/or density, for example, by the addition of glycerol or sucrose (see Appendix B). In addition, round bottomed beakers, baffles and double bladed stirrers may be used to maintain suspension. Alternatively, the whole sample may be passed through the orifice.

6 Operational procedures

6.1 Instrument location. The instrument should be sited in a clean environment that is free from excessive electrical noise and vibration. If organic solvents are to be used the area should be well ventilated.

For particle size analysis below about $2\ \mu\text{m}$, it may be necessary to put the instrument into a dust-free room or a clean air cabinet.

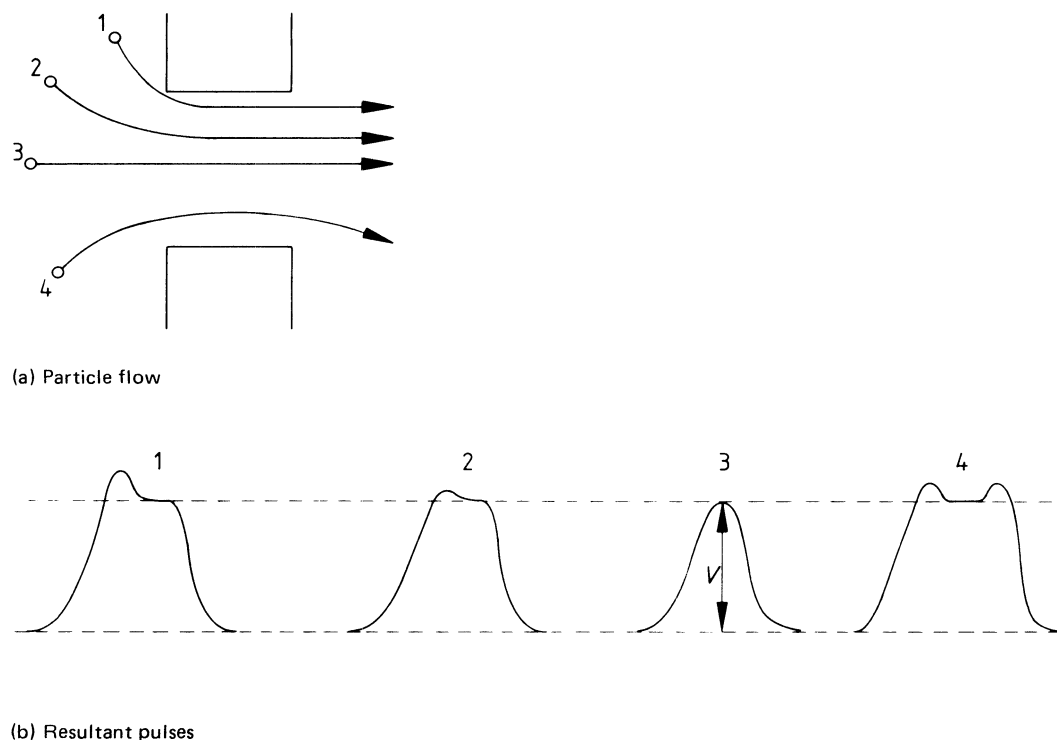
Electrical background noise (see C.3) or interference can cause extra counts. This may be reduced by operating the instrument within an earthed Faraday cage. Mains transmitted noise can be reduced with an isolating transformer.

6.2 Choice of electrolyte solution. An electrolyte solution should be selected in which the sample is insoluble. The electrolyte solution should not dissolve, flocculate, react or otherwise interfere with the state of dispersion of the sample in the measurement time, typically up to 15 min.

Particles insoluble in water can be analysed in aqueous electrolytes, such as 50 g/L hydrated trisodium orthophosphate solution, or 10 g/L sodium chloride solution. Particles soluble in water can often be analysed in 50 g/L lithium chloride solution in methanol, or 50 g/L ammonium thiocyanate solution in acetone.

Particles sparingly soluble in electrolyte solution may be suspended in the solution saturated with sample material and filtered. The saturated solution should not be left standing and the analysis should be carried out as rapidly as possible. The temperature should be constant as there may be a tendency for large particles to grow at the expense of small ones or desaturation could occur. The common ion effect can sometimes be used to preserve the integrity of the material being analysed, e.g. barium sulphate particles may be sized in an aqueous 50 g/L sodium sulphate solution.

The determining factor for the concentration of the electrolyte is the orifice resistance, which should be in the range of $5\ \text{k}\Omega$ to $100\ \text{k}\Omega$. For small orifices in the $10\ \mu\text{m}$ to $30\ \mu\text{m}$ range, “normal” electrolyte concentrations may often be increased to yield better resolution of particle pulses from electronic noise for particle sizes below $1\ \mu\text{m}$ to $2\ \mu\text{m}$; e.g. increase 10 g/L sodium chloride solution to 40 g/L to 60 g/L, and 20 g/L trisodium orthophosphate solution to 60 g/L to 80 g/L. Any adverse effects of such higher electrolyte concentrations (e.g. flocculation) may be checked as recommended in 6.6.



NOTE Pulses correspond to those shown in (a). Pulse 3 is a normal pulse, corresponding to particle volume V .

Figure 4 — Pulses resulting from axial and non-axial passage of particles

NOTE Appendix B lists many electrolyte solutions that have been found suitable for a wide range of materials. Instrument manufacturers will be able to recommend others.

6.3 Electrolyte solution preparation. The electrolyte solution should be well filtered before use as it is essential that its background count should be as low as is practicable. The glassware used should be pre-rinsed with filtered electrolyte solution.

Initially, the solution should be filtered using either a ceramic filter, a No. 5 sinter or a glass fibre paper, to remove most particles larger than about $2\ \mu\text{m}$. This solution is then passed twice through a $0.8\ \mu\text{m}$ membrane filter for use with orifices larger than $50\ \mu\text{m}$. For use with smaller orifices, filter twice at $0.45\ \mu\text{m}$ and in extreme cases twice more at $0.22\ \mu\text{m}$. If small volumes of filtrate are required it is usual to omit the first stage and filter directly through the $0.8\ \mu\text{m}$ or $0.45\ \mu\text{m}$ membrane.

Filter non-aqueous electrolyte solutions through glass-fibre filters with a glass sinter support and for the final filtration use solvent-resistant membranes.

To save time when filtering, stack successively smaller filters in a filter unit.

If aqueous electrolyte solutions are to be stored, a preservative should be added before filtration to prevent micro-organism growth.

Background counts should not exceed the values shown in Table 1 at the smallest size level to be used ($d_{\min}$).

6.4 Sample preparation and dispersion. The sample should be subdivided to about $0.2\ \text{cm}^3$ by one of the methods listed in BS 3406-1.

NOTE Confidence in the sampling efficiency may be increased by comparing the results of two complete analyses on separate samples.