BRITISH STANDARD

BS 5540-6: 1990

# Evaluating particulate contamination of hydraulic fluids —

## Part 6: Method of calibrating liquid automatic particle-count instruments (using mono-sized latex spheres)

Confirmed February 2011

# Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Machinery and Components Standards Policy Committee (MCE/-) to Technical Committee MCE/18, upon which the following bodies were represented:

Advanced Manufacturing Technology Research Institute
Association of British Mining Equipment Companies
Bath University Library
British Compressed Air Society
British Fluid Power Association
British Steel Industry
Department of Trade and Industry (National Engineering Laboratory)
Institution of Mechanical Engineers
Ministry of Defence

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

British Hydromechanics Research Association
British Railways Board
Society of Motor Manufacturers and Traders Ltd.

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# Contents

# Foreword

This Part of BS 5540 has been prepared under the direction of the Machinery and Components Standards Policy Committee and is one of a series relating to the evaluation of particulate contamination in hydraulic fluids.

This Part of BS 5540 describes a method of calibrating liquid automatic particle counters working on the light obscuration principle, using mono-sized latex spheres.

Other Parts in the series are as follows.

— *Part 1: Qualifying and controlling of cleaning methods for sample containers;*

— *Part 2: Method of calibrating liquid automatic particle-count instruments (using AC Fine Test Dust contaminant);*

— *Part 3: Methods of bottling fluid samples;*

— *Part 4: Method of defining levels of contamination (solid contamination code);*

— *Part 5: Method of reporting contamination analysis data.*

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**Summary of pages**

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, 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

In hydraulic fluid power applications power is transmitted and controlled through a liquid under pressure within an enclosed circuit. The liquid is both a lubricant and power-transmitting medium.

The presence of solid contamination particles in the liquid interferes with the ability of the fluid to lubricate and causes wear to the hydraulic components. The extent of contamination in the liquid has a direct bearing on the performance and reliability of the system and has to be controlled to levels that are considered appropriate for the system.

Quantitative determination of particulate contamination requires precision in obtaining the sample and determining the extent of contamination. The liquid automatic particle counter working on the light obscuration principle has become an accepted means of determining the extent of contamination.

The accuracy of repeatability and reproducibility of particle count data is affected by both the procedures used and the method of calibration.

## 1 Scope

This Part of BS 5540 describes a method for the calibration of liquid automatic particle counters (APCs) working on the light obscuration principle. It establishes a uniform and precise method of calibrating these instruments which will enhance the degree of reproducibility of particle count data between laboratories.

The calibration material used is mono-sized latex spheres whose size can be verified by a number of different methods. The particle size parameter reported is the equivalent optical diameter based upon the projected area of a spherical particle.

The method described first confirms the maximum concentration limits of the instrument being calibrated, verifies the particle size distribution of the latex spheres and, finally, enables the instrument to be calibrated in an accurate and precise manner.

The calibration curve of an APC is the reference that relates the particle size to the electrical threshold settings of the instrument. The development of the calibration curve is of primary importance in establishing the accurate sizing of particles being counted. A procedure is described that enables the voltage threshold value for any particle size within the sizing range of the sensor to be obtained.

This method is intended for application to any APC working on the light obscuration principle in which particles interrupt the passage of light from a source to a detector. It assumes that the counter is equipped with at least two channels with adjustable voltage thresholds.

Although this Part of BS 5540 is intended for fluid power applications, it may also be applicable to other fields where this method of calibration is considered appropriate.

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

## 2 Definitions

For the purposes of this Part of BS 5540 the following definitions apply.

**2.1**
**agglomerate**

two or more particles that are in intimate contact and can not be separated by the small shear forces generated by gentle stirring

**2.2**
**channel**

a particle size range as set into the liquid automatic particle counter

**2.3**
**coincidence**

the presence of more than one particle within the sensing volume at the same time

**2.4**
**dynamic sizing range**

the diameter of the smallest particle detected compared to the diameter of the largest particle passing through the sensor

NOTE This is usually quoted by the instrument manufacturer.

**2.5**
**equivalent optical diameter**

the diameter reported by the counting/sizing device in use. It is the diameter of a sphere giving the same optical response as the particle

**2.6**
**light obscuration**

the reduction of intensity of a light beam through the sensing volume caused by adsorption and/or scattering of the light by a single particle

**2.7**
**minimum particle size**

the diameter of the smallest latex sphere used in the calibration procedure that is also within the size range that can be resolved by the counter

**2.8**
**mono-sized latex spheres**

spherical polymeric calibration material having a narrow size range which is essentially Gaussian in distribution and having a coefficient of variation of typically less than 12 %

**2.9**
**"noise" level**

the minimum voltage setting of the instrument's detection circuit below which spurious signals of electrical noise become significant and are recorded as particles

**2.10**
**saturation level**

the maximum counting rate above which the instrument will miscount

NOTE 1 The saturation level is usually specified by the manufacturer of the instrument.
NOTE 2 A procedure for checking the saturation level is described in **6.3**.

**2.11**
**sensing volume**

the illuminated volume through which particles pass and can be detected

**2.12**
**sensor**

a device through which the sample to be measured is passed and which contains the sensing volume

**2.13**
**voltage threshold levels**

voltage levels representing the passage of reference particles of specific sizes against which the particle pulse is compared

NOTE Adjustment is usually made using potentiometers.

## 3 Apparatus

**3.1** *Liquid automatic particle counter (APC),* working on the light obscuration principle, fitted with a sensor suitable for counting within the specified range.

NOTE The particle counter should include an automatic bottle sampling apparatus or a similar means of allowing for the passage of liquid directly to the sensor and then to a measuring vessel. The air or gas used to pressurize the sample chamber causing sample flow through the sensor should be filtered through a 0.45 $\mu$m membrane filter and should be free from oil and water.

**3.2** *Vacuum apparatus,* for filtering the various liquids used in the procedure via a 0.45 $\mu$m membrane filter, which is compatible with the liquids to be used.

**3.3** *Solvent dispensers,* each fitted with a 0.45 $\mu$m membrane filter directly at the outlet.

**3.4** *Sample agitating device,* that will not alter the basic size distribution of the latex spheres during agitation

NOTE An ultrasonic bath has been shown to be an acceptable means of both dispersing agglomerates within the liquid and removing air introduced by manual agitation. The ultrasonic bath would normally be of 0.5 L capacity and typically between 50 W and 100 W at 40 kHz to 80 kHz.

**3.5** *Sample containers,* which are a number of cylindrical glass bottles possessing the following features:

a) dimensions that are compatible with the sample bottle facility in use with the counter (usually 250 mL);

b) a flat bottom;

c) either of the following:

1) a polypropylene threaded cap forming a seal with the bottle without the use of an insert;

2) a cap with a suitable internal seal.

**3.6** *Dosing pipettes,* comprising a range of one mark graduated pipettes complying with BS 1583.

**3.7** *Optical microscope,* for use if it is found necessary to verify the absolute diameter of the latex spheres.

NOTE A binocular microscope with a magnification range of × 40 to × 400 and a mechanical X-Y stage is considered appropriate. The eyepiece graticule should be calibrated using a reference stage micrometer and microscope illumination should be either oblique or incident, as required.

## 4 Materials

**4.1** *Cleaning liquids for glassware,* comprising the following:

a) distilled/deionized water;

b) liquid detergent that is water soluble;

c) isopropyl alcohol (IPA), reagent grade, that has been filtered through a 0.45 $\mu$m membrane filter.

**4.2** *Latex spheres calibration material,* comprising a range of six mono-sized latex sphere dispersions suspended in a suitable carrier liquid which is totally miscible with the suspension liquid.

NOTE This range should be selected so that it covers the sizing range of the sensor being calibrated. The minimum size of sphere should be carefully selected so that the complete distribution is above the "noise" level of the instrument. The manufacturer of the instrument should be consulted to verify the choice made.

**4.3** *Suspension liquid,* consisting of distilled or deionized water that has been filtered through a 0.45 $\mu$m membrane filter.

## 5 Preliminary procedures

NOTE The calibration procedures described in clause **6** generally involve the use of only two channels of a counter. Where the principle of detection is by using more than one voltage threshold detector, the instrument should be serviced by the manufacturer, or competent agent, and the voltage threshold levels adjusted to "trigger" at the correct value before proceeding with calibration.

### 5.1 Preparation and cleaning of apparatus

NOTE The procedure described in **5.1.1** is based upon BS 5540-3.

#### 5.1.1 *Procedure*

**5.1.1.1** Wash the apparatus with warm tap water containing liquid detergent [**4.1** b)] to an approximate concentration of one part of detergent to 20 parts of water and rinse thoroughly with warm tap water.

**5.1.1.2** Rinse with distilled/deionized water and allow to drain in an inverted position.

**5.1.1.3** Rinse thoroughly with filtered isopropyl alcohol [**4.1** c)] and allow to drain in the inverted position.

**5.1.1.4** Using suspension liquid (**4.3**) supplied from a solvent dispenser (**3.3**), flush-out the glassware and allow to drain in the inverted position.

**5.1.1.5** Clean the sample container caps in a similar manner and fit them to their respective containers.

**5.1.1.6** Store the cleaned apparatus under clean air conditions.

#### 5.1.2 *Verification of cleanliness levels*

Using the suspension liquid (**4.3**) adopt the procedure specified in BS 5540-1.

NOTE As it is usual to analyse up to 100 mL of liquid this requires a larger liquid volume than that specified in BS 5540-1.

The required cleanliness level (RCL) shall be less than five particles greater than 5 $\mu$m/mL per container.

### 5.2 Sensor system cleanliness

**5.2.1** Clean the sensor and associated pipework prior to use, by flushing with a filtered solvent before analysis of the samples. The liquid shall be either the suspension liquid (**4.3**), or a liquid that is totally miscible with it, and that is dispensed from a dispenser (**3.3**).

**5.2.2** If the sensor has previously been used to analyse a liquid that is not miscible with the liquid to be analysed, carefully clean the sensor (see **6.6**) before proceeding.

**5.2.3** Inspect the sensing volume on a regular basis for the presence of particles in either the sensing volume itself or the entry to it.

**5.2.4** Reverify the RCL.

### 5.3 Determination of "noise" level of the instrument

**5.3.1** Since all particle counters are affected by "noise", to a greater or lesser extent, determine the level in the manner prescribed by the instrument manufacturer.

**5.3.2** If a procedure is not prescribed by the manufacturer, use the following procedure (which is recommended by some manufacturers of automatic particle counters) to indicate the level at which "noise" becomes significant.

a) Disconnect the sensor from the sampling system, inspect for blockage and flush with filtered suspension liquid or a solvent that is compatible with it, and allow to dry.

b) Place the sensor on a surface that is free from vibration so that the axis of the sensor connections is horizontal.

c) Adjust the level of the first and lowest threshold voltage setting until the instrument counts at a rate of approximately 60 counts per minute. This is deemed the "noise" level.

NOTE If this procedure can not be completed dry the manufacturer, or competent agent, should be consulted.

**5.3.3** Use the "noise" level so determined to indicate the minimum sizing diameter and make all subsequent measurements at voltage settings above this value.

### 5.4 Precautions

#### 5.4.1 *Instrument location*

Locate the instrument in a class K clean environment or better in accordance with BS 5295-1.

#### 5.4.2 *Electrical interference*

As the APC is typically a high sensitivity device and may be affected by radio frequency or electromagnetic interference, take precautions to ensure that the test area environment does not interfere with the operation of the counter.

In addition, ensure that the voltage supply to the instrument is stable and free from "spikes".

NOTE A constant voltage transformer is considered appropriate.

#### 5.4.3 *Instrument operation*

Make all measurements at sphere concentrations that are less than 50 % of the manufacturer's stated maximum.

NOTE A procedure for establishing the maximum concentration limit of the sensor/instrument combination is described in **6.3**.

#### 5.4.4 *Chemicals*

Since some of the chemicals used in the procedures may be harmful, toxic and flammable, adopt good laboratory practices in their preparation and use, taking care to ensure chemical compatibility of the materials used.

## 6 Calibration procedures

NOTE The procedures detailed in this clause assume that the particle counter is operating within its limitations and is set to count in a cumulative or "total" mode and is equipped with at least two channels with adjustable voltage thresholds.
If the voltage range of individual channels is limited, it will be necessary to obtain data using other and higher channels.

### 6.1 Redispersing of latex spheres

To redisperse the latex spheres, which are received as concentrated suspensions (some containing surfactants to reduce agglomeration) and which if allowed to stand for substantial periods may settle out and agglomerate, proceed as follows:

a) hand shake vigorously for a period of 5 s;

b) partially immerse in an ultrasonic bath for a period of 30 s (see to **3.4**);

c) repeat a) and b);

d) use the spheres immediately afterwards.

NOTE Latex spheres are normally supplied for use in aqueous liquids, and any sensor wetted with non-aqueous liquids that are not water miscible needs careful cleaning before calibration (see **6.6**).

CAUTION. Aged latex samples may not redisperse or may contain contaminating micro-organisms. Unless otherwise stated for the product, discard after 3 years.

### 6.2 Preparation of calibration sample

**6.2.1** Rinse the concentrate bottle and its dropper tip and cap with prefiltered suspension liquid and place the cap in its normal attitude on a cleaned, non-shedding surface.

**6.2.2** Add approximately 50 mL of filtered suspension liquid to a clean sample container, followed by a suitable volume of the previously agitated latex suspension to give a final concentration less than the operational limit.

**6.2.3** Add a further volume of filtered suspension liquid so that the total volume is approximately 80 % of the volume of the sample container, flush the cap of the container with pre-filtered suspension liquid and recap.

**6.2.4** Shake the suspension by hand for about 5 s and degas the suspension by either using the vacuum apparatus on the bottle sampling apparatus or immersing the sample in an ultrasonic bath for 10 s. Analyse the sample without delay.

**6.2.5** If necessary perform an exploratory count to determine the volume of carrier fluid necessary to provide sufficient numbers of particles without exceeding the operating limit defined in **5.4.3**.

### 6.3 Verification of maximum concentration limit

NOTE The procedure described in **6.3.1** is designed to measure the maximum concentration limit for the sensor/counter combination. Since it is carried out using mono-sized latex spheres which have a very narrow size distribution, this concentration limit can be considered to be the highest possible. In practice contaminant distributions often have a wider size range, and coincidence at smaller particle sizes can lead to them being miscounted as larger particles. This procedure should be carried out initially and after any major change or repair to the sensor.

#### 6.3.1 *Procedure*

**6.3.1.1** Prepare 1 L of suspension as described in **6.1** and **6.2** using latex spheres appropriate to the sensor, e.g. any distribution having a mean size in the region of 10 % to 50 % of the sensor's dynamic range. Use an arbitrary quantity (e.g. two drops) of the concentrated latex suspension and disperse thoroughly.

**6.3.1.2** Count a sample of this bulk suspension diluted to 50 % using a threshold setting (determined from a previous calibration) below the minimum size of the distribution.

**6.3.1.3** From the count in **6.3.1.2**, adjust, by either dilution or adding more concentrate, the concentration of the bulk suspension so that it is at about, or just above, twice the instrument manufacturer's stated operational limit of the counter.

**6.3.1.4** Prepare a series of samples representing nominal concentrations of 10 %, 20 %, 40 %, 80 %, 100 %, 120 %, 140 % and 180 % of the instrument manufacturer's operational limit.

NOTE The exact concentrations are not important but it is important that the ratios of the concentrations should be accurately known.

**6.3.1.5** Prepare the samples as detailed in **6.2.4** and count the samples in accordance with the instrument manufacturer's instructions, using the voltage threshold level used in **6.3.1.2**. Make at least three runs to verify the stability of the suspension.

#### 6.3.2 *Analysis of data*

From the particle count data, determine the mean count at each concentration and plot the number of spheres per unit volume versus the nominal concentration on linear graph paper.

Consider the point where the relation departs from linear by more than 5 % to be the operational limit for the sensor/instrument combination.

### 6.4 Verification of the size distribution of the latex spheres

NOTE The purpose of this stage is to verify that the size distribution of the spheres is acceptable for calibration.

#### 6.4.1 *Procedure*

NOTE This procedure uses the multichannel facility feature of most automatic particle counters, but can also be performed by making successive runs from the sample where the number of channels is limited. Alternatively, a multichannel analyser may be used.

**6.4.1.1** Obtain from the supplier or by direct microscopy the maximum and minimum particle sizes and the distribution of the sizes of mono-sized spheres to be analysed. If the minimum size is not available, estimate the size by using the quoted variance and taking the limits equivalent to ± 3 standard deviations.

**6.4.1.2** Convert this size range into a threshold setting range for the instrument using a previous calibration curve obtained from latex spheres.

**6.4.1.3** Divide this threshold range to give 10 to 20 equal increments.

**6.4.1.4** Prepare a bulk sample of the spheres of interest (see **6.2**) so that one sample can be counted at all settings.

NOTE Multichannel instruments require a less bulky sample than two-channel instruments.

**6.4.1.5** Count the sample at the lowest threshold setting and ensure that the maximum concentration limit is not exceeded. Adjust the concentration if necessary.

NOTE The sample should be sufficiently concentrated that background counts are insignificant.

**6.4.1.6** Count over the full size range of threshold settings determined in **6.4.1.2**.

NOTE If a multichannel device is used, it may be more convenient to maintain the setting of the first (and lowest) threshold value at its initial level, so that the counts at other runs and settings may be referred to it, and give the percentage distribution.

#### 6.4.2 *Data analysis*

**6.4.2.1** If the same minimum setting is used, reference the particle counts at the various settings for an individual run to it and plot the percentage count against the voltage threshold setting on linear graph paper.

NOTE A typical percentage distribution curve is shown in Figure 1.

If the data is in the form of particle counts, plot the particle count against voltage threshold setting on log/linear graph paper.

NOTE At the lowest threshold settings the curves should level off. If they do not, the cause may be lack of cleanliness in preparing the sample, or the spheres can not all be distinguished from the noise as would occur if the size of the spheres is too small. Similarly the curves should level off to zero counts at high threshold settings; failure to do so suggests agglomeration of the spheres, and more careful preparation of the sample may clear the problem. If a curve is very asymmetric it suggests that the size distribution of the spheres is not Gaussian and thus not suitable for calibration purposes.

From these curves obtain the settings for calibration (see **6.5**) as follows:

a) the first and lowest threshold from the point on the curve where the slope is zero;

b) the second threshold from the mid-point of the curve.

**6.4.2.2** Repeat for other sizes of latex spheres.

### 6.5 Calibration using half-count procedure

NOTE The half-count procedure described in this subclause is based upon the principle that the first (lowest) channel is set so that all the spheres in the narrow distribution are counted and the second (higher) channel is adjusted to count half the total number recorded. This corresponds to the mean of the distribution. If the voltage range of the individual channels is limited it will be necessary to obtain calibration data using higher channels.

**6.5.1** Clean the apparatus in accordance with **5.2** and prepare the latex sphere suspension in accordance with **6.1** and **6.2**.

**6.5.2** Adjust the threshold of the first (or lowest) channel to a value that will count all of the spheres in the narrow distribution using either of the following:

a) previous calibration data;

b) the minimum settings obtained from the verification procedure (see **6.4**).

NOTE If this voltage threshold is on or below the noise voltage level determined in accordance with **5.3** then the size of sphere is unsuitable.

**6.5.3** Adjust the voltage threshold of the second (or next) channel to a value that approximates to the mean of the distribution or the mid-point of the curve obtained in **6.4**.

**6.5.4** Without further changes to the threshold level of the first channel, count the sample in accordance with the instrument manufacturer's recommendations, and adjust the settings of the second channel to record a half count of the first channel within 2.5 %.

**6.5.5** Record the voltage threshold setting of the second channel.

**6.5.6** Repeat **6.5.1** to **6.5.5** using different size ranges of mono-sized latex spheres within the range of the sensor used, i.e. a range of spheres biased towards the smaller sizes.

NOTE 1 For example, for a sensor whose size range is nominally 2 $\mu$m to 120 $\mu$m, the sizes of spheres would be in the range 2.5 $\mu$m to 50 $\mu$m with at least three sizes in the range 2.5 $\mu$m to 10 $\mu$m. This emphasizes the necessity for having sufficient data points in the lower 10 % of the dynamic range where the response of the sensor may not be linear.

NOTE 2 If larger sized spheres (> 20 $\mu$m) are used a method of stirring will be necessary to maintain a constant suspension.

**6.5.7** Plot a graph of mean particle size against voltage threshold setting using log/log graph paper and draw the best smooth curve through the points.

NOTE 1 This is the calibration curve for the sensor/instrument combination from which the voltage threshold settings corresponding to the required size ranges can be read off.

NOTE 2 Some counters operate on a particle size distribution principle, having many (up to 256) channels. With these instruments, a single analysis of a prepared suspension of each latex sphere size permits calibration.

### 6.6 Use of the sensor with different fluids

NOTE When the sensor is to be used to analyse a liquid that is not compatible with the liquid previously used, the transparent windows of the sensor may have films or droplets of previous liquid remaining upon them, leading to erratic counting.

**6.6.1** When changing fluids, flush with a succession of liquids, each one compatible with the preceding one, e.g. to change from oil to water, a typical sequence would be as follows:

a) flush with petroleum ether (boiling range 100 °C to 120 °C) or 1,1,1-trichloroethane;

b) flush with isopropan-2-ol;

c) flush with distilled or deionized water.

**6.6.2** Monitor the success of flushing by analysing a sample of 0.45 $\mu$m membrane filtered suspension liquid and check that low particle counts have been obtained before proceeding with further calibrations.

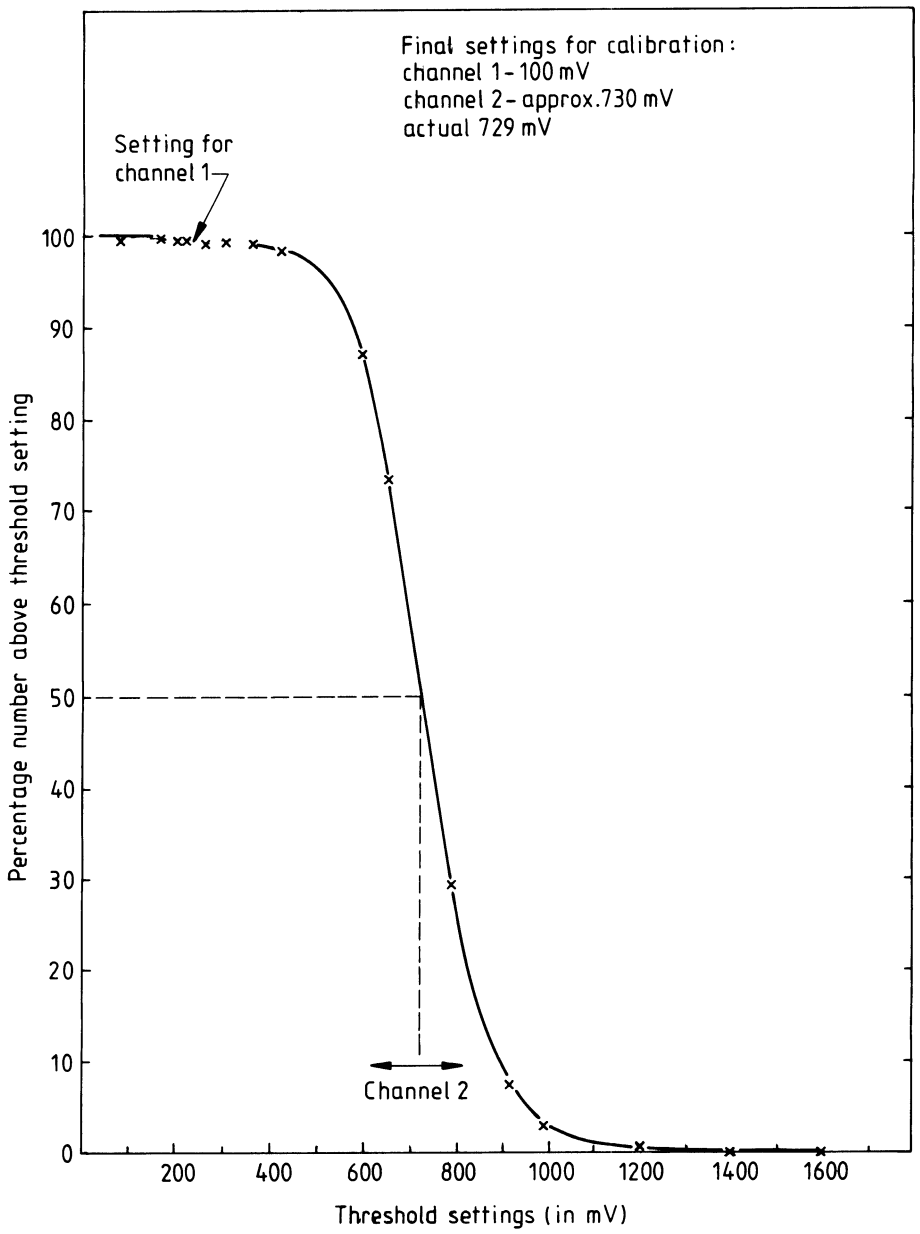


**Figure 1 — Typical example of percentage distribution curve for 38.8 $\mu$m spheres**

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# Publication(s) referred to

BS 1583, *Specification for one-mark pipettes.*
BS 5295, *Environmental cleanliness in enclosed spaces.*
BS 5295-1, *Specification for clean rooms and clean air devices.*
BS 5540, *Evaluating particulate contamination of hydraulic fluids.*
BS 5540-1, *Qualifying and controlling of cleaning methods for sample containers.*
BS 5540-3, *Methods of bottling fluid samples.*

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