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Metallic powders — Determination of envelope-specific surface area from measurements of the permeability to air of a powder bed under steady-state flow conditions

Poudres métalliques — Détermination de la surface spécifique d'enveloppe à partir de mesures de la perméabilité à l'air d'un lit de poudre dans des conditions d'écoulement permanent

Reference number
ISO 10070:1991(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10070 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*.

Annexes A and B of this International Standard are for information only.

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Introduction

The measurement of the permeability of a packed powder bed to a laminar gas flow is the basis of this International Standard. The determination can be made either at constant pressure drop (steady-state flow) or at variable pressure drop (constant volume).

The permeability measured is influenced by the porosity of the bed. For a given particle shape, the values of permeability and porosity can be used to calculate a specific surface area of the powder by means of equations of different types.

The surface area so calculated includes only those walls of the pores in the bed which are swept by the gas flow. It does not take into account closed or blind pores. It is defined as the envelope-specific surface area. It may be very different from the total surface area of particles as measured, for instance, by gas adsorption methods.

A single equation is used in the standard methods described and this entails certain limitations with respect to the type of powder (particle shape) and the porosity of the powder bed for which the method is applicable. Consequently this is not an absolute method, and the value obtained depends upon the procedure used and the assumptions made.

The specific surface area determined can be converted into a mean equivalent spherical diameter (see definitions, clause 3).

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Metallic powders — Determination of envelope-specific surface area from measurements of the permeability to air of a powder bed under steady-state flow conditions

1 Scope

1.1 This International Standard specifies a method of measuring the air permeability and the porosity of a packed bed of metal powder, and of deriving therefrom the value of the envelope-specific surface area.The permeability is determined under steady-state flow conditions, using a laminar flow of air at a pressure near atmospheric. This International Standard does not include the measurement of permeability by a constant volume method.

Several different methods have been proposed for this determination, and several instruments are available commercially. They give similar, reproducible results, provided the general instructions given in this International Standard are respected and the test parameters are identical.

This International Standard does not specify a particular commercial apparatus and corresponding test procedure. However, for the convenience of the user, an informative annex has been included (annex A) which is intended to give some practical information on three specific methods:

— the Lea and Nurse method, involving an apparatus which can be built in a laboratory (see A.1);

— the Zhang Ruifu method, using similar equipment (see A.2);

— the Gooden and Smith method, involving an apparatus which can be built in a laboratory but for which a commercial apparatus also exists (see A.3).

These methods are given as examples only. Other equipment available in various countries is acceptable within the scope of this International Standard.

1.2 This method is applicable to all metallic powders, including powders for hardmetals, up to 1 000 µm in diameter, but it is generally used for particles having diameters between 0,2 µm and 50 µm. It should not be used for powders composed of particles whose shape is far from equiaxial, i.e. flakes or fibres, unless specifically agreed upon between the parties concerned.

This method is not applicable to mixtures of different metallic powders or powders containing binders or lubricant.

If the powder contains agglomerates, the measured surface area may be affected by the degree of agglomeration. If the powder is subjected to a deagglomeration treatment (see annex B), the method used shall be agreed upon between the parties concerned.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3252:1982, *Powder metallurgy — Vocabulary*.

ISO 3954:1977, *Powders for powder metallurgical purposes — Sampling*.

ISO 4022:1987, *Permeable sintered metal materials — Determination of fluid permeability*.

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 permeability: Ability of a porous material to allow a fluid to flow through it.

NOTE 1 In this standard, the fluid used is dry air.

3.2 interstices: Spaces between particles in a powder bed, through which the air flows.

3.3 permeable porosity: Volume of interstices divided by the volume of the bed.

3.4 envelope volume: Volume occupied by the particles in a powder bed, excluding the volume of the interstices. In permeametry, the envelope volume comprises the volume of the solid matter plus the volume of all the pores which do not contribute to gas flow (closed pores, blind pores, micropores, surface micropores, surface roughness, etc.). Since this volume cannot be measured by any known method, it is taken, for the purposes of this International Standard, as being equal to the effective volume, as determined by liquid pyknometry.

3.5 envelope density: Mass of a powder bed divided by its envelope volume. The envelope density may be less than the solid density when particles contain pores that do not contribute to the gas flow through the bed.

3.6 mass-specific surface area: The surface area of a powder divided by its mass. This area depends on the type of method used for its determination.

3.7 envelope-specific surface area: The specific surface area of a powder as determined by gas permeametry in accordance with this International Standard.

3.8 volume-specific surface area: The surface area of a powder divided by its effective volume (i.e. by its envelope volume).

3.9 equivalent sphere diameter: Diameter of theoretical non-porous spherical particles of identical size, with which the same method of permeametry as that used for the powder under examination would give the same volume-specific surface area.

4 Symbols and their meanings

Table 1 — Symbols used in the text

Symbol	Meaning	Unit	Observations
	Powder bed		
A	Cross-sectional area	m^2	Area of whole cross-section of bed perpendicular to flow direction: $A = \frac{\pi d^2}{4}$
d	Diameter of measuring cell	m	
L	Thickness (or height)	m	
m	Mass of powder	kg	
ϱ_e	Envelope density	kg/m^3	
ϱ	Solid density	kg/m^3	
ε_p	Permeable porosity		$\varepsilon_p = 1 - \frac{m}{AL\varrho_e}$
ε	Total porosity		$\varepsilon = 1 - \frac{m}{AL\varrho}$
	Gas flow		
q	Volume flow rate	m^3/s	Converted to standard conditions (STP)
p	Mean gas pressure	N/m^2	
Δp	Pressure drop	N/m^2	
η	Viscosity of gas	Ns/m^2	
T	Temperature of gas	K	
M	Molar mass of gas	kg/mol	$M = 0{,}029$ kg/mol for air
R	Molar gas constant	$\frac{J}{mol \cdot K}$	$R = 8{,}31 \frac{J}{mol \cdot K}$
	Calculation		
K	Kozeny-Carman factor		For the purposes of this International Standard, $K = 5{,}0$
δK_0	Compound constant		For the purposes of this International Standard, the generally accepted value of 2,25 is used
S_w	Mass-specific surface area	m^2/kg	
S_V	Volume-specific surface area	m^{-1}	$S_V = \varrho_e S_w$
Φ	Permeability	m^2	
D	Equivalent sphere diameter	m	$D = \frac{6}{S_V} = \frac{6}{\varrho_e S_w}$

5 General principles

5.1 Basically, permeametry is the experimental determination of the permeability Φ of a powder bed, the porosity of which is known.

The permeability is determined by measuring the volume flow rate q and the drop in pressure Δp of a dry gas (generally air) continuously traversing the bed under laminar flow conditions.

The permeability coefficient is then calculated from Darcy's law:

$$\Phi = \frac{q\eta L}{A\,\Delta p} \qquad \ldots (1)$$

5.2 The Carman-Arnell equation relates specific surface area to the porosity and permeability of a packed bed of powder, and takes into account both the viscous flow and the slip flow. This equation can be written:

$$\Phi = \frac{\varepsilon_p}{K\eta}\left[\frac{\varepsilon_p^2}{S_V^2(1-\varepsilon_p)^2} + \frac{8}{3}\sqrt{\frac{2RT}{\pi M}} \times \frac{\delta\, k_o\, \eta\, \varepsilon_p}{pS_V(1-\varepsilon_p)}\right] \qquad \ldots (2)$$

The solution of equation (2), which is quadratic in S_V, can be simplified by calculating the value of two terms, the Kozeny term S_K and the slip flow term S_m, and then combining them to give S_V.

The Kozeny term S_K is given by the equation

$$S_K = \sqrt{\frac{A\,\Delta p\,\varepsilon_p^3}{K(1-\varepsilon_p)^2 L\,\eta\,q}} \qquad \ldots (3)$$

This term is identical to the Kozeny-Carman equation for S_V and gives the contribution to the surface area of the powder due to streamline flow.

The slip flow term S_m is given by the equation

$$S_m = \frac{A\,\Delta p}{KLq} \times \frac{8}{3}\sqrt{\frac{2RT}{\pi M}} \times \frac{\delta\, k_o\, \varepsilon_p^2}{p(1-\varepsilon_p)} \qquad \ldots (4)$$

or, in the case of air,

$$S_m = 81\, S_K^2 \frac{(1-\varepsilon_p)\,\eta}{p\,\varepsilon_p}\sqrt{T} \qquad \ldots (5)$$

S_V is then given by

$$S_V = \frac{S_m}{2} + \sqrt{\frac{S_m^2}{4} + S_K^2} \qquad \ldots (6)$$

and the mass-specific surface area S_w by

$$S_w = \frac{S_V}{\varrho_e} \qquad \ldots (7)$$

The equivalent sphere diameter D is given by

$$D = \frac{6}{S_V} = \frac{6}{\varrho_e S_w} \qquad \ldots (8)$$

The Carman-Arnell equation (2) shall be used when the volume-specific surface area is greater than $10^6\ m^{-1}$ (mean particle size less than 6 µm), because the slip flow component of the permeability becomes significant in addition to the viscous flow term.

For coarser powders, the Kozeny-Carman equation (3) may be used by agreement between the parties concerned; the error introduced by neglecting slip flow is about 10 % at a mean particle size of 6 µm and increases as the powder becomes finer.

The mass-specific surface area S_w is given by the equation

$$S_w = \sqrt{\frac{\varepsilon_p^3 A\,\Delta p}{5{,}0\,(1-\varepsilon_p)^2 q\eta L \varrho_e^2}} \qquad \ldots (9)$$

5.3 The methods and instruments used in practice differ depending on the way in which the volume flow rate of the gas and the pressure drop are measured. Annex A describes three methods by way of example: the Lea and Nurse method, the Zhang Ruifu method and the Gooden and Smith method.

5.4 The Kozeny-Carman relation applies only over a limited range of bed porosities, the range depending on the type of powder. It applies best to equiaxial powders. The Kozeny factor K varies with the particle shape and particle size distribution. In this International Standard, the value of K is taken to be 5,0 but other values may be used by agreement between the parties concerned.

5.5 Due to the limitations noted in 5.4, the variation of the specific surface area as a function of porosity shall first be determined experimentally for any particular type of powder.

For example, make several successive determinations of the permeability, using test portions of the same mass from the same laboratory sample, and packing the powder bed to give a decreasing series of porosities. Over a certain range of porosities, the specific surface area will be practically constant. Only determinations made within this range shall be taken as valid.

5.6 In the above equations, the permeable porosity ε_p of the powder bed and the envelope density ϱ_e of the particles are used. They are related by the equation

$$\varepsilon_p = 1 - \frac{m}{A\,L\,\varrho_e} \qquad \ldots (10)$$

The envelope density ϱ_e is equal to the solid density only for smooth, non-porous particles. In such cases, $\varepsilon_p = \varepsilon$.

In all other cases, the envelope density ϱ_e shall be measured by an appropriate pyknometric method. The solid density value ϱ, or another density, may be adopted instead of the envelope density by agreement between the parties concerned.

6 Procedure

6.1 Preparation of test portion

Sampling shall have been carried out in accordance with ISO 3954. The test portion shall be taken from the test sample in the as-delivered state. Drying, in an appropriate atmosphere, or de-agglomeration (see annex B), is only permitted by agreement between the parties concerned.

Weigh the test portion to within 0,1 %.

6.2 Preparation of packed powder bed

The thickness L of the bed shall be not less than 50 times the mean particle diameter, and the bed diameter shall be not less than 100 times the mean particle diameter.

NOTE 2 At the surface of a test bed, discontinuities occur due to wall and end effects. These effects are negligible (producing an error of less than 2 % in the permeability), provided that the diameter and thickness of the test bed are as specified above.

The test portion is held in the cell by means of a porous paper disc at each end, and supported by a rigid perforated plate.

Introduce the test portion into the measuring cell in one pour, gently tapping the side of the cell to settle the powder. Pack the bed, covered with a porous paper disc, using a piston with grooves or holes to facilitate the expulsion of gas from the powder during the packing operation. Packing is achieved by applying a force slowly on the piston, up to a value which will give a porosity in the desired range and/or uniform packing of the bed.

NOTE 3 If there is evidence that the porosity of the packed bed is not homogeneous, then incremental pouring and packing is recommended.

Extract the piston using a rotary motion to minimize disturbance of the powder bed.

6.3 Determination

Measure the thickness of the bed to within 0,25 %. The temperature during the test shall not vary by more than $\pm$ 3 °C from the temperature at which the apparatus was calibrated.

Pass a constant flow of gas through the powder bed. When the gas flow has stabilized, measure the flow rate and pressure drop. The pressure drop shall be small compared with atmospheric pressure (less than about 4 000 N/m^2), so that the effect of the compressibility of the gas is negligible (see annex A, clause A.2, for a case in which the compressibility effect is taken into account and corrected for).

If necessary, a blank test shall be carried out to correct for the effect of the paper disc.

7 Expression of results

The specific surface area of the powder is calculated either by using equations (3), (5) and (6), or from equation (9).

The result shall be expressed in terms of one or more of the following quantities, using the units indicated:

— mass-specific surface area S_w, in square metres per kilogram or square metres per gram;

— volume-specific surface area S_V, in square metres per cubic metre or square centimetres per cubic centimetre;

— equivalent sphere diameter D from equation (8), in metres or micrometres.

8 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the sample;

c) the method and apparatus used;

d) any drying or de-agglomeration procedure used;

e) the density adopted (see 5.6);

f) the permeable porosity ε_p of the bed;

g) the equation used for the calculation of the specific surface area;

h) the value of the Kozeny-Carman factor if not taken as equal to 5,0 (see 5.4);

i) the result obtained;

j) details of any incident which may have affected the test result;

k) any operation not specified in this International Standard or regarded as optional.

Annex A
(informative)

Examples of methods of determining the permeability to air of a powder bed

A.1 Lea and Nurse method
(see figure A.1)

In this method, a steady-state flow of dry air is fed first through the powder bed and then through the fixed capillary resistance of a flowmeter open to the atmosphere. The pressure drop Δp across the bed is measured by means of a manometer (reading h_1) and the flow rate q by means of the capillary flowmeter (reading h_2). The relationship between the flow rate q and the flowmeter reading h_2 can be established by a suitable calibration method.

Thus the Lea and Nurse method is an absolute one, as all the quantities in equation (A.1) below are either known or measured:

$$S_w^2 = \frac{\varepsilon_p^3 C_1 h_1 A}{5{,}0\,(1-\varepsilon_p)^2 C_2 h_2 \eta L \varrho_e^2} \qquad \ldots (A.1)$$

where

C_1 is the calibration factor for the manometer ($\Delta p = C_1 h_1$);

C_2 is the calibration factor for the capillary flowmeter ($q = C_2 h_2$);

the other symbols are as defined in table 1.

In order to increase the accuracy of the permeability determination, it is recommended that the flow rate be adjusted to give three different values of h_2 and three corresponding manometer readings h_1. The mean of the three values of the ratio h_1/h_2 is used in equation (A.1).

NOTES

4 In order to test for bed uniformity, the determination is repeated with different amounts of powder packed to the same porosity or under the same packing force. If the bed is uniform, the results will be the same.

5 When testing a new type of powder, it recommended that its specific surface area be determined for a series of bed porosities. The porosity range for these determinations is defined by the porosity range over which the specific surface area does not vary appreciably. In general, the bed porosity should be between 0,45 and 0,7.

6 A linear relationship between flow rate and pressure drop indicates non-turbulent flow, permitting the use of the Carman-Arnell equation (2) or the Kozeny-Carman equation (9).

7 It is recommended that certified reference powders be used periodically to check the accuracy and the correct functioning of the instrument.

A.2 Zhang Ruifu method

A.2.1 This method uses an apparatus similar in principle to that described for the Lea and Nurse method, but the pressure drop Δp across the powder bed can be up to 20 000 N/mm^2. Moreover, a single determination by this method of the apparent equivalent diameter D_K for a known value of ε_p, permits the direct measurement of various types of specific surface and diameter.

In order to allow for the air compressibility effect, the basic Carman-Arnell equation (2) in 5.2 is written:

$$\Phi = \frac{\varepsilon_p}{K}\left[\frac{\varepsilon_p^2}{S_V^2(1-\varepsilon_p)^2} + \frac{\varepsilon_p}{S_V(1-\varepsilon_p)} \times Z\lambda\right]$$

where, for air at ordinary temperature,

Z is the slip flow factor (taken as being equal to 3,4);

λ is the mean free path, expressed at the mean pressure p in the powder bed, i.e.

$$\lambda = 0{,}097 \times 10^{-6} \times \frac{p_n}{p}$$

where p_n is the standard atmospheric pressure (101 300 N/m^2).

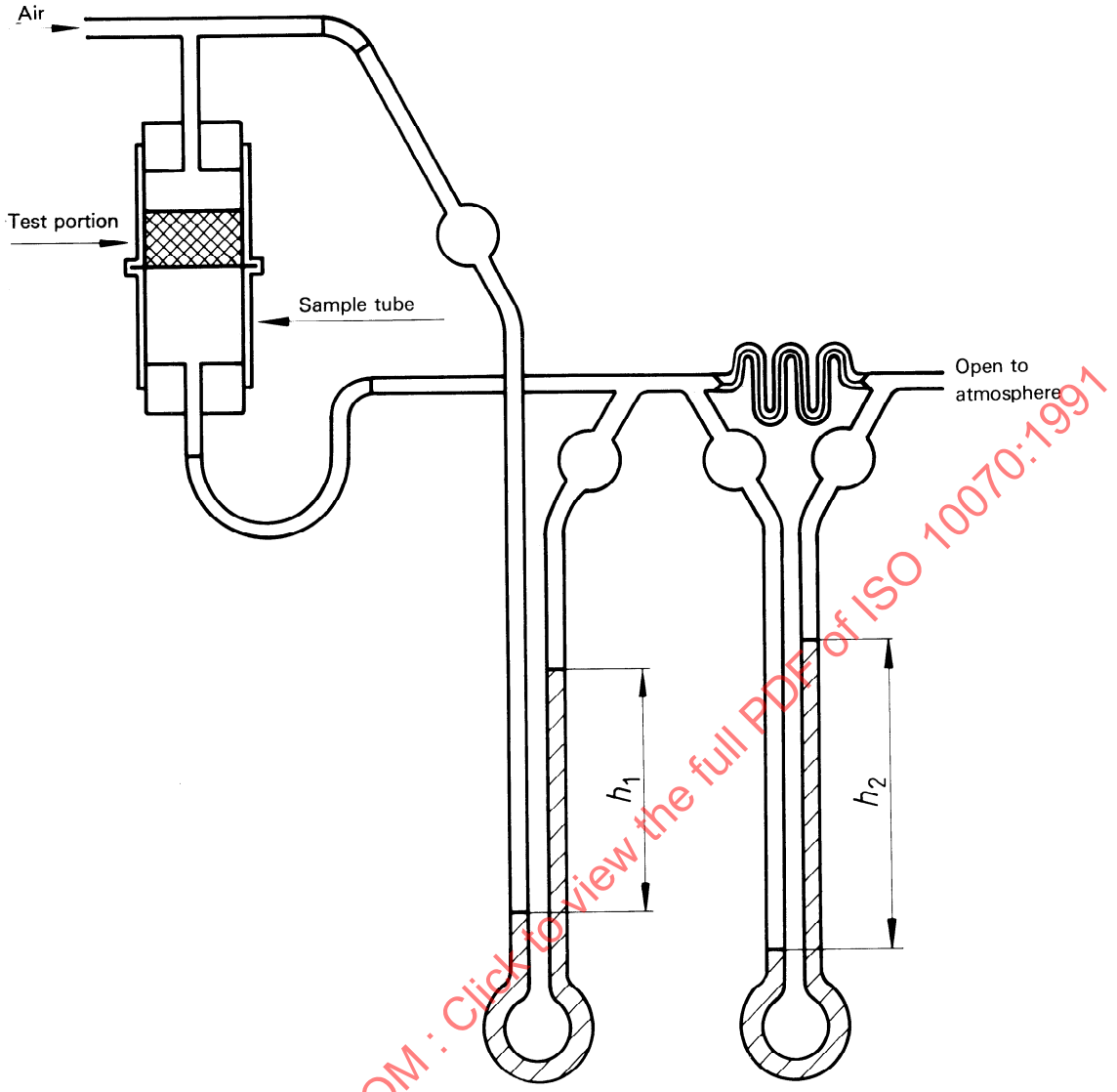

Figure A.1 — The Lea and Nurse permeability apparatus with manometer and flowmeter

A.2.2 The experimental results are processed as follows:

The viscous flow volume-specific surface area S_K [the Kozeny term in equation (2)] is first considered and the corresponding viscous flow equivalent sphere diameter

$$D_K = \frac{6}{S_K}$$

is calculated from the equation

$$D_K = \sqrt{\frac{36K\,(1-\varepsilon_p)^2\,L\eta q}{A\,\Delta p\,\varepsilon_p^3}} \times \left(1 - 2{,}5 \times 10^{-6}\,\Delta p\right)$$

Then

a) If D_K is greater than about 10 µm, no slip flow correction is required, and the final result can be calculated from the equations

$$S_V = S_K$$

$$D = D_K$$

$$S_w = \frac{S_K}{\varrho_e}$$

b) If D_K is less than 10 µm, it is necessary to take into account the slip flow component S_m of the specific surface area. It is convenient to introduce an intermediate quantity, the factor

$$\beta, \text{ defined by } \beta = \frac{S_m}{2S_K}$$

This factor can be calculated from the equation

$$\beta = \frac{10{,}2(1-\varepsilon_p)\eta}{\varepsilon_p D_K}$$

rewritten as

$$\beta = 10{,}2 \times \frac{1-\varepsilon_p}{\varepsilon_p} \times \frac{\eta_n p_n}{D_K\, p}$$

where

λ is $0{,}097 \times 10^{-6}$ metres;

p_n is the standard atmospheric pressure ($101\ 300\ N/m^2$).

The viscous flow and slip flow components of the specific surface area are given by the equations

$$S_K = \frac{6}{D_K}$$

$$S_m = \frac{12\beta}{D_K}$$

The final results are calculated using the equations

$$S_V = \frac{6}{D_K}\left(\beta + \sqrt{1+\beta^2}\right)$$

$$S_w = \frac{S_V}{\varrho_e}$$

$$D = \frac{D_K}{\beta + \sqrt{1+\beta^2}}$$

A.3 Gooden and Smith method (see figure A.2)

A.3.1 General

In this method, dry air is fed at a constant overpressure p_o, first through the powder bed, then through the fixed capillary resistance of a flowmeter connected to the atmosphere through an adjustable capillary tube or needle valve.

A single reading p' of the capillary flowmeter gives the flow rate. The pressure drop through the powder bed is then given by

$$\Delta p = p_o - p'$$

The specific surface area is calculated using the Kozeny-Carman equation (9) in which Δp has been replaced by $p_o - p'$, q has been replaced by $C' \cdot p'$ (where C' is the capillary constant of the flowmeter) and both sides have been squared to give

$$S_w^2 = \frac{\varepsilon_p^3 (p_o - p') A}{5{,}0\ (1-\varepsilon_p)^2 C' p' \eta L \varrho_e^2} \qquad \ldots (A.2)$$

The Fisher Subsieve Sizer[1] has been developed to carry out this method. The apparatus includes a sliding chart which gives a direct reading of the bed porosity and the equivalent sphere diameter of the powder.

No knowledge of the parameters p_o, A, C' or η is required, since the apparatus does not give an absolute determination. The apparatus requires calibration; this can be done using a reference powder or a calibrated orifice simulating a powder bed.

The equivalent sphere diameter is given by the equation

$$D^2 = \frac{180\, C' \eta (1-\varepsilon_p)^2 p' L}{A \varepsilon_p^3 (p_o - p')} \qquad \ldots (A.3)$$

where $\varepsilon_p = 1 - \dfrac{m}{AL\varrho_e}$

As the chart is designed for a mass of powder in the bed corresponding to an envelope volume of 1 cm^3 (i.e. m in grams is numerically equal to ϱ_e in g/cm^3), then

$$\varepsilon_p = 1 - \frac{1}{AL}$$

where A is in square centimetres and L in centimetres, and equation (A.3) can be written

$$D = \frac{CL}{(AL-1)^{3/2}} \times \sqrt{\frac{h}{H-h}} \qquad \ldots (A.4)$$

where

C is the flowmeter constant of the instrument;

h is the manometer reading, in centimetres of water;

H is the overall pressure head of the regulator, in centimetres of water;

A is the cross-sectional area of the powder bed, in square centimetres;

L is the thickness of the powder bed, in centimetres.

1) Fisher Subsieve Sizer is the trade-name an apparatus supplied by Fisher Scientific Co., 711 Forbes Avenue, Pittsburgh, PA 15219, USA. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the apparatus named. Equivalent instruments may be used if they can be shown to lead to the same results.

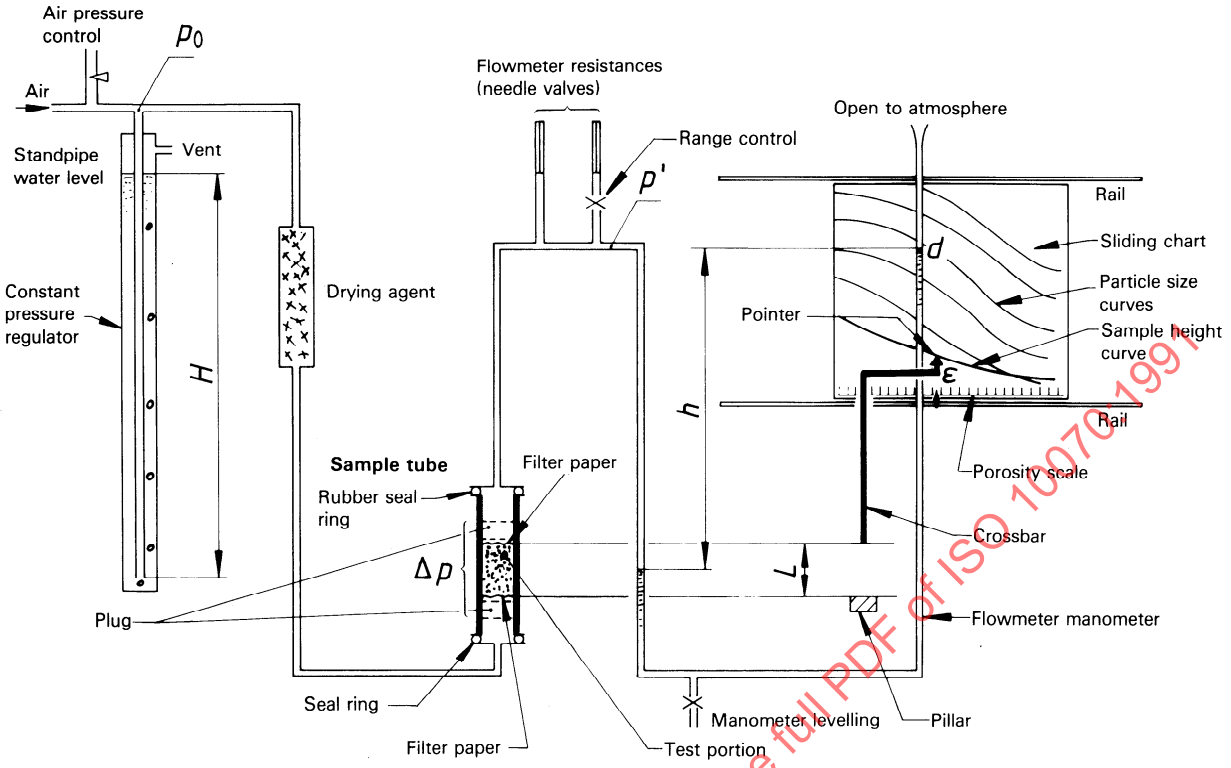

Figure A.2 — Schematic diagram of the Fisher Subsieve Sizer apparatus used in the Gooden and Smith method

The Fisher Subsieve Sizer includes two flow resistances, each corresponding to a different measurement range for the equivalent sphere diameter, namely:

0,2 µm to 20 µm

and

20 µm to 50 µm.

A.3.2 Operating procedure

A.3.2.1 General

The instructions supplied by the manufacturer shall be followed except where indicated otherwise in the following sub-clauses. Particular attention shall be given to proper maintenance of the apparatus, with specific reference to the instructions on

— periodic checking of the water level in the pressure regulator standpipe;

— the manometer level before the sample tube is inserted;

— the sample packing assembly;

— the condition of the drying agent.

A.3.2.2 Calibration of apparatus

Calibration shall be carried out in accordance with the manufacturer's instructions, using a Jewel calibrator tube as the primary standard. However, a calibration check is recommended before and after any single determination or series of determinations. The calibrator tube shall be checked frequently for cleanness using a microscope.

A.3.2.3 Temperature of test

Make all particle size determinations within $\pm$ 3 °C of the ambient temperature at which calibration was carried out.

A.3.2.4 Mass of test portion

The mass in grams of the test portion shall be numerically equal (within $\pm$ 0,01 g) to the envelope density of the powder in g/cm^3.

The value of the solid density may be used rather than that of the envelope density, but only if it is known that the particles in the powder have no pores, or by agreement between the parties concerned.

A.3.2.5 Determination of specific surface area

The determination shall be carried out in accordance with the manufacturer's instructions by the same operator who has carried out the calibration, or as agreed upon by the parties concerned.

A.3.2.6 Determination of porosity and mean sphere diameter

A.3.2.6.1 Direct reading

No calculation is needed when the porosity value is in the range covered by the sliding chart and the test portion has an envelope volume of 1 cm^3.

A.3.2.6.2 Extension of the range of the apparatus

The chart supplied with the apparatus covers the porosity range from 0,40 to 0,80. However, it is possible to test samples that are not compressed to a porosity of 0,40 by increasing the mass of the test portion normally specified. Corrections should be made to the indicated porosity and mean sphere diameter. Assuming that the test portion has an envelope volume of V cm^3 instead of 1 cm^3 (i.e. a mass of $V\varrho_e$ grams), the actual bed porosity will be

$$\varepsilon_p = 1 - [V\,(1 - \varepsilon_i)]$$

where ε_i is the porosity indicated on the chart.

The mean sphere diameter will be

$$D = D_i\,V\left(\frac{\varepsilon_i}{\varepsilon_p}\right)^{3/2}$$

where D_i is the mean diameter indicated on the chart.

Annex B
(informative)

Preliminary treatment of powder for de-agglomeration

B.1 General

This annex gives examples of methods which can be used to break down agglomerates in the powder sample.

B.2 Rolling

Spread the powder out in a thin layer on glazed paper and roll lightly with a glass rod.

B.3 Shaking

Place a sample of the powder in a clean, dry bottle sufficiently large to be only one-tenth filled. Seal the bottle and shake vigorously for 2 min. Allow to stand unopened for 2 min. Remove the lid and, before taking the test portion, stir the powder gently to distribute throughout the sample any fine fraction which has settled on the surface.

B.4 Rod milling

This is of particular use in the de-agglomeration of refractory metal powders. Place a sample of the powder (30 g in the case of tungsten or molybdenum powder, 50 g in the case of tungsten carbide powder) in a 250 ml glass reagent bottle, of diameter about 60 mm, containing 50 tungsten rods, of length about 75 mm and diameter about 4 mm, with ground surfaces. Seal the bottle and rotate for 60 min at a speed of about 150 rpm. After milling, screen the charge through a 1 mm sieve to remove the rods.

Other milling materials and conditions (such as a plastic bottle, cemented carbide rods, rods of a different size) may be used, provided equivalent results are achieved.