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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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 2, *Sampling and testing methods for powders (including powders for hardmetals)*.

This second edition cancels and replaces the first edition (ISO 10070:1991), which has been technically revised.

The main changes compared to the previous edition are as follows:

— introduction of an automated test device based on the Gooden and Smith method, including procedure and calibration.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The measurement of the permeability of a packed powder bed to a laminar gas flow is the basis of this document. The determination can be made either at constant pressure drop (steady-state flow) or at variable pressure drop (constant volume). This document deals only with determinations made under steady-state flow conditions.

The permeability measured is influenced by the porosity of the powder 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 different formulae.

The surface area so calculated includes only those walls of the pores in the powder bed which are swept by the gas flow. It does not take into account closed or blind pores. It is known as the envelope-specific surface area. It can 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 in this document. It 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 Clause 3).

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

This document 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 document does not include the measurement of permeability by a constant volume method.

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

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

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

— the Zhang Ruifu method, using a similar test device (see A.2);

— the Gooden and Smith method, involving a test device which can be built in a laboratory but for which a commercial test device also exists. (Two types of commercial test device exist; one of these is no longer available for purchase, but is still being used, see A.3.)

These methods are given as examples only. Other test devices available in various countries are acceptable within the scope of this document.

This testing 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 75,0 µm. It is not intended to 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 testing 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 can be affected by the degree of agglomeration. If the powder is subjected to a de-agglomeration treatment (see Annex B), the method used is to be agreed upon between the parties concerned.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3954, *Powders for powder metallurgical purposes — Sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
envelope density
mass of a powder bed divided by its *envelope volume* (3.3)

Note 1 to entry: The envelope density may be less than the solid density when particles contain pores that do not contribute to the gas flow through the powder bed.

3.2
envelope-specific surface area
specific surface area of a powder as determined by gas permeametry

3.3
envelope volume
volume occupied by the particles in a powder bed, excluding the volume of the *interstices* (3.5)

Note 1 to entry: 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 document, as being equal to the effective volume, as determined by pyknometry.

3.4
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* (3.9)

3.5
interstices
spaces between particles in a powder bed, through which the air flows

3.6
mass-specific surface area
surface area of a powder divided by its mass

Note 1 to entry: This area depends on the type of method used for its determination.

3.7
permeability
ability of a porous material to allow a fluid to flow through it

Note 1 to entry: to entry In this document, the fluid used is dry air.

3.8
permeable porosity
volume of *interstices* (3.5) divided by the volume of the powder bed

3.9
volume-specific surface area
surface area of a powder divided by its effective volume (i.e. by its envelope volume)

4 Symbols

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 powder 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 - 0 °C, 1 atm)
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	$\frac{J}{mol\,K}$	$R = 8,31 \frac{J}{mol\,K}$
	Calculation		
K	Kozeny-Carman factor		For the purposes of this document, K = 5,0
δK_0	Compound constant		For the purposes of this document, the generally accepted value of 2,25 is used
S_w	Mass-specific surface area	m^2/kg	
S_K	Kozeny term	m^{-1}	Formula (3)
S_m	Slip flow term	m^{-1}	Formula (4)
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 Permeability

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 powder bed under laminar flow conditions.

The permeability is then calculated from Darcy's law, as shown in Formula (1):

$$\Phi = \frac{q\eta L}{A\,\Delta p} \tag{1}$$

5.2 Carman-Arnell and Kozeny-Carman formulae

The Carman-Arnell formula, as shown in Formula (2), 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 formula can be written as:

$$\Phi = \frac{\varepsilon_p}{K\eta}\left[\frac{\varepsilon_p^2}{S_V^2\left(1-\varepsilon_p\right)^2} + \frac{8}{3}\sqrt{\frac{2RT}{\pi M}} \times \frac{\delta K_0 \eta \varepsilon_p}{p S_V \left(1-\varepsilon_p\right)}\right] \tag{2}$$

The solution of Formula (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 Formula (3):

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

The Kozeny term is identical to the Kozeny-Carman formula 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 Formula (4)

$$S_m = \frac{A\,\Delta p}{KLq} \times \frac{8}{3}\sqrt{\frac{2RT}{\pi M}} \times \frac{\delta K_0 \varepsilon_p^2}{p\left(1-\varepsilon_p\right)} \tag{4}$$

or, in the case of air, Formula (5):

$$S_m = 81 \times S_K^2 \frac{\left(1-\varepsilon_p\right)\eta}{p\,\varepsilon_p}\sqrt{T} \tag{5}$$

S_v is then given by Formula (6):

$$S_V = \frac{S_m}{2} + \sqrt{\frac{S_m^2}{4} + S_K^2} \tag{6}$$

and the mass-specific surface area S_w by Formula (7):

$$S_w = \frac{S_V}{\varrho_e} \tag{7}$$

The equivalent sphere diameter D is given by Formula (8):

$$D = \frac{6}{S_V} = \frac{6}{\varrho_e S_w} \tag{8}$$

The Carman-Arnell formula, Formula (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 formula, Formula (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 Formula (9):

$$S_w = \sqrt{\frac{\varepsilon_p^3 A \Delta p}{K\left(1-\varepsilon_p\right)^2 q\eta L \varrho_e^2}} \tag{9}$$

5.3 General

The methods and test devices 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 Kozeny-Carman relation applies only over a limited range of powder 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 document, the value of K is taken to be 5,0 but other values may be used by agreement between the parties concerned.

Due to the limitations of the Kozeny-Carman relation, 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.

5.4 Envelope density

In Formulae (1) to (9), the permeable porosity ε_p of the powder bed and the envelope density ϱ_e of the particles are used. They are related by Formula (10):

$$\varepsilon_p = 1 - \frac{m}{AL\varrho_e} \tag{10}$$

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 be 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 powder bed shall be not less than 50 times the mean particle diameter and the powder bed diameter shall be not be less than 100 times the mean particle diameter.

NOTE At the surface of a test powder 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 powder 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 test portion by covering it 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 powder bed.

If there is evidence that the porosity of the packed powder 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 powder bed to within 0,25 %. The temperature during the test shall not vary by more than ± 3 °C from the temperature at which the test device was calibrated.

Pass a constant flow of gas through the powder bed. When the gas flow has stabilized, measure the volume 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 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 Formulae (3), (5) and (6), or from Formula (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 Formula (8), in metres or micrometres.

8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 10070:2019;

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

c) the method and test device used;

d) any drying or de-agglomeration procedure used;

e) the density adopted (see 5.4);

f) the permeable porosity ε_p of the powder bed;

g) the formula 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 5.3);

i) the result obtained;

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

k) any operation not specified in this document is 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 powder bed is measured by means of a manometer (reading h_1) and the volume flow rate, q, by means of the capillary flowmeter (reading h_2). The relationship between the volume 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 Formula (A.1) are either known or measured:

$$S_w^2 = \frac{\varepsilon_p^3 C_1 h_1 A}{K\left(1-\varepsilon_p\right)^2 C_2 h_2 \eta L \varrho_e^2} \tag{A.1}$$

where

C_1 is the calibration factor 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.

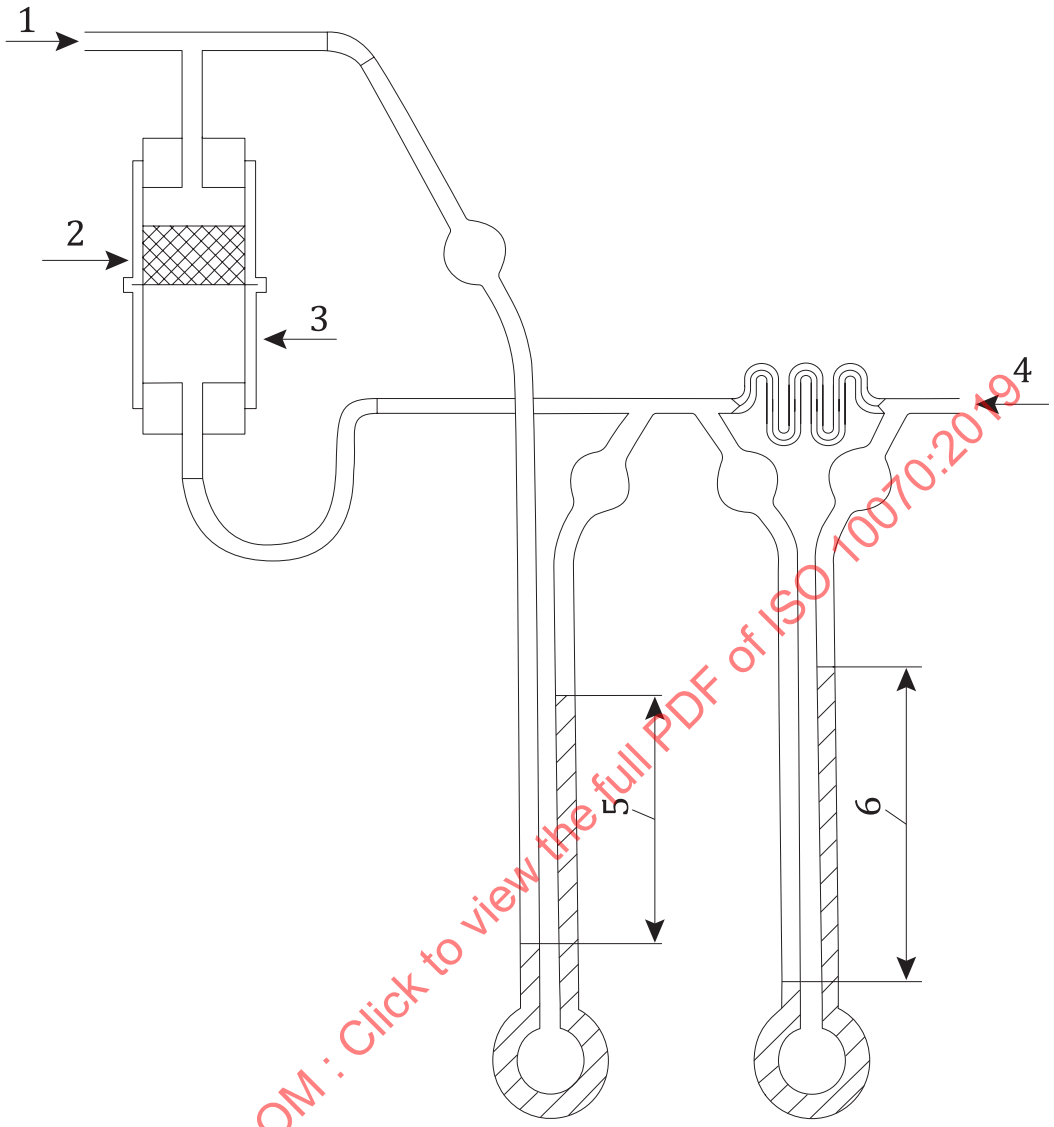
In order to increase the accuracy of the permeability determination, it is recommended that the volume flow rate be adjusted to give three different values of h_2 and three corresponding manometer readings h_1. The mean of the three values of the ratio h_1/h_2 is used in Formula (A.1).

NOTE 1 In order to test for powder bed uniformity, the determination is repeated with different amounts of powder packed to the same porosity or under the same packing force. If the powder bed is uniform, the results will be the same.

When testing a new type of powder, its specific surface area is determined for a series of powder 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 powder bed porosity is expected to be between 0,45 and 0,7.

NOTE 2 A linear relationship between volume flow rate and pressure drop indicates non-turbulent flow, where use of the Carman-Arnell equation, Formula (2), or the Kozeny-Carman equation, Formula (3), is appropriate.

Reference powders may be used periodically to check the accuracy and the correct functioning of the test device.



Key

1 air
2 test portion
3 sample tube
4 open to atmosphere
5 h_1
6 h_2

Figure A.1 — Lea and Nurse permeability test device with manometer and flowmeter

A.2 Zhang Ruifu method

A.2.1 Method

This method uses a test device 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 formula, Formula (2) in 5.2 is written as Formula (A.2):

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

where

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 according to Formulae (A.3):

$$\lambda = 0{,}097 \times 10^{-6} \times \frac{p_n}{p} \tag{A.3}$$

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

A.2.2 Results

The experimental results are processed as follows:

The viscous flow volume-specific surface area S_K [the Kozeny term in Formula (2)] is first considered and the corresponding viscous flow equivalent sphere diameter according to Formulae (A.4):

$$D_K = \frac{6}{S_K} \tag{A.4}$$

is calculated from Formula (A.5)

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

Then

a) If D_K is greater than about 10 µm, no slip flow correction is required, and the final result can be calculated Formulae (A.6) to (A.8):

$$S_V = S_K \tag{A.6}$$

$$D = D_K \tag{A.7}$$

$$S_w = \frac{S_K}{\varrho_e} \tag{A.8}$$

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 β defined by Formulae (A.9)

$$\beta = \frac{S_m}{2S_K} \tag{A.9}$$

This factor can be calculated from Formula (A.10)

$$\beta = \frac{10{,}2\left(1-\varepsilon_p\right)\eta}{\varepsilon_p D_K} \tag{A.10}$$

rewritten as Formulae (A.11)

$$\beta = 10{,}2 \times \frac{\left(1-\varepsilon_p\right)}{\varepsilon_p} \times \frac{\eta p_n}{D_K p} \tag{A.11}$$

where 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 Formulae (A.12) and (A.13):

$$S_K = \frac{6}{D_K} \tag{A.12}$$

$$S_m = \frac{12\beta}{D_K} \tag{A.13}$$

The final results are calculated using Formulae (A.14) to (A.16):

$$S_V = \frac{6}{D_K}\left(\beta + \sqrt{1+\beta^2}\right) \tag{A.14}$$

$$S_w = \frac{S_V}{\varrho_e} \tag{A.15}$$

$$D = \frac{D_k}{\beta + \sqrt{1+\beta^2}} \tag{A.16}$$

A.3 Gooden and Smith method

A.3.1 General

In this method, dry air is fed at a constant overpressure p_0, 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 Formula (A.17):

$$\Delta p = p_o - p' \tag{A.17}$$

The specific surface area is calculated using the Kozeny-Carman equation, Formula (9), in which Δp has been replaced by $p_o - p'$, q has been replaced by $C'p'$ (where C' is the capillary constant of the flowmeter) and both sides have been squared to give Formula (A.18):

$$S_w^2 = \frac{\varepsilon_p{}^3\left(p_o - p'\right)A}{5{,}0\left(1-\varepsilon_p\right)^2 C'p'\eta L\varrho_e^2} \tag{A.18}$$

A.3.2 Non-automated test device

Many years ago, a test device[1)] was developed to carry out this method (see Figure A.2). The test device includes a sliding chart which gives a direct reading of the powder 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 test device does not give an absolute determination. The test device 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 Formula (A.19)

$$D^2 = \frac{180C'\eta\left(1-\varepsilon_p\right)^2 p'L}{A\varepsilon_p^3\left(p_o - p'\right)} \tag{A.19}$$

where according to Formulae (A.20)

$$\varepsilon_p = 1 - \frac{1}{AL\varrho_e} \tag{A.20}$$

As the chart is designed for a mass of powder in the powder 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 according to Formulae (A.21):

$$\varepsilon_p = 1 - \frac{1}{AL} \tag{A.21}$$

where A is in square centimetres and L in centimetres, and Formula (A.19) can be written as Formula (A.22):

$$D = \frac{CL}{\left(AL-1\right)^{3/2}} \times \sqrt{\frac{h}{H-h}} \tag{A.22}$$

where

C is the flowmeter constant of the test device;

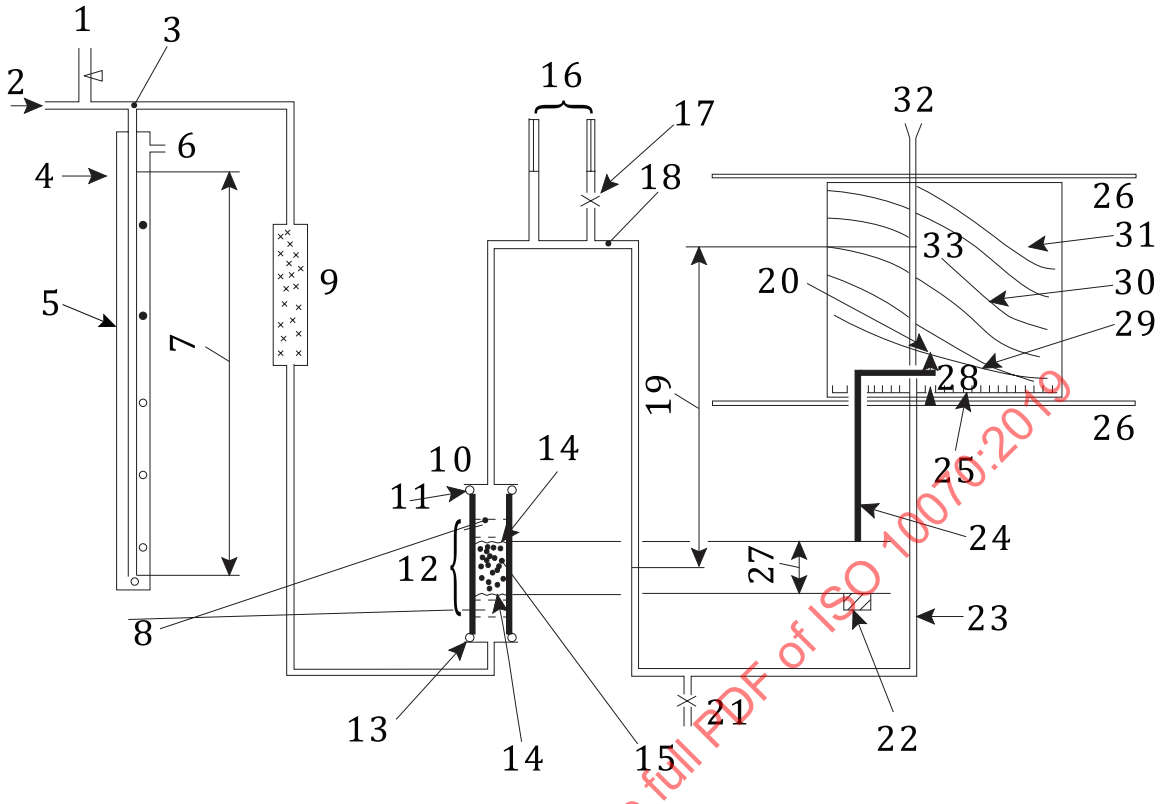
h is the manometer reading, in centimetres of water;

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) The Fisher Subsieve Sizer, which is no longer commercially available, nor is it supported with parts and service. It is included here because of several test devices still operating in the field. In-house repair or parts replacement is discouraged, as these have been found to detrimentally affect results and precision. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the apparatus named.



Key

1	air pressure control	18	b'
2	air	19	h
3	p_0	20	pointer
4	strandpipe water level	21	manometer levelling
5	constant pressure regulator	22	pillar
6	vent	23	flowmeter manometer
7	H	24	crossbar
8	plug	25	porosity scale
9	drying agent	26	rail
10	sample tube	27	L
11	rubber seal ring	28	ε
12	Δp	29	sample height curve
13	seal ring	30	particle size curves
14	filter paper	31	sliding chart
15	test portion	32	open to atmosphere
16	flowmeter resistances (needle valves)	33	d
17	range control		

Figure A.2 — Schematic diagram of the non-automated test device used in the Gooden and Smith method

The test device 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.3.2.1 Operating procedure

A.3.2.1.1 General

The instructions supplied by the manufacturer shall be followed except where indicated otherwise in the following subclauses. Particular attention shall be given to proper maintenance of the test device, 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.3.2.1.2 Calibration of test device

The manufacturer's instructions include calibration directions, using a Jewel calibrator tube[2)] as the primary standard. However, a calibration check is recommended before and after any single determination or series of determinations. The calibrator tube should be checked frequently for cleanness using a microscope.

A.3.2.1.3 Temperature of test

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

A.3.2.1.4 Mass of test portion

The mass in grams of the test portion shall be numerically equal (within ± 0,1 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.3.2.1.5 Determination of specific surface area

the determination is 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.3.2.1.6 Determination of porosity and mean sphere diameter — 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. The porosity and “average particle diameter” are read directly from the chart.

A.3.3 Automated test device

An automated digital test device[3)] (see Figure A.3) has recently been developed to carry out the Gooden and Smith method as a replacement for the non-automated test device. It operates similarly,

2) The Jewel calibrator tube is no longer commercially available. A “Master” Jewel calibrator tube is maintained by ASTM International Subcommittee B09.03 for calibration and traceability of currently existing in-house calibrator tubes. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the apparatus named.

3) The Sub-sieve AutoSizer (MIC SAS II™), manufactured and sold by Micromeritics Instrument Corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the apparatus named. Equivalent test devices may be used if they can be shown to lead to the same results.