INTERNATIONAL STANDARD

ISO 2738

Third edition
1999-11-01

Sintered metal materials, excluding hardmetals — Permeable sintered metal materials — Determination of density, oil content and open porosity

Matériaux métalliques frittés, à l'exclusion des métaux-durs — Matériaux métalliques frittés perméables — Détermination de la masse volumique, de la teneur en huile et de la porosité ouverte

Reference number
ISO 2738:1999(E)

Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 2738 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 3, *Sampling and testing methods for sintered metal materials (excluding hardmetals)*.

This third edition cancels and replaces the second edition (ISO 2738:1987) which has been technically revised.

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Sintered metal materials, excluding hardmetals — Permeable sintered metal materials — Determination of density, oil content and open porosity

1 Scope

This international Standard specifies methods of determining the density, oil content and open porosity of permeable sintered metal materials.

It applies in particular to porous metal bearings and to structural parts produced by pressing and sintering metal powders.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 758, *Liquid chemical products for industrial use — Determination of density at 20 °C.*

ISO 13944, *Lubricated metal-powder mixes — Determination of lubricant content — Modified Soxhlet extraction method.*

3 Symbols and designations

Table 1 — Symbols and designations

Symbol	Designation	Unit
m_1	Initial mass of the test piece	g
m_2	Mass of the test piece after oil extraction and drying	g
m_3	Mass of the fully impregnated test piece	g
m_a	Mass of the fully or partially impregnated test piece plus supporting device (for example suspension wire) weighed in air	g
m_w	Mass of the fully or partially impregnated test piece plus supporting device (for example suspension wire) weighed in water	g
V	Volume of the test piece	cm^3
ρ_w	Density of the water used	g/cm^3
ρ_1	Density of the oil initially in the test piece [a]	g/cm^3
ρ_2	Density of the impregnating oil used [a]	g/cm^3

[a] The oil density is assumed to be known or, if not, to be determined in accordance with ISO 758.

4 Terms and definitions

For the purposes of this International Standard the following terms and definitions apply.

4.1
density
the density of the test piece may be expressed in two ways:

4.1.1
dry density
the mass, after drying, divided by the volume

4.1.2
fully impregnated density (wet density)
the fully impregnated mass divided by the volume

4.2
oil content
the oil content of the test piece may be expressed in two ways:

4.2.1
percentage by volume
the volume of the oil divided by the volume of the test piece and multiplied by 100

4.2.2
percentage of the volume of the open porosity
the volume of the oil divided by the volume of the open porosity and multiplied by 100

4.3
open porosity (of the test piece)
the oil content after full impregnation divided by the volume of the test piece and multiplied by 100 expressed as a percentage by volume

4.4
volume (of the test piece)
total volume including the pores

5 Test procedures

Depending upon which of the properties is to be determined, some or all of the test procedures in clause 8 are carried out. Table 2 shows the test procedures that are carried out for the property to be determined. The values obtained for the various parameters are inserted in the respective formulae given in clause 9 to obtain the desired property.

Table 2 — Test procedures

Test procedure	Symbol for result obtained	Properties to be determined				
		Density		Oil content		Open porosity
		Dry	Fully impregnated	% (*V*/*V*)	% of open porosity	
Initial weighing of the test piece (see 8.1)	m_1			×	×	
Extraction of the oil contained in the pores of the test piece (see 8.2)		×		×	×	×
Determination of the mass of the test piece after oil extraction and drying (see 8.3)	m_2	×		×	×	×
Full impregnation of the test piece with an oil of known density (see 8.4)			×		×	×
Determination of the mass of the fully impregnated test piece (see 8.5)	m_3		×		×	×
Determination of the volume of the test piece (see 8.6)	V	×	×	×		×

6 Equipment

6.1 Analytical balance, of sufficient capacity and accurate to 0,01 %.

6.2 Soxhlet extractor, with oil solvent.

6.3 Device for weighing the test piece in air and in liquid

NOTE The liquid is usually water (see Figures 1, 2 and 3).

6.4 Vessel, large enough to accommodate the test piece and the device (6.3) for weighing it, containing distilled or deionized, and preferably degassed water, with 0,05 % (*V*/*V*) to 0,10 % (*V*/*V*) wetting agent added.

6.5 Apparatus for vacuum impregnation of the test piece with oil

6.6 Impregnation oil, of known density (see ISO 758 for the determination of the density of liquids).

6.7 Thermometer, accurate to ± 0,5 °C.

7 Test piece

7.1 Usually the test piece is tested whole. If this is not possible, the test piece may be cut or broken into smaller parts to facilitate the various operations. It is often most appropriate to test only a critical section of a component.

7.2 If the test piece has a mass less than 5 g a number of test pieces shall be tested together to obtain the average value.

7.3 The surface of the test piece shall be free of adhering dirt, grease or other foreign material.

7.4 The surface of the test piece shall be free from surplus oil. When removing any such surplus oil with an oil-absorbent material, care shall be taken to avoid removing oil contained in the pores.

NOTE The presence of surplus oil on the surface of the test piece is most likely to occur after the full impregnation treatment.

8 Test procedures

8.1 Determination of the initial mass of the test piece

Weigh the test piece in the condition in which it was received, to obtain m_1.

NOTE If the test piece is known to contain no oil, the procedures described in 8.2 and 8.3 are omitted. In this case, m_1 is substituted for m_2 in the formulae given in 9.1 and 9.3.

8.2 Removal of oil from the test piece by solvent extraction

Approximately 3 h of soaking and about ten solvent changes are required to remove the oil from test pieces of average density and small wall thickness. For thick walls and high density, up to 24 h are sometimes required.

NOTE 1 The Soxhlet extractor is a convenient apparatus for soaking the test piece in warm, freshly distilled oil solvent. The distillation rate determines the number of cycles and hence the number of solvent changes that occur. A suitable Soxhlet unit is described in ISO 13944.

Continue the extraction to constant mass after evaporation of the solvent left in the pores.

NOTE 2 Experience will indicate the best extraction time and distillation rate to use.

Dry the test piece to constant mass (i.e. until the reduction in mass produced by the last extraction does not exceed 0,01 % at a temperature of 20 °C above the boiling point of the solvent) then cool in a desiccator and weigh.

Choose the solvent so that complete dissolution of the oil in question is ensured. This requirement shall be tested for separately. The solvent used shall be stated in the test report.

For practical control purposes, other methods for removing the oil may be used (such as heating well below sintering temperatures in a protective atmosphere). In cases of dispute, the modified Soxhlet extraction method shall be the reference method (see ISO 13944).

8.3 Determination of the mass of the dried test piece

Weigh the test piece after solvent extraction and drying to obtain m_2.

8.4 Impregnation with oil and surface coating

8.4.1 Full impregnation (for determination of the open porosity)

Submerge the test piece in oil, contained in any suitable vessel capable of withstanding a vacuum. Reduce the pressure on the surface of the oil to 70 kPa maximum.

Continue the vacuum treatment until no further bubbles appear on the surface of the oil.

Restore the pressure in the vacuum chamber to that of the ambient atmosphere. Allow the test piece to remain submerged in the oil for a period of 10 min.

NOTE 1 For the majority of porous metals a single vacuum treatment is sufficient for ensuring full impregnation. In some cases a second vacuum treatment is necessary to achieve full impregnation. This can be established by reducing the pressure a second time, and if no further air bubbles appear, it can be safely assumed that the first treatment has achieved full impregnation.

The oil shall be completely immiscible with water and shall wet the porous metal.

NOTE 2 Generally the oil shall have a viscosity at 40 °C of between 22 mm^2/s and 68 mm^2/s[1)] which corresponds to ISO VG 22 to VG 68 as specified in ISO 3448. With a low-viscosity oil, impregnation is faster than with a high-viscosity oil.

Remove the test piece from the oil, allow to drain and remove the surplus surface oil as described in 7.4.

8.4.2 Partial impregnation (suitable for determination of the volume)

The requirements of the oil are the same as stated in 8.4.1.

Submerge the test piece in hot oil (70 °C ± 10 °C) until no further air bubbles appear. Cool the test piece to room temperature whilst still submerged in oil by removing it from the hot oil and quickly transferring it to cold oil. Remove the cooled test piece from the cold oil, allow to drain and remove the surplus surface oil as described in 7.4.

8.4.3 Surface coating methods (suitable for determination of the volume)

Coat the porous surface of the test piece with a film that, by means of surface tension, prevents the water from entering the pores.

The following techniques have been found to be suitable for particular types of porous metal. However, before any are used, the effectiveness of the technique with respect to the type and shape of porous metal shall first be established.

8.4.3.1 Petroleum jelly

Smear the surface of the test piece with petroleum jelly and remove any excess.

8.4.3.2 Silicones

Many silicone fluids produce surface films which are not wetted by water. Dip the test piece into either the silicone fluid or a dilute solution of the silicone fluid in a suitable solvent and dry to constant mass.

8.4.3.3 Paraffin wax

Dip the test piece in a 5 % solution of paraffin wax in a suitable solvent and dry to constant mass.

8.5 Determination of the mass of the fully impregnated test piece

Weigh the test piece after full oil impregnation to obtain m_3.

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1) 1 mm^2/s corresponds to 1cSt.

8.6 Determination of the volume of the test piece

8.6.1 Determine the volume V of the test piece by weighing in air to obtain m_a, and then submerge in water or other liquid of known density ρ_w to obtain m_w. The volume V in cm^3 is given by the equation:

$$V = \frac{m_a - m_w}{\rho_w}$$

8.6.2 With porous metals, it is essential that the liquid used is not absorbed by the pores. For this reason, the pores are impregnated with oil, and water is usually used as the test liquid. But it is not always necessary to impregnate a test piece fully; therefore, in order to ensure that no water enters the pores when the test piece is submerged in the water, the test piece may be partially impregnated or surface coated as described in 8.4.2 and 8.4.3. However, as a reference method, the test piece shall be fully impregnated with oil as described in 8.4.1.

NOTE After weighing in water, the test piece should be re-weighed in air (having removed any adhering water), to confirm that no water has been absorbed.

8.6.3 Figures 1, 2 and 3 show methods of suspending the test piece during weighing. In general, the mass and volume of the device should be as small as possible.

The maximum recommended diameters of non corroding metal suspension wires for various mass sizes are given in Table 3.

Immersion baskets also should be made of non corroding metal (see Figure 1).

Table 3 — Recommended wire diameters

Mass g	Wire diameter mm
mass $<$ 50	0,12
50 $\leqslant$ mass $<$ 200	0,25
200 $\leqslant$ mass $<$ 600	0,40
600 $\leqslant$ mass $\leqslant$ 1 000	0,50

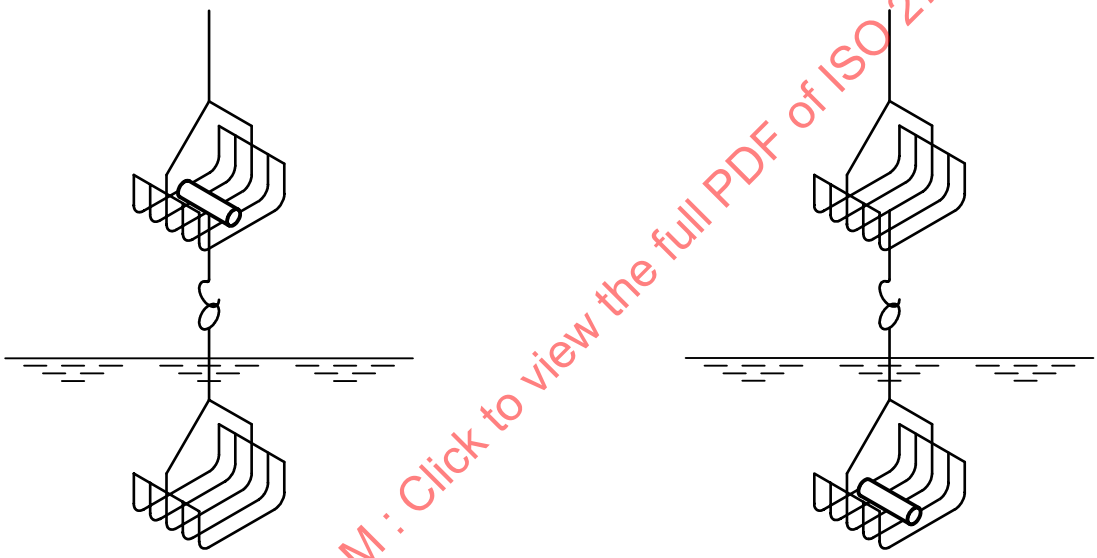
8.6.4 The test piece can be suspended from a piece of thin wire, and the total mass of the test piece and wire determined in air and in water. Allowance is made for the volume of the wire submerged in the water, but this is often insignificantly small when compared with the volume of the test piece. This allowance can be made by weighing the wire in air and then when submerged to an adequate depth in water, only the wire breaking the surface. Alternatively, the length of wire which is submerged can be measured, and the correction made based on the known volume of a unit length of wire.

8.6.5 Ensure that all air bubbles are removed from the surface of the test piece and the supporting device. It is recommended that 0,05 % (*V*/*V*) to 0,10 % (*V*/*V*) of wetting agent be added to the water.

8.6.6 The test piece and the water shall be at the same temperature. The normal test temperature is between 18 °C and 22 °C and the density, ρ_w of pure water in this range may be taken as 0,998 g/cm^3. For temperatures outside this range, the water density shall be taken from Table 4.

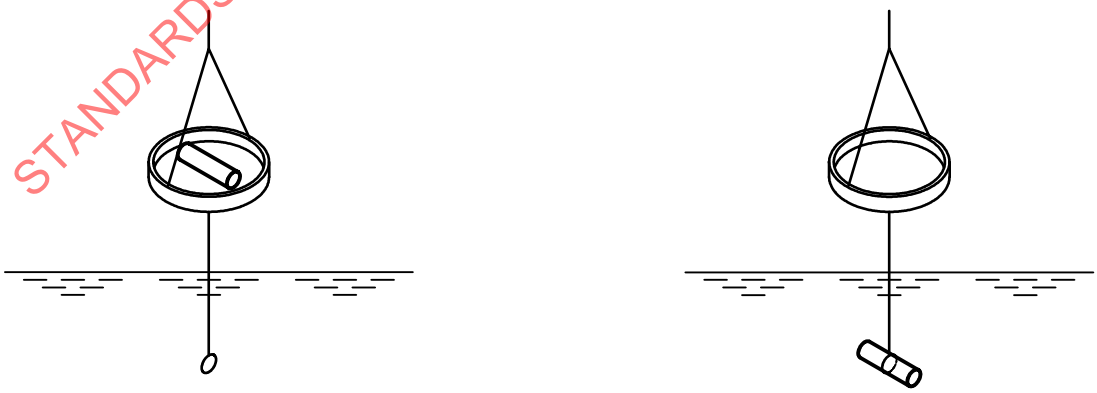
Table 4 — Density of air-free water[2)]

Temperature °C	Density g/cm³	Temperature °C	Density g/cm³
18	0,998 6	25	0,997 0
19	0,998 4	26	0,996 8
20	0,998 2	27	0,996 5
21	0,998 0	28	0,996 2
22	0,997 8	29	0,995 9
23	0,997 5	30	0,995 6
24	0,997 3		



a) Weighing in air m_a b) Weighing in water m_w

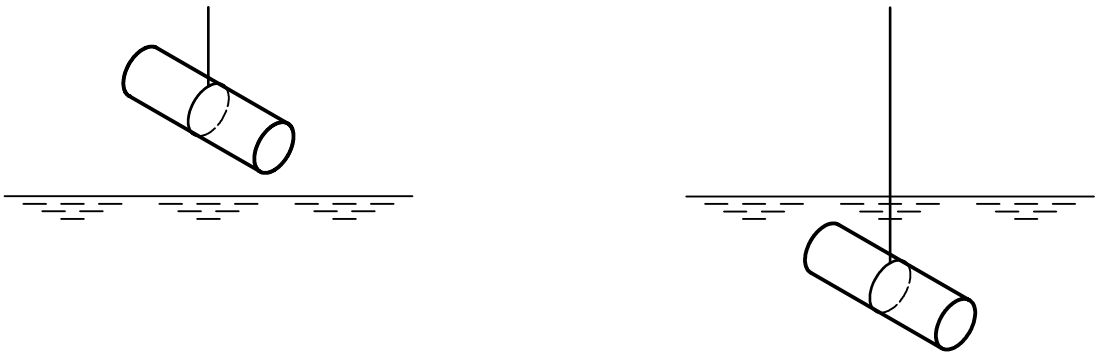
Figure 1



a) Weighing in air m_a b) Weighing in water m_w

Figure 2

2) See [2].



a) **Weighing in air** m_a b) **Weighing in water** m_w

Figure 3

9 Expression of results

9.1 Density

The dry density, expressed in g/cm³, is given by the formula:

$$\frac{m_2}{V} = \frac{m_2 \cdot \rho_w}{m_a - m_w}$$

The fully impregnated density (wet density), expressed in g/cm³, is given by the formula:

$$\frac{m_3}{V} = \frac{m_3 \cdot \rho_w}{m_a - m_w}$$

Report the density to the nearest 0,01 g/cm³.

9.2 Oil content

The oil content, expressed as a percentage by volume, is given by the formula:

$$\frac{m_1 - m_2}{\rho_1 \cdot V} \times 100$$

Report the oil content to the nearest 0,1 % (*V*/*V*).

The oil content, expressed as a percentage of the open porosity, is given by the formula:

$$\frac{m_1 - m_2}{\rho_1} \times \frac{\rho_2}{m_3 - m_2} \times 100$$

Report the oil content to the nearest 0,1 % in absolute value.

9.3 Open porosity

The open porosity, expressed as a percentage by volume, is given by the formula:

$$\frac{m_3 - m_2}{\rho_2 \cdot V} \times 100$$

Report the open porosity to the nearest 0,1 % (*V*/*V*).

10 Precision

10.1 For ferrous sintered parts, the repeatability interval I_r is 0,06 g/cm³ for dry or wet density. It is 1,6 percentage points for porosity. Duplicate results from the same laboratory should not be considered suspect at the 95 % confidence level unless they differ by more than I_r.

10.2 For ferrous sintered parts, the reproducibility interval I_R is 0,085 g/cm³ for dry or wet density and 2,4 percentage points for porosity. Test results from two different laboratories should not be considered suspect at the 95 % confidence level unless they differ by more than I_R.

11 Test report

The test report shall include the following information:

a) reference to this International Standard, i.e. ISO 2738;

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

c) whether the test piece has been subdivided or if a critical section has been tested (section described);

d) whether a number of test pieces have been tested together (number stated);

e) the method used and the result obtained;

f) the value of the density of the oil initially present in the test piece, as well as the origin of this value (measured, known or assumed) in the case of determination of oil content;

g) all operations not specified by this International Standard, or regarded as optional;

h) details of any occurrence which may have affected the result.