INTERNATIONAL STANDARD

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First edition
1996-12-15

Lubricated metal-powder mixes — Determination of lubricant content — Modified Soxhlet extraction method

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Mélanges de poudres métalliques lubrifiées — Détermination de la teneur en lubrifiant — Méthode d'extraction au Soxhlet, modifiée

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Reference number
ISO 13944:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

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International Standard ISO 13944 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 2, *Sampling and testing methods for powders (including powders for hardmetals)*.

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This first edition cancels and replaces ISO 4495:1978, of which it constitutes a technical revision.

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Lubricated metal-powder mixes — Determination of lubricant content — Modified Soxhlet extraction method

1 Scope

This International Standard specifies a method for the determination of the lubricant content of a powder mix. The method is also suitable for measuring the content of elements, e.g. graphite and oxygen, the determination of which is interfered with by the presence of a lubricant.

A condition of the application of the method is that a suitable solvent for the lubricant concerned is known and available.

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2 Principle

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The lubricant is extracted from a weighed test portion using a suitable solvent. The test portion is reweighed after the extraction, and the percentage mass loss, representing the extracted lubricant, calculated.

The extracted test portion can then be used to determine, by the normal methods, the content of other constituents, without any interference from the lubricant.

3 Apparatus and materials

3.1 Analytical balance, capable of weighing the sintered-glass filter crucible (see 3.2.3) together with the test portion to the nearest 1 mg.

3.2 Soxhlet apparatus, as shown in figure 1, with ungreased joints, consisting of the following parts:

3.2.1 Allihn (bulb-type) condenser.

3.2.2 Soxhlet extractor, with a volume of 150 ml to 200 ml.

3.2.3 Sintered-glass filter crucible (porosity grade P 160[1]), **filter paper** (with a filtering speed of 1 000 ml/min), **glass wool** and a **length of glass tubing** with a diameter of about 30 mm and long enough to serve the purpose mentioned in 5.3.

In cases when the lubricant content to be determined is less than 0,5 %, all these items shall be rinsed with the solvent (3.3) before use.

1) As defined in ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading classification and designation.*

3.2.4 Round-bottom flask, with a capacity of 500 ml, containing a boiling aid.

3.2.5 Heating mantle, of sufficient power to evaporate the solvent at a rate of not less than 25 ml/min.

3.3 Organic solvent, suitable for extraction of the lubricant concerned. Examples of such solvents are xylene, toluene and petroleum ether.

WARNING — Inhalation of the vapours of organic solvents such as toluene is dangerous.

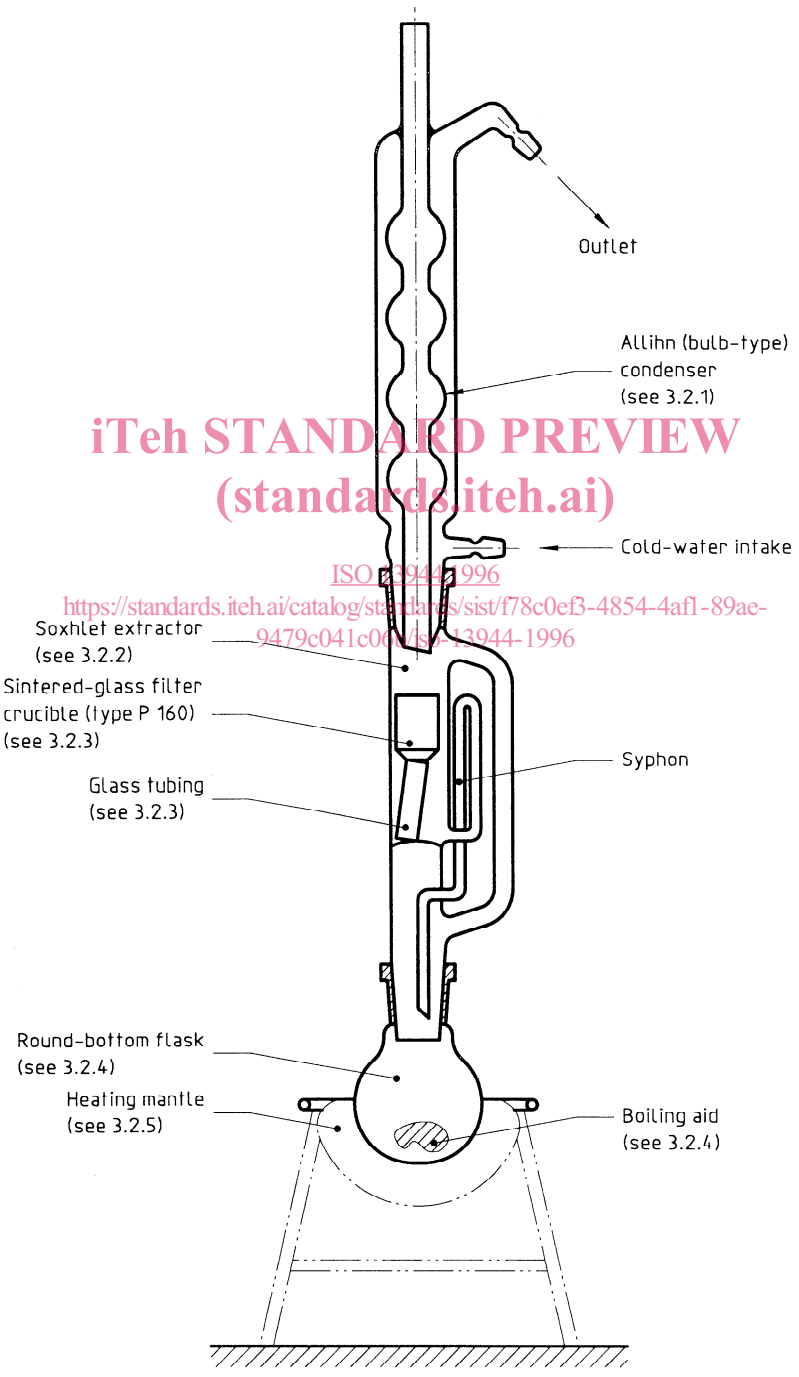

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Figure 1 — Soxhlet apparatus (3.2)

4 Test portions

4.1 The determination shall be carried out on two test portions.

4.2 In general, the test portions shall be taken from the powder in the as-received condition.

4.3 The mass of each test portion shall be approximately 50 g if the lubricant content is less than or equal to 2 % or approximately 25 g if the lubricant content is greater than 2 %.

5 Procedure

5.1 Weigh the filter crucible together with a filter paper and a plug of glass wool in the mouth of the crucible to the nearest 1 mg (m_1).

5.2 Place the test portion on the filter paper in the bottom of the crucible, and cover with the plug of glass wool. Weigh the crucible plus filter paper plus test portion plus glass wool together to the nearest 1 mg (m_2).

5.3 Place the crucible plus contents in the Soxhlet extractor, using the length of glass tubing to bring the upper edge of the crucible level with the upper bend of the syphon.

5.4 Introduce about 300 ml of the solvent into the round-bottom flask, and connect it to the Soxhlet extractor (see figure 1).

5.5 Heat the solvent to its boiling point. It is recommended that a preliminary test be made, for a given type of powder and solvent, to establish the minimum time required for complete extraction. In general, this time will be less than 30 min. For powders containing 0,8 % zinc stearate and with toluene as the solvent, an extraction time of 10 min to 20 min, concluding at the end of an extraction cycle, has been found to be sufficient. The time established in the preliminary test will apply to all further tests with the same combination of powder and solvent.

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5.6 On completion of the extraction, remove the crucible plus contents from the Soxhlet apparatus and suck dry on a Buchner flask. Then fill with fresh solvent and again suck dry. As a final washing operation, fill the crucible with diethyl ether (make sure that the temperature of the crucible and its contents is below 30 °C in order to avoid ignition) and suck dry. Allow any ether residues to evaporate for about 15 min in order to avoid ignition when the crucible is placed in a drying oven, where it is kept at 110 °C for 30 min.

5.7 After cooling in a desiccator, weigh the crucible plus contents to the nearest 1 mg (m_3).

5.8 Test portions can be taken by means of a small spoon from the powder remaining after the extraction for the determination of other constituents of the powder mix (e.g. graphite by means of equipment for the determination of carbon or oxygen by one of the methods described in the various parts of ISO 4491).

6 Expression of results

6.1 The lubricant content, expressed as a percentage by mass, is given by the formula

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

where

m_1 is the mass, in grams, of the crucible together with the filter paper and the glass-wool plug;

m_2 is the mass, in grams, of the crucible plus filter paper plus glass wool plus test portion;

m_3 is the mass, in grams, of the crucible plus contents after the extraction.

6.2 The maximum difference between the two determinations shall not exceed 0,1 % (*m/m*). If the difference exceeds 0,1 % (*m/m*), discard the results and repeat the two determinations with fresh test portions.

6.3 Report the arithmetic mean of the two determinations, rounded to the nearest 0,05 % (*m/m*).

7 Test report

The test report shall include the following information:

a) a reference to this International Standard;

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

c) the solvent used;

d) the extraction time;

e) the result obtained;

f) details of any operation not specified by this International Standard, as well as any operation regarded as optional;

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

h) the date of the test.

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