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

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

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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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)*.

This second edition cancels and replaces the first edition (ISO 13944:1996), which has been technically revised.

Lubricated metal-powder mixes — Determination of lubricant content — 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 preparing samples 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.

2 Principle

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

3.2.4 Round-bottomed 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.

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

3.4 Automated system for Soxhlet extraction method, which can be used as an alternative to the test set-up described in 3.2. The basic principles of an automated system should be the same as for the manual test set-up described in 3.2.

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

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). It is important to ensure that the filter paper is dry. If needed, the filter paper should be dried before the analysis to avoid errors caused by moisture.

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-bottomed flask, and connect it to the Soxhlet extractor (see Figure 1).

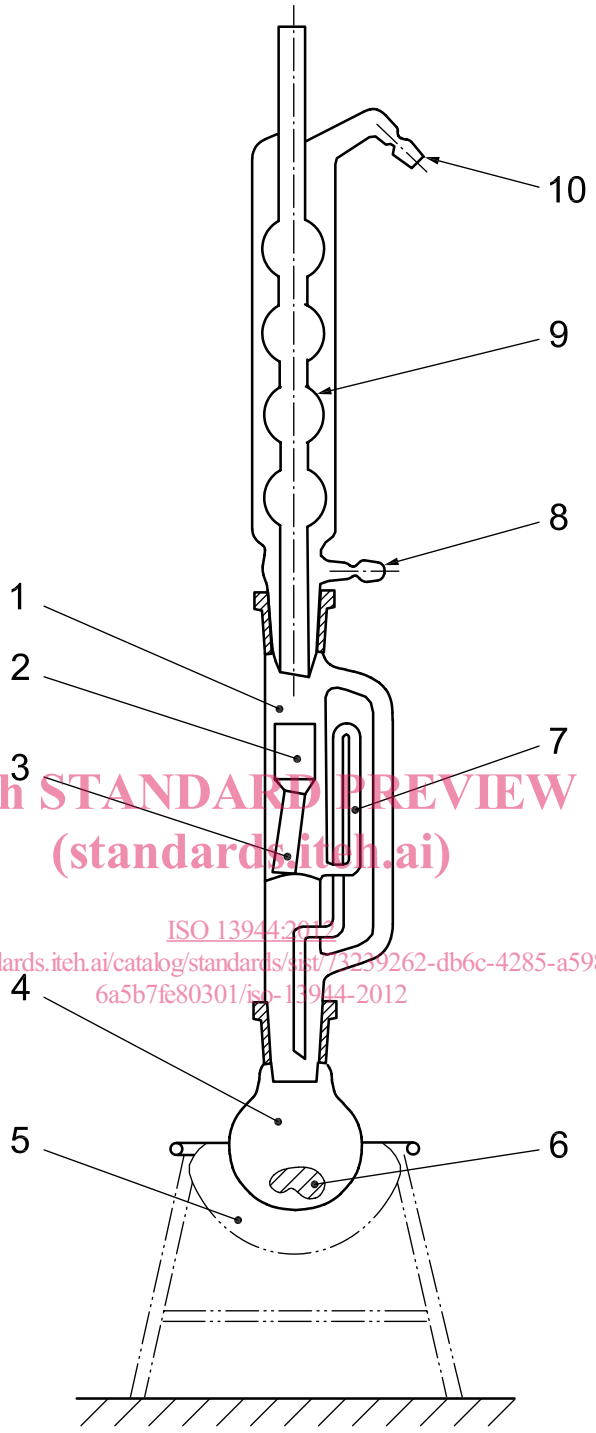
5.5 Heat the solvent to its boiling point. It is recommended that a preliminary test be made, for a given type of powder and solvent, to establish the minimum time required for complete extraction. In general, this time will be less than 30 min. For powders containing 0,8 % zinc stearate and with toluene 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.

5.6 On completion of the extraction, remove the crucible plus contents from the Soxhlet apparatus and suck dry on a Büchner flask. Then fill with fresh solvent and suck dry again. 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.

As an alternative to the final washing operation, the samples can be dried at 125 °C for 30 min under inert atmosphere.

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, *Metallic powders — Determination of oxygen content by reduction methods*). Before taking a test portion, the remaining sample shall be homogenized since the extraction procedure might have segregated the sample.



Key

1	Soxhlet extractor (see 3.2.2)	6	boiling aid (see 3.2.4)
2	sintered-glass filter crucible (Type P 160) (see 3.2.3)	7	syphon
3	glass tubing (see 3.2.3)	8	cold-water intake
4	round-bottomed flask (see 3.2.4)	9	Allihn (bulb-type) condenser (see 3.2.1)
5	heating mantle (see 3.2.5)	10	outlet

Figure 1 — Soxhlet apparatus (3.2)

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.