INTERNATIONAL STANDARD ISO 4495

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Lubricated metallic powders — Determination of lubricant content — Soxhlet extraction method

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

First edition — 1978-08-15

UDC 621.762 : 669-492.2 : 543.722

Ref. No. ISO 4495-1978 (E)

Descriptors : metallic powder, chemical analysis, determination of content, lubricants, extraction analysis.

Price based on 2 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4495 was developed by Technical Committee ISO/TC 119, *Powder metallurgical materials and products*, and was circulated to the member bodies in June 1977.

It has been approved by the member bodies of the following countries :

Austria	Italy	Spain
Bulgaria	Korea, Rep. of	Sweden
Canada	Mexico	Turkey
Chile	Poland	United Kingdom
Czechoslovakia	Portugal	U.S.A.
France	Romania	U.S.S.R.
Germany	South Africa, Rep. of	Yugoslavia

No member body expressed disapproval of the document.

Printed in Switzerland

Lubricated metallic powders — Determination of lubricant content — Soxhlet extraction method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the lubricant content of lubricated metallic powders under standardized conditions using the Soxhlet apparatus.

2 PRINCIPLE

Extraction of the lubricant by an appropriate solvent in a Soxhlet unit. Volatilization of the solvent after extraction and determination of the mass of the residue.

3 APPARATUS AND REAGENT

3.1 Analytical balance, capable of weighing at least 100 g to an accuracy of ± 0,001 g.

3.2 Soxhlet unit, as in the figure, consisting of the following parts :

3.2.1 Bulb-type condenser.

3.2.2 Soxhlet extractor of volume 75 to 100 ml.

3.2.3 Extraction thimble (cellulose), **filter paper** and **cotton wool,** which in the case where the lubricant content is below 0,5 % have been previously extracted with solvent.

The length of the thimble must be greater than the height of the syphon.

3.2.4 Round bottom flask with a capacity of 100 ml, containing a boiling aid.

NOTE — The conical joints of the Soxhlet unit must not be greased.

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

3.3 Evaporating dish.

3.4 Organic solvent, suitable for extraction of the lubricant involved. Examples of such solvents are chlorinated hydrocarbons, xylene, toluene and petroleum ether.

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

4 SAMPLING

4.1 In general, the powder should be tested in the as-received condition.

4.2 The lubricant content shall be determined on two test portions.

4.3 The mass of the test portion shall be approximately 10 g. (If the lubricant content is greater than 2 %, the mass of the test portion shall be approximately 5 g.)

5 PROCEDURE

5.1 Weigh, to the nearest 0,001 g, approximately 10 g (or 5 g) of the test sample. Pour it into the cellulose extraction thimble (3.2.3), which has previously been dried. Plug the extraction thimble with a swab of cotton-wool and insert in the extractor (3.2.2). Very fine grained powder must be wrapped in filter paper of suitable porosity before insertion into the thimble.

5.2 Weigh either the flask (3.2.4) together with the boiling aid, or the evaporating dish (3.3), to the nearest 0,001 g.

5.3 Fill the flask with about 75 ml of the solvent (3.4) and connect it to the Soxhlet extractor (see figure).

5.4 Heat the solvent to the boiling temperature. It is recommended that a preliminary test be made, for a given type of lubricated powder and appropriate solvent, to establish the minimum time required for full extraction. In general, this time will be at least 2 h. This time will apply for all further tests for the same combination of powder and solvent.

5.5 After the extraction is finished, evaporate the solvent in the flask, first by boiling gently on a water-bath until there is no visible solvent left and then in a drying cabinet at a temperature slightly (10 to 20 °C) above the boiling temperature of the solvent for 30 min.

Alternatively, after the extraction transfer the solution from the flask to the evaporating dish (see 5.2) and carry out the same procedure.

5.6 After cooling, weigh the flask or the evaporating dish to the nearest 0,001 g.

6 EXPRESSION OF RESULTS

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

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

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the flask with boiling aid, or of the evaporating dish;

m_3 is the mass, in grams, of the flask with boiling aid and residue, or of the evaporating dish and residue.

6.2 The maximum permissible difference between the two determinations shall not exceed 0,1 % in absolute value.

6.3 Report the arithmetic mean of the two determinations rounded to the nearest 0,05 %

7 TEST REPORT

The test report shall include the following information :

a) reference to this International Standard;

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

c) the solvent used;

d) time of extraction;

e) the result obtained;

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

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

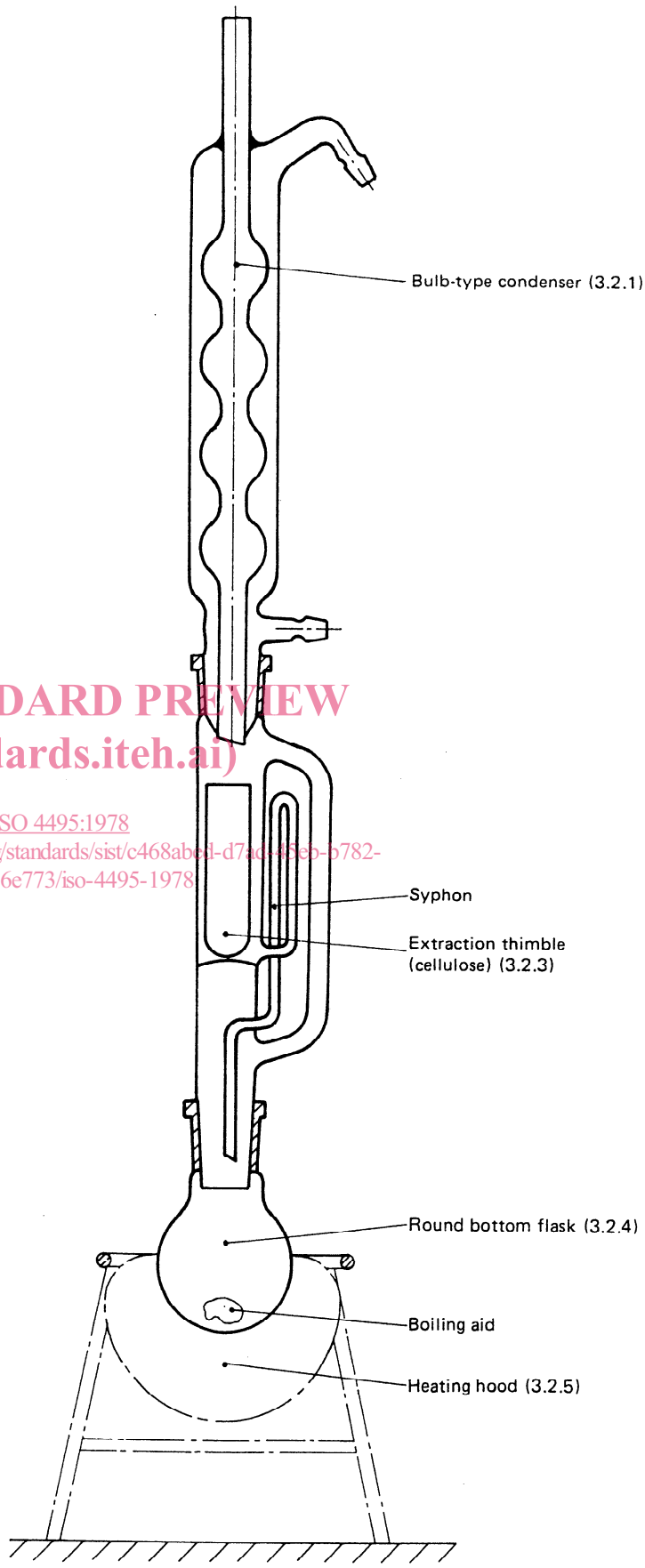

FIGURE — **Soxhlet unit (3.2)**