
INTERNATIONAL STANDARD



4495

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

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

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FOREWORD

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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.

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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.

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 $\pm 0,001$ 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.