

**ORGANIC MATERIALS
CONVENTIONAL AMOUNT OF ASH**

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No use restriction*This is a translation, the French original shall be used in all cases of litigation**Date of translation : 17/03/2004***FOREWORD***This document is in technical conformity with RNUR test method n° 1145.**It must not be modified without the agreement of RNUR.***1.OBJECT AND FIELD OF APPLICATION**

The object of this method is to determine the ash content of organic materials containing mineral fillers.

This method applies to the following materials :

- adhesives, adhesives-mastics, sealants, sound-deadening materials,
- paints, primers and similar products,
- gasket sealing compounds,
- thermosetting resins,
- thermoplastic and thermosetting materials containing fibreglass or mineral fillers,
- composite materials containing organic products (brake linings and clutch facings, friction bushes and laminated bushes),
- fibrous materials such as cloth, padding, cardboard and asbestos cloth.

Determination of the ash content in petroleum products (greases, oils...) shall be made using the test method D50 1404.

2.PRINCIPLE

This determination is based on the elimination of organic materials by calcination at a defined temperature and for a given time.

The temperature at which the calcination occurs is chosen in relation to the presumed composition of the material.

3.EQUIPMENT

- Balance accurate to within 0,1 mg.
- Silica dishes, 55 mm in diameter and 25 mm high.
- Bunsen burner.
- Muffle furnace able to reach the temperature of 1 000 °C, fitted with a temperature control device to within ± 20 °C.
- Desiccator.
- Drying agent such as phosphorous anhydride, calcium chloride, silica gel, etc...
- Ventilated oven.
- Crucible tongs.

4.TEST SPECIMENS

Three cases are to be considered :

4.1.MATERIALS IN SOLID FORM

Use test specimens of any shape but with maximum dimensions as follows :

- thickness : 2 mm for thermoset materials, 4 mm for thermofusible materials,

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- length and width : 35 mm.

The mass of test specimens must be between 1 and 4 g. If the test is to be carried out on parts of mass less than 1 g, use 2 or more so as to obtain a total mass greater than 1 g.

4.2.LIQUID OR PASTE MATERIALS FREE FROM SOLVENT (ADHESIVES AND MASTICS)

The sampling mass must be approximately 2 g.

In the case where the mass of ash obtained in these conditions is less than 10 mg, repeat the test with a greater quantity of product.

4.3.LIQUID OR PASTE MATERIALS CONTAINING A SOLVENT (RESINS, PAINTS, ADHESIVES, ETC...)

Determine, first of all, the amount of dry extract in the material according to the test method D55 1017. Deduce from it the sampling mass to be used so that approximately 2 g of the product dry extract is used.

5.METHOD OF OPERATION

5.1.PREPARATION OF THE DISH

Before each series of tests, weigh the dry, clean dish to within ± 1 mg. Place it in the muffle furnace at the selected test temperature, for 1 hour. After cooling in the desiccator, check that its mass has not changed after the treatment. Otherwise, repeat these operations until its mass is constant. Let m_1 be its mass in grams.

5.2.PREPARATION OF THE SAMPLE

Place the sample to be examined in the dish.

Three cases are then to be considered :

1. Thermoplastic and thermoset materials, composite materials.
Proceed with drying for 2 hours in a ventilated oven at 105 °C, cool in the desiccator and weigh, let m_2 be this mass in grams.
1. Liquid or paste materials not containing a solvent.
Weigh directly the sample contained in the dish, let m_2 be this mass in grams.
1. Liquid or paste materials containing a solvent.
Weigh directly the sample contained in the dish, let m_2 be this mass in grams.
Place, first of all, the dish containing the sample in a ventilated oven at the temperature and period of time defined for measuring the dry extract of the material in order to eliminate the solvent.
Place the dish containing the sample on a metallic support and ignite the sample with a Bunsen burner.
Repeat this operation several times until the material no longer burns (this operation shall not be carried out for paints, primers and similar products).

5.3.TEST PROCEDURE

- Set the muffle furnace at the test temperature stipulated in the standards or technical specifications.
- By default, select the test temperature from the following range in terms of the data shown in the appendix :
A : 450 °C \pm 20 °C
B : 600 °C \pm 20 °C
C : 800 °C \pm 20 °C
- Place the dish in the muffle furnace for 4 h \pm 0,5 h, except in special agreement (see appendix).
- Leave to cool in the desiccator and weigh, let m_3 be this mass in grams.

Number of tests :

Carry out the test on two samples. If the results vary by more than 5 %, proceed with an additional test on a third sampling.

6.CALCULATION AND EXPRESSION OF RESULTS

Calculate the ash content of the material using one of the two following formulae :

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FORMULA 1 : ash content in terms of the initial mass

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

FORMULA 2 : ash content in relation to 100 parts of dry extract

$$\frac{m_3 - m_1}{m_2 - m_1} \cdot \frac{100}{S}$$

dans lesquelles :
 m_1 = mass of the dish in grams
 m_2 = total initial mass of the dish and the sample, in grams
 m_3 = total mass of the dish and residue after calcination, in grams
 S = the amount of dry extract determined according to test method D55 1017.

7.TEST REPORT

The content must be as follows :

- the method of expression used for the calculation of the amount of ash (formula 1 or 2),
- the precise references of the material,
- the temperature used for the test, 450 °C, 600 °C, 800 °C, or other,
- the name of the supplier,
- the number of the manufacturing batch or the reference of the part from which the sample was taken.

It must also mention the appearance of the ash (containing fibreglass for example) as well as any incident or anomaly likely to have affected the results.

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appendix

CHOICE OF CALCINATION CONDITIONS

The amount of ash determined by a calcination test is reproducible only if the sample is not subjected to decomposition or oxidation in the vicinity of the test temperature selected.

In the absence of knowledge on the behaviour of the sample in terms of temperature, it is recommended to previously subject it to a thermogravimetry test and to select as the calcination temperature, from the three recommended temperatures (or if necessary, another temperature which shall be mentioned in the test report) situated at a plateau of the curve showing the evolution of mass in terms of the temperature. If necessary, ensure that the calcination time in the muffle furnace is neither insufficient nor excessive by controlling the variation in mass after a second period of one hour in the furnace.

A few general rules as a guidance to the choice of test temperature :

1. Compositions which may give rise during calcination to compounds which may be sublimated (lead, antimony salts...) shall be, if possible, subjected to the 450 °C test. Furthermore, it is recommended to operate with a dish fitted with one vent hole cover. This cover shall be removed when calcining on the Bunsen burner and replaced for introduction into the muffle furnace.
2. When the compositions contain a carbonate based (calcium, magnesium..) filler, it is recommended to avoid the temperature of 600 °C which is close to the critical temperature zone for decomposition of these compounds. It is advisable to operate either at 450 °C (before the carbonate decomposition), or at 800 °C (carbonate decomposed in the form of oxide).
3. When the compositions contain asbestos fibres which sustain a crystallisation water loss between 600 and 700 °C, it is recommended to calcinate, either at 450 °C, or at 800 °C, according to whether it is desirable to preserve the structure of these fibres. A similar phenomenon may be noted with other minerals such as clays.
4. Molybdenum bisulphide contained in certain compositions for its lubricating properties, is subjected, on contact with oxygen, to calcining and oxidation. It is recommended to use the temperature of 600 °C which allows the molybdenum bisulphide to be collected in the state of molybdenum trioxide (MoO_3). This compound sublimates above 700 °C.
5. For the measurement of ash in paints, it is usual to adopt a temperature of 800 °C.

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8.RECORDS AND REFERENCE DOCUMENTS

8.1.RECORDS

8.1.1.CREATION

- OR : 01/03/1981 – CREATION OF THE NORME

8.1.2.SUBJECT OF THE MODIFICATION

- A : 28/06/1996 – INTRODUCED INTO IDEM (*French only*)
- B : 24/11/1997 – CORRECTION TO THE INTRODUCTION INTO IDEM

8.2.REFERENCE DOCUMENTS

8.2.1.PSA DOCUMENTS

8.2.1.1.Normes

D501404, D551017.

8.2.1.2.Others

8.2.2.EXTERNAL DOCUMENTS

8.3.EQUIVALENT TO :

8.4.CONFORMS TO :

8.5.KEY WORDS

MATERIAUX, ORGANIQUES
(*Materials, Organic*)