

A.17. OXIDIZING PROPERTIES (SOLIDS)

1. METHOD

1.1. INTRODUCTION

It is useful to have preliminary information on any potentially explosive properties of the substance before performing this test.

This test is not applicable to liquids, gases, explosive or highly flammable substances, or organic peroxides.

This test need not be performed when examination of the structural formula establishes beyond reasonable doubt that the substance is incapable of reacting exothermically with a combustible material.

In order to ascertain if the test should be performed with special precautions, a preliminary test should be performed.

1.2. DEFINITION AND UNITS

Burning time: reaction time, in seconds, taken for the reaction zone to travel along a pile, following the procedure described in 1.6.

Burning rate: expressed in millimetres per second.

Maximum burning rate: the highest value of the burning rates obtained with mixtures containing 10 to 90 % by weight of oxidizer.

1.3. REFERENCE SUBSTANCE

Barium nitrate (analytical grade) is used as reference substance for the test and the preliminary test.

The reference mixture is that mixture of barium nitrate with powdered cellulose, prepared according to 1.6, which has the maximum burning rate (usually a mixture with 60% barium nitrate by weight).

1.4. PRINCIPLE OF THE METHOD

A preliminary test is carried out in the interests of safety. No further testing is required when the preliminary test clearly indicates that the test substance has oxidizing properties. When this is not the case, the substance should then be subject to the full test.

In the full test, the substance to be tested and a defined combustible substance will be mixed in various ratios. Each mixture is then formed into a pile and the pile is ignited at one end. The maximum burning rate determined is compared with the maximum burning rate of the reference mixture.

1.5. QUALITY CRITERIA

If required, any method of grinding and mixing is valid provided that the difference in the maximum rate of burning in the six separate tests differs from the arithmetic mean value by no more than 10 %.

1.6. DESCRIPTION OF THE METHOD

1.6.1. Preparation

1.6.1.1. Test substance

Reduce the test sample to a particle size $< 0,125$ mm using the following procedure: sieve the test substance, grind the remaining fraction, repeat the procedure until the whole test portion has passed the sieve.

Any grinding and sieving method satisfying the quality criteria may be used.

Before preparing the mixture the substance is dried at $105\text{ }^{\circ}\text{C}$, until constant weight is obtained. If the decomposition temperature of the substance to be tested is below $105\text{ }^{\circ}\text{C}$, the substance has to be dried at a suitable lower temperature.

1.6.1.2. Combustible substance

Powdered cellulose is used as a combustible substance. The cellulose should be a type used for thin-layer chromatography or column chromatography. A type with fibre-lengths of more than 85% between 0,020 and 0,075 mm has proved to be suitable. The cellulose powder is passed through a sieve with a mesh-size of 0,125 mm. The same batch of cellulose is to be used throughout the test.

Before preparing the mixture, the powdered cellulose is dried at $105\text{ }^{\circ}\text{C}$ until constant weight is obtained.

If wood-meal is used in the preliminary test, then prepare a soft-wood wood-meal by collecting the portion which passes through a sieve mesh of 1,6 mm, mix thoroughly, then dry at $105\text{ }^{\circ}\text{C}$ for four hours in a layer not more than 25 mm thick. Cool and store in an air-tight container filled as full as practicable until required, preferably within 24 hours of drying.

1.6.1.3. Ignition source

A hot flame from a gas burner (minimum diameter 5 mm) should be used as the ignition source. If another ignition source is used (e.g. when testing in an inert atmosphere), the description and the justification should be reported.

1.6.2. Performance of the test

Note:

Mixtures of oxidizers with cellulose or wood-meal must be treated as potentially explosive and handled with due care.

1.6.2.1. Preliminary test

The dried substance is thoroughly mixed with the dried cellulose or wood-meal in the proportions 2 of test substance to 1 of cellulose or wood-meal by weight and the mixture is formed into a small cone-shaped pile of dimensions 3,5 cm (diameter of base) x 2,5 cm (height) by filling, without tamping, a cone-shaped former (e.g. a laboratory glass funnel with the stem plugged).

The pile is placed on a cool, non-combustible, non-porous and low heat-conducting base plate. The test should be carried out in a fume cupboard as in 1.6.2.2.

The ignition source is put in contact with the cone. The vigour and duration of the resultant reaction are observed and recorded.

The substance is to be considered as oxidizing if the reaction is vigorous.

In any case where the result is open to doubt, it is then necessary to complete the full train test described below.

1.6.2.2. Train test

Prepare oxidizer cellulose-mixtures containing 10 to 90 % weight of oxidizer in 10 % increments. For borderline cases, intermediate oxidizer cellulose mixtures should be used to obtain the maximum burning rate more precisely.

The pile is formed by means of a mould. The mould is made of metal, has a length of 250 mm and a triangular cross-section with an inner height of 10 mm and an inner width of 20 mm. On both sides of the mould, in the longitudinal direction, two metal plates are mounted as lateral limitations which project 2 mm beyond the upper edge of the triangular cross-section (figure). This arrangement is loosely filled with a slight excess of mixture. After dropping the mould once from a height of 2 cm onto a solid surface, the remaining excess substance is scraped off with an obliquely positioned sheet. The lateral limitations are removed and the remaining powder is smoothed, using a roller. A non-combustible, non-porous and low heat-conducting base plate is then placed on the top of the mould, the apparatus inverted and the mould removed.

Arrange the pile across the draught in a fume cupboard.

The air-speed should be sufficient to prevent fumes escaping into the laboratory and should not be varied during the test. A draught screen should be erected around the apparatus.

Due to hygroscopicity of cellulose and of some substances to be tested, the test should be carried out as quickly as possible.

Ignite one end of the pile by touching with the flame.

Measure the time of reaction over a distance of 200 mm after the reaction zone has propagated an initial distance of 30 mm.

The test is performed with the reference substance and at least once with each one of the range of mixtures of the test substance with cellulose.

If the maximum burning rate is found to be significantly greater than that from the reference mixture, the test can be stopped; otherwise the test should be repeated five times for each of the three mixtures giving the fastest burning rate.

If the result is suspected of being a false positive, then the test should be repeated using an inert substance with a similar particle size, such as kieselguhr, in place of cellulose. Alternatively, the test substance cellulose mixture, having the fastest burning rate, should be retested in an inert atmosphere (< 2 % v/v oxygen content).

2. DATA

For safety reasons the maximum burning rate - not the mean value - shall be considered to be the characteristic oxidizing property of the substance under test.

The highest value of burning rate within a run of six tests of a given mixture is relevant for evaluation.

Plot a graph of the highest value of burning rate for each mixture versus the oxidizer concentration. From the graph take the maximum burning rate.

The six measured values of burning rate within a run obtained from the mixture with the maximum burning rate must not differ from the arithmetic mean value by more than 10 %; otherwise the methods of grinding and mixing must be improved.

Compare the maximum burning rate obtained with the maximum burning rate of the reference mixture (see 1.3).

If tests are conducted in an inert atmosphere, the maximum reaction rate is compared with that from the reference mixture in an inert atmosphere.

3. REPORT

3.1. TEST REPORT

The test report shall, if possible, include the following information:

- the identity, composition, purity, moisture content etc. of the substance tested;
- any treatment of the test sample (e.g. grinding, drying, ...);
- the ignition source used in the tests;
- the results of measurements;
- the mode of reaction (e.g. flash burning at the surface, burning through the whole mass, any information concerning the combustion products, ...);
- all additional remarks relevant for the interpretation of results, including a description of the vigour (flaming, sparking, fuming, slow smouldering, etc.) and approximate duration produced in the preliminary safety / screening test for both test and reference substance;
- the results from tests with an inert substance, if any;
- the results from tests in an inert atmosphere, if any.

3.2. INTERPRETATION OF THE RESULT

A substance is to be considered as an oxidizing substance when:

- (a) in the preliminary test, there is a vigorous reaction;
- (b) in the full test, the maximum burning rate of the mixtures tested is higher than or equal to the maximum burning rate of the reference mixture of cellulose and barium nitrate.

In order to avoid a false positive, the results obtained when testing the substance mixed with an inert material and/ or when testing under an inert atmosphere should also be considered when interpreting the results.

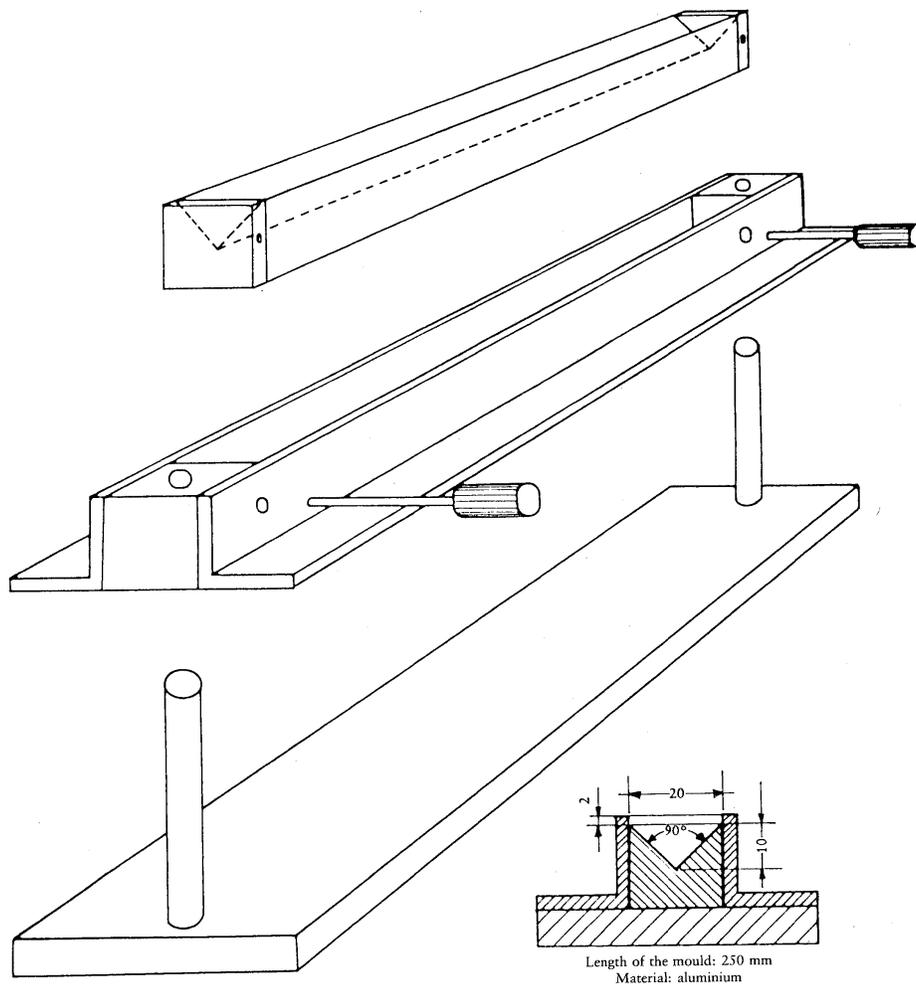
4. REFERENCES

- (1) NF T 20-035 (SEPT 85). Chemical products for industrial use. Determination of the oxidizing properties of solids.

Appendix

Figure

Mould and accessories for the preparations of the pile
(All dimensions in millimetres)



Please notice that only European Community's legislation published in the paper editions of the Official Journal of the European Communities is deemed authentic. When preparing this document, care has been taken to ensure correctness of the text; nevertheless possibility of errors cannot be completely excluded, so differences may exist between this version and the one agreed and published in the paper edition of the Official Journal. In case of doubt the reader is advised to consult the Official Journal.

This method can be found in Dir 92/69/EEC (O.J. L383 A)
A complete list of Annex V Testing Methods and the corresponding OJ can be downloaded from a previous page in this site.