

Chapter 2.3

Test methods

2.3.0 General

Unless otherwise provided for in Chapter 2.2 or in this Chapter, the test methods to be used for the classification of dangerous goods are those described in the Manual of Tests and Criteria.

2.3.1 Exudation test for blasting explosives of Type A

2.3.1.1 Blasting explosives of type A (UN No. 0081) shall, if they contain more than 40% liquid nitric ester, in addition to the testing specified in the Manual of Tests and Criteria, satisfy the following exudation test.

2.3.1.2 The apparatus for testing blasting explosive for exudation (figs. 1 to 3) consists of a hollow bronze cylinder. This cylinder, which is closed at one end by a plate of the same metal, has an internal diameter of 15.7 mm and a depth of 40 mm. It is pierced by 20 holes 0.5 mm in diameter (four sets of five holes) on the circumference. A bronze piston, cylindrically fashioned over a length of 48 mm and having a total length of 52 mm, slides into the vertically placed cylinder. The piston, whose diameter is 15.6 mm, is loaded with a mass of 2 220 g so that a pressure of 120 kPa (1.20 bar) is exerted on the base of the cylinder.

2.3.1.3 A small plug of blasting explosive weighing 5 to 8 g, 30 mm long and 15 mm in diameter, is wrapped in very fine gauze and placed in the cylinder; the piston and its loading mass are then placed on it so that the blasting explosive is subjected to a pressure of 120 kPa (1.20 bar).

The time taken for the appearance of the first signs of oily droplets (nitroglycerine) at the outer orifices of the cylinder holes is noted.

2.3.1.4 The blasting explosive is considered satisfactory if the time elapsing before the appearance of the liquid exudations is more than five minutes, the test having been carried out at a temperature of 15 °C to 25 °C.

Test of blasting explosive for exudation

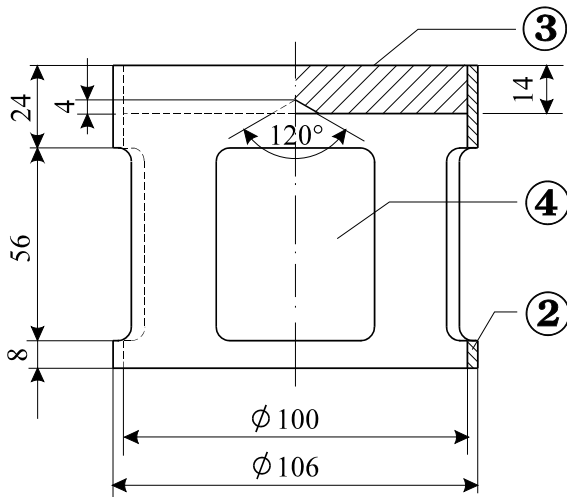


Fig. 1: Bell-form charge; mass 2220 g; capable of being suspended from a bronze piston

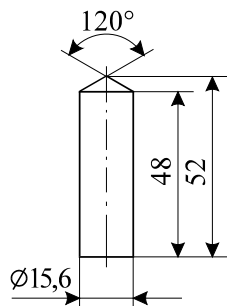


Fig. 2: Cylindrical bronze piston; dimensions in mm

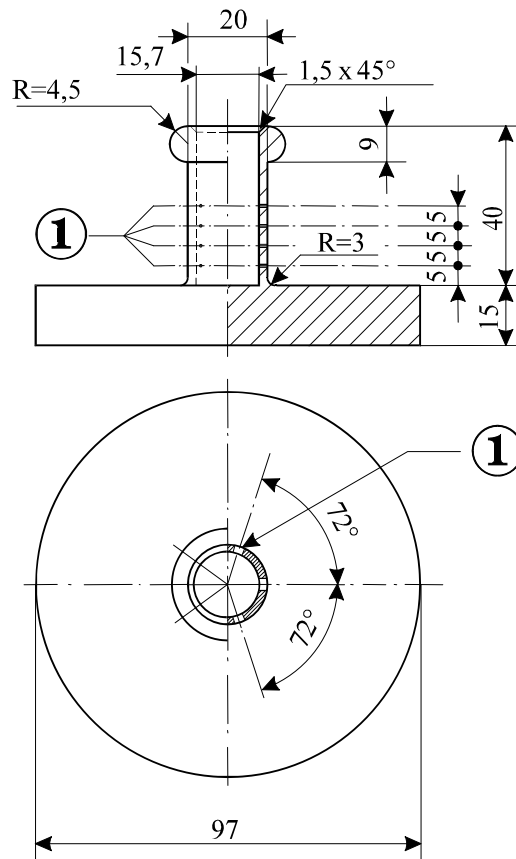


Fig. 3: Hollow bronze cylinder, closed at one end
Plan and cut; dimensions in mm

Fig. 1 to 3:

- (1) 4 series of 5 holes at 0.5 \varnothing
- (2) copper
- (3) iron plate with centre cone at the inferior face
- (4) 4 openings, approximately 46 x 56, set at even intervals on the periphery

2.3.2 Tests relating to nitrated cellulose mixtures of Class 4.1

- 2.3.2.1 Nitrocellulose heated for half an hour at 132 °C shall not give off visible yellowish-brown nitrous fumes (nitrous gases). The ignition temperature shall be above 180 °C. See 2.3.2.3 to 2.3.2.8, 2.3.2.9 (a) and 2.3.2.10 below.
- 2.3.2.2 3 g of plasticized nitrocellulose, heated for one hour at 132 °C, shall not give off visible yellowish-brown nitrous fumes (nitrous gases). The ignition temperature shall be above 170 °C. See 2.3.2.3 to 2.3.2.8, 2.3.2.9 (b) and 2.3.2.10 below.
- 2.3.2.3 The test procedures set out below are to be applied when differences of opinion arise as to the acceptability of substances for carriage by rail.
- 2.3.2.4 If other methods or test procedures are used to verify the conditions of stability prescribed above in this section, those methods shall lead to the same findings as could be reached by the methods specified below.
- 2.3.2.5 In carrying out the stability tests by heating described below, the temperature of the oven containing the sample under test shall not deviate by more than 2 °C from the prescribed temperature; the prescribed duration of a 30-minute or 60-minute test shall be observed to within two minutes. The oven shall be such that the required temperature is restored not more than five minutes after insertion of the sample.

2.3.2.6 Before undergoing the tests in 2.3.2.9 and 2.3.2.10, the samples shall be dried for not less than 15 hours at the ambient temperature in a vacuum desiccator containing fused and granulated calcium chloride, the sample substance being spread in a thin layer; for this purpose, substances which are neither in powder form nor fibrous shall be ground, or grated, or cut into small pieces. The pressure in the desiccator shall be brought below 6.5 kPa (0.065 bar).

2.3.2.7 Before being dried as prescribed in 2.3.2.6 above, substances conforming to 2.3.2.2 shall undergo preliminary drying in a well-ventilated oven, with its temperature set at 70 °C, until the loss of mass per quarter-hour is less than 0.3% of the original mass.

2.3.2.8 Weakly nitrated nitrocellulose conforming to 2.3.2.1 shall first undergo preliminary drying as prescribed in 2.3.2.7 above; drying shall then be completed by keeping the nitrocellulose for at least 15 hours over concentrated sulphuric acid in a desiccator.

2.3.2.9 Test of chemical stability under heat

(a) Test of the substance listed in paragraph 2.3.2.1 above.

(i) In each of two glass test tubes having the following dimensions:

length	350 mm,
internal diameter	16 mm,
thickness of wall	1.5 mm

is placed 1 g of substance dried over calcium chloride (if necessary the drying shall be carried out after reducing the substance to pieces weighing not more than 0.05 g each). Both test tubes, completely covered with loose-fitting closures, are then so placed in an oven that at least four-fifths of their length is visible, and are kept at a constant temperature of 132 °C for 30 minutes. It is observed whether nitrous gases in the form of yellowish-brown fumes clearly visible against a white background are given off during this time.

(ii) In the absence of such fumes the substance is deemed to be stable.

(b) Test of plasticized nitrocellulose (see 2.3.2.2)

(i) 3 g of plasticized nitrocellulose are placed in glass test tubes, similar to those referred to in (a), which are then placed in an oven kept at a constant temperature of 132 °C.

(ii) The test tubes containing the plasticized nitrocellulose are kept in the oven for one hour. During this time no yellowish-brown nitrous fumes (nitrous gases) shall be visible. Observation and appraisal as in (a).

2.3.2.10 Ignition temperature (see 2.3.2.1 and 2.3.2.2)

(a) The ignition temperature is determined by heating 0.2 g of substance enclosed in a glass test tube immersed in a Wood's alloy bath. The test tube is placed in the bath when the latter has reached 100 °C. The temperature of the bath is then progressively increased by 5 °C per minute;

(b) The test tubes must have the following dimensions:

length	125 mm,
internal diameter	15 mm,
thickness of wall	0.5 mm

and shall be immersed to a depth of 20 mm;

(c) The test shall be repeated three times, the temperature at which ignition of the substance occurs, i.e., slow or rapid combustion, deflagration or detonation, being noted each time;

(d) The lowest temperature recorded in the three tests is the ignition temperature.

2.3.3 Tests relating to flammable liquids of Classes 3, 6.1 and 8

2.3.3.1 Determination of flash-point

2.3.3.1.1 The following methods for determining the flash-point of flammable liquids may be used:

International standards:

ISO 1516 (Determination of flash/no flash – Closed cup equilibrium method)

ISO 1523 (Determination of flash point – Closed cup equilibrium method)

ISO 2719 (Determination of flash point – Pensky-Martens closed cup method)

ISO 13736 (Determination of flash point – Abel closed-cup method)

ISO 3679 (Determination of flash point – Rapid equilibrium closed cup method)

ISO 3680 (Determination of flash/no flash – Rapid equilibrium closed cup method)

National standards:

American Society for Testing Materials International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, Pennsylvania, USA 19428-2959;

ASTM D3828-07a, Standard Test Methods for Flash Point by Small Scale Closed-Cup Tester

ASTM D56-05, Standard Test Method for Flash Point by Tag Closed-Cup Tester

ASTM D3278-96(2004)e1, Standard Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus

ASTM D93-08, Standard Test Methods for Flash Point by Pensky-Martens Closed-Cup Tester

Association française de normalisation, AFNOR, 11, rue de Pressensé, F-93571 La Plaine Saint-Denis Cedex:

French standard NF M 07 - 019

French standards NF M 07 - 011 / NF T 30 - 050 / NF T 66 - 009

French standard NF M 07 - 036

Deutsches Institut für Normung, Burggrafenstraße 6, D-10787 Berlin:

Standard DIN 51755 (flash-points below 65 °C)

State Committee of the Council of Ministers for Standardization, RUS-113813, GSP, Moscow, M-49 Leninsky Prospect, 9:

GOST 12.1.044-84.

2.3.3.1.2 To determine the flash-point of paints, gums and similar viscous products containing solvents, only apparatus and test methods suitable for determining the flash-point for viscous liquids shall be used, in accordance with the following standards:

(a) International standard ISO 3679:1983;

(b) International standard ISO 3680:1983;

(c) International standard ISO 1523:1983;

(d) International standards EN ISO 13736 and EN ISO 2719, Method B.

2.3.3.1.3 The standards listed in 2.3.3.1.1 shall only be used for flash-point ranges which are specified therein. The possibility of chemical reactions between the substance and the sample holder shall be considered when selecting the standard to be used. The apparatus shall, as far as is consistent with safety, be placed in a draught-free position. For safety, a method utilizing a small sample size, around 2 ml, shall be used for organic peroxides and self-reactive substances (also known as "energetic" substances), or for toxic substances.

2.3.3.1.4 When the flash-point, determined by a non-equilibrium method is found to be 23 °C ± 2 °C or 60 °C ± 2 °C, it shall be confirmed for each temperature range by an equilibrium method.

2.3.3.1.5 In the event of a dispute as to the classification of a flammable liquid, the classification proposed by the consignor shall be accepted if a check-test of the flash-point, yields a result not differing by more than 2 °C from the limits (23 °C and 60 °C respectively) stated in 2.2.3.1. If the difference is more than 2 °C, a second check-test shall be carried out, and the lowest figure of the flash-points obtained in either check-test shall be adopted.

2.3.3.2 Determination of initial boiling point

The following methods for determining the initial boiling point of flammable liquids may be used:

International standards:

ISO 3924 (Petroleum products – Determination of boiling range distribution – Gas chromatography method)

ISO 4626 (Volatile organic liquids – Determination of boiling range of organic solvents used as raw materials)

ISO 3405 (Petroleum products – Determination of distillation characteristics at atmospheric pressure)

National standards:

American Society for Testing Materials International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, Pennsylvania, USA 19428-2959;

ASTM D86-07a, Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure

ASTM D1078-05, Standard Test Method for Distillation Range of Volatile Organic Liquids

Further acceptable methods:

Method A.2 as described in Part A of the Annex to Commission Regulation (EC) No 440/2008²³.

2.3.3.3 Test for determining peroxide content

To determine the peroxide content of a liquid, the procedure is as follows:

A quantity p (about 5 g, weighed to the nearest 0.01 g) of the liquid to be titrated is placed in an Erlenmeyer flask; 20 cm³ of acetic anhydride and about 1 g of powdered solid potassium iodide are added; the flask is shaken and, after 10 minutes, heated for 3 minutes to about 60 °C. When it has been left to cool for 5 minutes, 25 cm³ of water are added. After this, it is left standing for half an hour, then the liberated iodine is titrated with a decinormal solution of sodium thiosulphate, no indicator being added; complete discoloration indicates the end of the reaction. If n is the number of cm³ of thiosulphate solution required, the percentage of peroxide (calculated as H₂O₂) present in the sample is obtained by the formula:

$$\frac{17n}{100p}$$

2.3.4 Test for determining fluidity

To determine the fluidity of liquid, viscous or pasty substances and mixtures, the following test method shall be used.

2.3.4.1 Test apparatus

Commercial penetrometer conforming to ISO 2137:1985, with a guide rod of 47.5 g ± 0.05 g;
sieve disc of duralumin with conical bores and a mass of 102.5 g ± 0.05 g (see Figure 1);
penetration vessel with an inside diameter of 72 mm to 80 mm for reception of the sample.

2.3.4.2 Test procedure

The sample is poured into the penetration vessel not less than half an hour before the measurement. The vessel is then hermetically closed and left standing until the measurement. The sample in the hermetically closed penetration vessel is heated to 35 °C ± 0.5 °C and is placed on the penetrometer table immediately prior to measurement (not more than two minutes). The point S of the sieve disc is then brought into contact with the surface of the liquid and the rate of penetration is measured.

2.3.4.3 Evaluation of test results

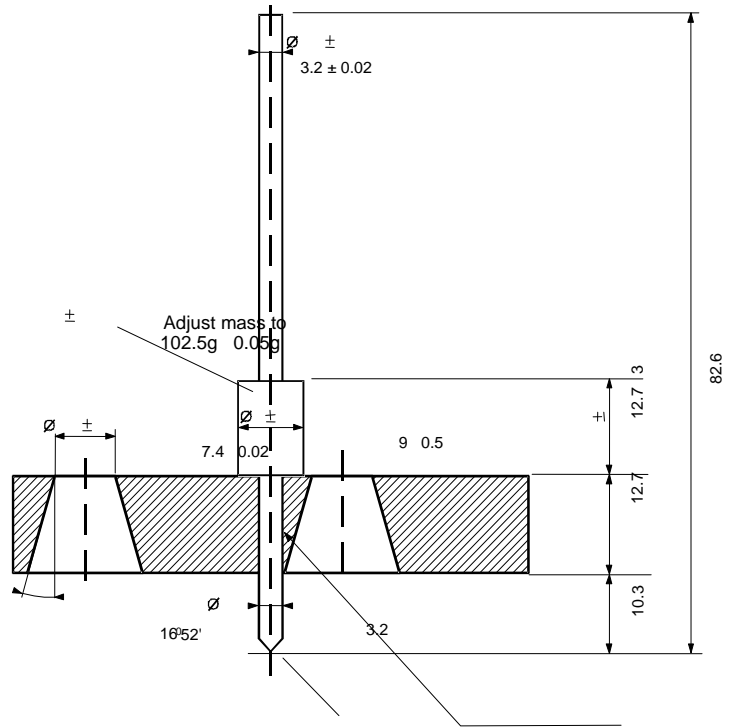
A substance is pasty if, after the centre S has been brought into contact with the surface of the sample, the penetration indicated by the dial gauge:

- (a) after a loading time of 5 s ± 0.1 s, is less than 15.0 mm ± 0.3 mm; or
- (b) after a loading time of 5 s ± 0.1 s, is greater than 15.0 mm ± 0.3 mm, but the additional penetration after another 55 s ± 0.5 s is less than 5.0 mm ± 0.5 mm.

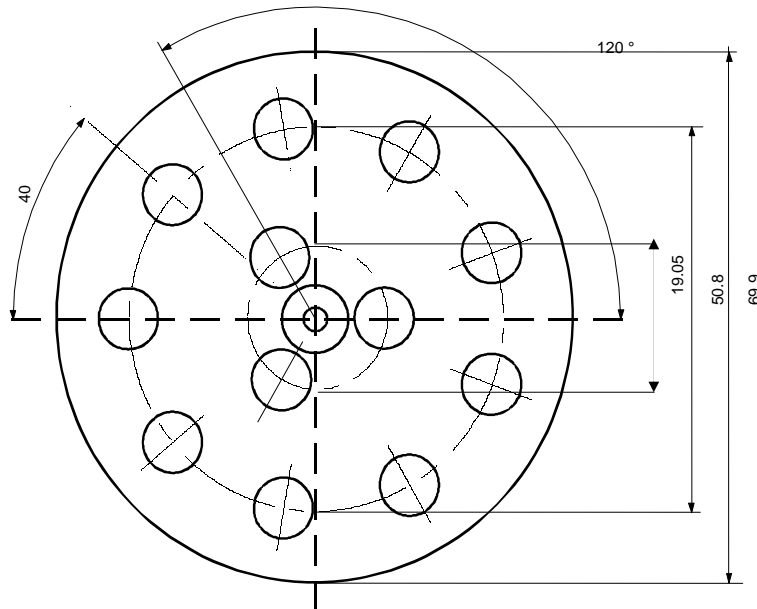
NOTE: In the case of samples having a flow point, it is often impossible to produce a steady level surface in the penetration vessel and, hence, to establish satisfactory initial measuring conditions for the contact of the point S. Furthermore, with some samples, the impact of the sieve disc can cause an elastic deformation of the surface and, in the first few seconds, simulate a deeper penetration. In all these cases, it may be appropriate to make the evaluation in paragraph (b) above.

²³ Commission Regulation (EC) No 440/2008 of 30 May 2008 laying down test methods pursuant to Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) (Official Journal of the European Union, No. L 142 of 31 May 2008, p.1-739 and No. L 143 of 3 June 2008, p.55).

Figure 1
Penetrometer



Press



Tolerances not specified are ± 0.1 mm.

2.3.5 Classification of organometallic substances in classes 4.2 and 4.3

Depending on their properties as determined in accordance with tests N.1 to N.5 of the Manual of Tests and Criteria, Part III, section 33, organometallic substances may be classified in Class 4.2 or 4.3, as appropriate, in accordance with the flowchart scheme given in Figure 2.3.5.

NOTE 1: Depending on their other properties and on the precedence of hazard table (see 2.1.3.10), organometallic substances may have to be classified in other classes as appropriate.

2: Flammable solutions with organometallic compounds in concentrations which are not liable to spontaneous combustion or, in contact with water, do not emit flammable gases in dangerous quantities, are substances of Class 3.

Figure 2.3.5: Flowchart scheme for the classification of organometallic substances in classes 4.2 and 4.3^{(a), (b)}

- (a) Test methods N.1 to N.5 can be found in the Manual of Tests and Criteria, Part III, Section 33.
- (b) If applicable and testing is relevant, taking into account reactivity properties, Class 6.1 and 8 properties should be considered according to the table of precedence of hazards in 2.1.3.10.

