
**Plastics — Smoke generation —
Determination of the corrosivity of fire
effluents —**

Part 4:

**Dynamic decomposition method using a
conical radiant heater**

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*Plastiques — Production de fumées — Détermination de la corrosivité des
effluents du feu —*

*Partie 4: Méthode de décomposition dynamique utilisant un radiateur
conique*
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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard 11907-4 was prepared by Technical Committee ISO/TC61, *Plastics*, Subcommittee SC 4, *Burning behaviour*.

ISO 11907 consists of the following parts, under the general title *Plastics – Smoke generation – Determination of the corrosivity of fire effluents*:

- Part 1: Guidance
- Part 2: Static method
- Part 3: Dynamic decomposition method using a travelling furnace
- Part 4: Dynamic decomposition method using a conical radiant heater

Annex A of this part of ISO 11907 is for information only.

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Foreword
Foreword Switzerland

Plastics — Smoke generation — Determination of the corrosivity of fire effluents —

Part 4:

Dynamic decomposition method using a conical radiant heater

WARNINGS

1 Avoidance of misleading inferences

This standard method of test should be used solely to measure and describe the properties of materials, products or systems in response to heat or flame under controlled laboratory conditions and should not be considered or used by itself for describing or appraising the fire hazard of materials, products or systems under actual fire conditions or as the sole source on which regulations pertaining to corrosivity of fire effluents are based.

2 Avoidance of danger to test operators

The test procedures involve high temperatures and combustion products. Therefore, conditions exist for burns, ignition of extraneous objects or clothing, and inhalation of combustion products. Use eye protection, and use protective gloves for insertion and removal of test specimens. Do not touch either the cone heater or the associated fixtures while hot, except with the use of protective gloves.

Vent the combustion products flowing through the exposure chamber through a properly designed exhaust system. Vent the captured combustion products in a smoke hood at the end of a test.

Check the exhaust system for proper operation before testing, and discharge into a building exhaust system with adequate capacity. Make provisions for collecting and venting any combustion products that for whatever reason fail to be collected by the normal exhaust system of the apparatus.

1 Scope

1.1 This part of ISO 11907 specifies a test method for measuring the corrosive effect, by loss of metal from a target, of the combustion effluents of plastic materials or products.

1.2 The test method is intended for the evaluation of materials or products, for additional data to assist in the design of products, and for development and research purposes.

1.3 The method provides corrosion results for plastic materials or product specimens limited to a maximum size of 100 mm × 100 mm in area. The results of this method have not been investigated with respect to correlation to actual fires.

1.4 The method measures the increase in electrical resistance of a metal circuit. Such increase is related to the decrease in conductive cross-sectional area resulting from the metal loss due to corrosion.

1.5 The method should be used to measure and describe the response of materials and/or products to heat and flame under controlled conditions but should not be used to describe or appraise the fire hazard or fire risk of materials under actual fire conditions. However, results of this test may be used as elements of a fire hazard or risk assessment as they relate to a particular end use.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 11907. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 11907 are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5660-1:1993, *Fire tests – Reaction to Fire – Part 1: Rate of heat release from building products (Cone calorimeter method)*.

ISO 11907-1:1998, *Plastics – Smoke generation – Determination of the corrosivity of fire effluents – Part 1: Guidance*.

3 Definitions

For the purposes of this part of ISO 11907, the following definitions apply.

3.1 corrosion: The reaction of a metallic material with its environment, resulting in a measurable change of the material and possibly in an impairment of the functioning of a metal part or of an entire system.

NOTE – In most cases, the reaction is electrochemical. In others, however, it may be chemical (non-electrochemical) or physical.

3.2 corrosion damage: The physical and/or chemical damage or impaired function caused by chemical action.

3.3 corrosion by metal loss: The loss of metal on a target, expressed as reduction in thickness of the target metal.

3.4 corrosion testing device: Equipment used to determine corrosion in this standard.

3.5 corrosion target: The sensor used to determine, under specified test conditions, the degree of corrosion damage.

NOTE – This sensor can be a product, a component or a reference material used to simulate them.

3.6 irradiance (at a point on a surface): The radiant flux incident on an infinitesimally small element of the surface containing the point, divided by the area of that element.

NOTE – The specimen, once ignited, is also heated by its own flame.

3.7 exposure chamber: The enclosure in which a target is exposed to combustion products.

3.8 environmental chamber: The enclosure used to expose the targets to high humidity and temperature after the exposure to combustion products.

3.9 sustained flaming: Existence of a flame on or over the surface of the specimen for a minimum period of time (over 10 s).

NOTE – For flaming of less than 10 s, the flaming is considered transitory or flashing.

3.10 fire effluent: The totality of gases and/or aerosols (including suspended particles) created by combustion or pyrolysis.

3.11 fire model: A laboratory process, including the apparatus, the environment and the test procedure, intended to represent a certain phase of a fire.

3.12 fire scenario: A detailed description of conditions, including environmental, of one or more stages from before ignition to completion of combustion in an actual fire at a specific location, or in a real-scale simulation.

4 Symbols specific to this test method

A_0	Initial electrical resistance of the corrosion target, Ω .
A_1	Electrical resistance of the corrosion target at the end of 1 hour exposure to combustion products, Ω .
A_{24}	Electrical resistance of the corrosion target at the end of 24 hours in the environmental chamber, Ω .
C	Corrosion of a target, nm.
C_1	Corrosion of a target at the end of 1 hour exposure to combustion products, nm.
C_{24}	Corrosion of a target at the end of 24 hours in the environmental chamber, nm.
m	Specimen mass, g.
m_i	Initial specimen mass, g.
m_f	Final specimen mass, g.
m_{70}	70% of the total mass loss, g.
t_d	Sampling time, s.
q_v	Volumetric sampling rate of combustion products, m^3/s .

5 Principle

5.1 This test method is used to determine the corrosive effect of combustion products from burning plastic materials. Corrosion is determined by the reduction in thickness of the metal on standardized targets, which is directly related to the increase in electrical resistance of the target due to the decrease in conductive cross-sectional area. These targets are not necessarily representative of the intended end use.

5.2 The test is conducted in two parts.

In the first part, two weight loss tests are conducted by exposing the test specimen to a recommended irradiance of 50 kW/m^2 , or to other heating fluxes up to 100 kW/m^2 relevant to particular scenarios. A spark igniter is used to ignite the combustible vapours. The combustible mass loss is determined as the average mass loss of the two tests.

In the second part, the specimen is subjected to the same irradiance as before. The products of decomposition or combustion are channeled through a funnel, and a portion of the products continuously flows through an exposure chamber, which holds the corrosion targets, until the specimen has lost 70 % of the loss measured in the two preliminary tests. The corrosion of the corrosion target is determined by exposure of the target to combustion products for 1 hour, followed by 24 hour exposure of the corrosion target to a controlled humidity and temperature environment in a separate chamber. The increase in electrical resistance of each target is monitored and the reduction in thickness of the metal on the target is calculated from the increase in electrical resistance. This reduction in thickness is referred to as corrosion by metal loss.

5.3 Additional guidance for testing is given in annex A and in ISO 11907-1.

6 Apparatus

6.1 Corrosion-testing device

6.1.1 General

The corrosion-testing device consists of the following main components: a cone-shaped radiant electric heater, a temperature controller, an exhaust system, a specimen holder and mounting, an ignition circuit, a load cell, a heat flux meter and a gas-sampling system as detailed below.

A general view of the corrosion-testing device is shown in figure 1 and typical dimensions are shown in figures 1 to 8.

The cone calorimeter test equipment described in ISO 5660-1 may be used as a corrosion-testing device, provided that it is equipped with a gas-sampling system as described in 6.1.9.

6.1.2 Conical heater

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The active element of the heater consists of an electrical heater rod, rated at 5 00 W/240 V, tightly wound into the shape of a truncated cone (see figure 2). The heater is encased on the outside with a double-walled stainless-steel cone, and packed with a refractory fibre material of approximately 100 kg/m³ density.

The heater is capable of producing an irradiance on the surface of the specimen of up to 100 kW/m² with a uniformity of $\pm 2\%$ within the central 50 mm \times 50 mm area of the specimen.

The irradiance from the heater is held at a preset level by means of a temperature controller and three type K stainless-steel-sheathed thermocouples having an outside diameter of 1,5 mm to 1,6 mm and with an unexposed hot junction. Alternatively, either 3 mm outside diameter sheathed thermocouples with an exposed hot junction or 1 mm outside diameter sheathed thermocouples with an unexposed hot junction are suitable. They are symmetrically disposed and in contact with, but not welded to, the heater element (see figure 2). The thermocouples are of equal length and wired in parallel to the temperature controller.

6.1.3 Temperature controller

The temperature controller, which shall be capable of holding the heating-element temperature steady to within $\pm 2\text{ }^{\circ}\text{C}$, has a temperature input range of 0 to 1 000 $^{\circ}\text{C}$, a set scale with a resolution of 2 $^{\circ}\text{C}$, and automatic cold junction compensation. The controller uses a zero crossing type thyristor unit and is equipped with a safety feature such that, in the event of an open circuit in the thermocouple line, it will cause the temperature to fall to near the bottom of its range.

NOTE – A suitable temperature controller system is a "3-term" controller (proportional, integral and derivative) with a thyristor unit capable of switching currents up to 25 A at 240 V.

6.1.4 Device for monitoring the heater temperature

This device shall have a resolution of 2 °C.

6.1.5 Exhaust system

The exhaust-gas system consists of a high-temperature centrifugal exhaust blower, a hood, intake and exhaust ducts for the fan, and an orifice plate flow meter (see figure 3).

The orifice plate flow meter consists of a sharp-edged orifice (57 mm inside diameter) located in the exhaust stack, at least 350 mm downstream from the fan.

In other details, the geometry of the exhaust system is not critical. Where necessary, small deviations from the recommended dimensions given in figure 3 are allowed. For example, it is permissible for the inner diameter of the duct and the orifice plate to be slightly different (tolerance: ± 2 mm). The fan shall be located 900 mm to 1 200 mm downstream of the hood in such a way that the flow of air to the measurement orifice is uniformly mixed.

6.1.6 Load cell

The load cell has an accuracy of 0,1 g, a measurement range of at least 500 g, and a mechanical tare adjustment range of 3,5 kg. The general arrangement of the load cell with the conical heater is shown in figure 4.

The load cell shall be connected to a data acquisition system capable of displaying the sample mass at intervals of 5 s or less.

6.1.7 Specimen holder assembly

The specimen holder assembly consists of a specimen holder, an edge frame and retaining pins as shown in figure 5. The specimen holder is constructed from 2 mm nominal stainless steel and has outside dimensions of 111 mm \times 111 mm \times 24 mm high (± 2 mm). It is lined with a layer of a low-density (nominal density 65 kg/m³) refractory ceramic fibre blanket with thickness of at least 13 mm.

The edge frame is constructed from 2 mm nominal stainless steel with outside dimensions of 116 mm \times 116 mm \times 56 mm high (± 2 mm). The frame has an 8 mm lip on the top to provide an opening of 100 mm \times 100 mm on the top. There are two 3 mm \pm 0,5 mm diameter \times 130 mm \pm 3 mm long retaining pins to lock the test specimen in the edge frame.

6.1.8 Ignition circuit

The external ignitor is a spark plug with a gap of 3 mm, powered from a 10 kV transformer. The transformer is of a type specifically designed for spark-ignition use and has an isolated (ungrounded) secondary to minimize interference with the data-transmission lines. The electrode length and location of the spark plug is such that the spark gap is located 13 mm above the centre of the specimen.

6.1.9 Gas-sampling system

This consists of the following components and is shown schematically in figure 1.

6.1.9.1 Gas-collecting device

The gas-collecting device (see figure 6) is a truncated cone constructed from stainless steel and having a larger diameter of (173 \pm 5) mm, a smaller diameter of (60 \pm 5) mm and a height of (97 \pm 5) mm.

6.1.9.2 Rigid tubing

The rigid tubing is a (675 ± 75) mm long stainless-steel tube with an outside diameter of 6,3 mm which draws a gas sample from the combustion stream. One end of the tube is bent with the open end of the tube facing upwards and away from the specimen surface to avoid soot deposition. This end of the tube is (255 ± 10) mm from the specimen surface. The other end is connected to flexible tubing. The arrangement of the rigid tube with the funnel is shown in figure 6.

6.1.9.3 Means of heating the rigid tubing

The means of heating (for example electric heating tape) shall be capable of maintaining the minimum temperature of the rigid tubing, and shall be capable of providing temperatures of up to 120 °C.

6.1.9.4 Flexible tubing

The flexible tubing consists of 6,3 mm outside diameter and (255 ± 10) mm long heat-resistant tubing. It is used to connect the rigid tubing to the exposure chamber.

6.1.10 Corrosion-resistant exposure chamber

The exposure chamber (see figure 7) consists of a $(0,011\ 2 \pm 0,000\ 5)$ m³ chamber containing a corrosion target support stand and smoke baffle. The chamber has an O-ring seal plus inlet and outlet ports.

The corrosion target support stand (see figure 8) is constructed from a corrosion-resistant material.

6.1.11 Pump

The pump shall be capable of extracting a gas sample at a rate of $7,5 \times 10^{-5}$ m³/s (4,5 l/min).

6.1.12 Filter

The filter is used to protect the pump from smoke particulates. Use the pump manufacturer's instructions to provide a suitable filter element.

6.2 Corrosion target and instrumentation

6.2.1 Corrosion target

The target shall be designed to be able to connect to a corrosion-measuring instrument and is composed of two circuit elements of identical material assembled on a non-reactive substrate. One circuit element is the active circuit element used for measuring corrosion and the other, with a protective coating, is used as a reference. Both of the elements of the target are exposed to combustion products during the test. A schematic diagram of a target from Rohrback Cosasco¹ (Model 030788-S0.35-8061) is depicted in figure 9.

6.2.2 Corrosion-measuring instrument

The corrosion-measuring instrument consists of a Kelvin bridge modified for measurement of the change in electrical resistance of corrosion targets and with a resolution, for measuring changes in thickness of the target, of not worse than 5 nm.

¹ Rohrback Cosasco systems, Inc., 11841 East Smith Avenue, Santa Fe, CA 90670, USA. Also Rohrco House, Worton Grange, Imperial Way, Reading, RG2 OTD, UK.

This information is given for the convenience of users of this part of ISO 11907 and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

6.2.3 Flow meter

A flow meter having a measurement range from 0 to 5 l/min, with resolution of 0,5 l/min and an accuracy of 2 % of full-scale reading, or an equivalent flow meter may be used, to monitor and maintain a constant flow throughout the sampling part of the test.

6.2.4 Environmental chamber

The chamber shall be capable of being maintained at $(23 \pm 2) ^\circ\text{C}$ and $(75 \pm 5) \% \text{RH}$.

7 Test specimens

There shall be five specimens measuring $100 \text{ mm} \times 100 \text{ mm} \times 6 \text{ mm}$ thick. Other thicknesses may be used if agreeable to all parties. However, the maximum thickness shall not exceed 12 mm. The specimens shall be cut from a representative sample of the material or end product. Clause 11 describes the preparation of the specimens for testing.

8 Conditioning

Unless otherwise agreed by the parties concerned, condition the specimens for at least 24 hours at $(23 \pm 2) ^\circ\text{C}$ and $(50 \pm 5) \% \text{RH}$.

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9 Calibration of apparatus

9.1 Heater calibration

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At the start of each test day or after changing to a new irradiance level, set the temperature controller at an irradiance of 50 kW/m^2 , or another irradiance agreed by the parties concerned, using the heat flux meter. Operate the cone heater for at least 10 min and ensure that the controller is within $\pm 5 \%$ of the desired setting.

During start-up of the heater or when changing the heat flux, place a shield on the load cell to avoid excessive heating of the cell. Remove the shield prior to inserting the heat flux meter in the calibration position.

9.2 Load cell calibration

Calibrate the load cell each day of testing using standard weights in the range of the test specimen mass.

9.3 Corrosion instrument calibration

Calibrate the corrosion instrument each test day in accordance with the manufacturer's instructions.

10 Equipment preparation

10.1 Carry out the preparation under laboratory conditions of $(23 \pm 2) ^\circ\text{C}$, and $(50 \pm 5) \% \text{RH}$.

10.2 Put an empty specimen holder (or other shield) in place during warm-up and in between tests to avoid excessive heat transmission to the load cell.

10.3 Turn on the power to the cone heater, the exhaust blower and the load cell.

NOTE – It is important not to turn off the power to the load cell on a daily basis.

10.4 Ensure that the exhaust flow rate is $(0,024 \pm 0,002) \text{ m}^3/\text{s}$.

10.5 Perform the calibration procedures specified in clause 9.

10.6 Ensure that the collection device, the exposure chamber, all sampling lines and the filter are clean and clear of any residual matter.

10.7 Check the sampling system for leaks. This is done with the pump operating and after the specified flow rate has been established. Shut off the flow through the flexible tubing immediately before the exposure chamber. The system is airtight if the flow meter flow drops to zero. If the flow does not drop to zero, then leaks are present within the system. Locate and correct such leaks.

11 Specimen preparation

11.1 For sheet or moulded specimens, cut the specimen to a size of 100 mm × 100 mm. Wrap the specimen in a single layer of aluminium foil. Place the edge frame over the specimen and cut the aluminium foil along the open edges at the top of the edge frame to expose the test specimen. If necessary, fill the edge frame below the specimen with refractory blanket to the level of the retaining pins. Lock the assembly with retaining pins and place assembly on the bottom specimen holder.

11.2 For tubular specimens, cut them to 100 mm lengths to fill the specimen holder. The number of lengths required equals 100 divided by the outside diameter (in mm). Seal the ends using an adhesive ceramic cement. Apply the adhesive cement to the ends such that there are no visible air holes in the coating and there is no cement overlapping on the ends. Wrap the tubular specimens in a single layer of aluminium foil. Place the edge frame over the tubular specimens and cut the aluminium foil along the open edges at the top of the edge frame to expose the specimens.

If necessary, fill the edge frame below the specimens with a refractory blanket to the level of the retaining pins. Lock the assembly with the retaining pins and place the assembly on the bottom specimen holder.

NOTE – Dimensionally unstable products, for example products that warp or shrink during testing, should be restrained against excessive movement.

12 Procedure

12.1 Measurement of initial mass

Measure the initial mass of each of the test specimens and calculate the average initial mass of the five specimens. The initial mass of each specimen shall be within 10 % of the average initial mass.

Record the initial mass of each test specimen.

12.2 Intumescent specimens

Specimens that intumesce or deform so that they contact the spark plug prior to ignition, or the underside of the cone heater after ignition, shall be tested with a separation of 60 mm between the baseplate of the cone heater and the upper surface of the specimen. In this case, the heater calibration (see 9.1) shall be performed with the heat flux meter positioned 60 mm below the cone heater baseplate. It is stressed that the time to ignition measured with this separation is not comparable to the one measured with the separation of 25 mm.

12.3 Mass loss determinations prior to conducting corrosion tests

12.3.1 Remove the empty specimen holder.

12.3.2 Start the determination by placing the specimen, held in the specimen holder (holder initially at room temperature), on the load cell in such a way that the distance between the bottom of the radiant heater and the top of the edge frame is adjusted to (25 ± 1) mm using the sliding height-adjustment device (see figure 2). Simultaneously start the ignition timer and the mass-loss and time-recording systems. Collect the data at intervals of 5 s or less. Move the spark plug into place over the test specimen, and turn on the spark power.

12.3.3. Record the times when flaming occurs for less than 10 s (flashing or transitory flaming) and the time when sustained flaming occurs.

12.3.4 Collect the mass loss data until one of the following occurs:

a) the average mass loss over a 1 minute period has dropped below 1,5 g;

or

b) 60 minutes have elapsed.

12.3.5 Remove the specimen holder.

12.3.6 Repeat the procedures specified in 12.3.2 to 12.3.5 for a second specimen.

12.3.7 From the mass loss data from the two determinations, determine the average 70 % of total mass loss for the corrosion test, as follows:

$$m_{70} = 0,5[0,7(m_i - m_f)_1 + 0,7(m_i - m_f)_2]$$

where subscript 1 denotes the first determination and subscript 2 denotes the second.

12.4 Corrosion gas sampling test

ISO 11907-4:1998

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12.4.1 Conduct three corrosion gas sampling tests after determining the average 70 % of total mass loss.

12.4.2 When ready to test, first remove the empty specimen holder.

12.4.3 Set the minimum temperature of the rigid stainless-steel tubing to 105 °C.

12.4.4 Record the initial resistance of the corrosion target, A_0 .

12.4.5 Place the corrosion target in the exposure chamber. Seal the exposure chamber, and check for leakage as described in 10.7.

12.4.6 Start the gas sampling for corrosion testing and ensure a continuous sampling rate of $(7,5 \times 10^{-5})$ m³/s (4,5 l/min).

12.4.7 Start the test by placing the test specimen, held in the specimen holder, on the load cell and start data collection. Collect the data at intervals of 5 s or less. Start the timer. Move the spark plug into place over the test specimen, and turn on the spark power.

12.4.8 Stop the sampling flow when the specimen has lost 70 % of its mass, as determined in 12.3.7, or when 60 minutes have elapsed after start of the test, and record the sampling time.

12.4.9 Combustion product exposure: Seal off the inlet and outlet sampling lines of the exposure chamber using clamps, and allow the combustion products to react with the corrosion targets for a total of 1 hour measured from the start of the test.