INTERNATIONAL STANDARD

ISO 11907-2

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

Part 2:
Static method

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

Partie 2: Méthode statique

Reference number
ISO 11907-2:1995(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 ISO 11907-2 was prepared by Technical Committee ISO/TC 61, *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 test*
- *Part 3: Dynamic decomposition method using a travelling furnace*
- *Part 4: Dynamic decomposition method using a conical radiant heater*

At the time of publication of this part of ISO 11907, the other parts were at various stages of preparation.

Annexes A and B of this part of ISO 11907 are for information only.

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

Part 2:
Static method

1 Scope

1.1 This part of ISO 11907 specifies a test method using a small-scale laboratory apparatus for the determination of the corrosive effects of combustion products liberated by burning plastics. Although the method is applicable to all combustible materials which might constitute a corrosion risk in the event of their burning, the size and nature of the test specimen is such that the test is not directly applicable to end-use assessment of corrosion hazard.

1.2 The test method makes it possible to produce a ranking of materials in the order of the corrosivity of their combustion products under specific test conditions with regard to combustion temperature, available oxygen, ventilation and different humidity levels.

NOTE 1 In order to assess the risk of corrosion when materials burn, the ranking of materials by corrosivity should be combined with their heat and flame response characteristics (e.g. ignitability, surface spread of flame, rate of heat release). The assessment of risk of corrosion in the event of fire requires consideration of many factors including fuel load, intensity of burning, ventilation conditions, humidity levels and the nature of the exposed surfaces.

Although the method described in this part of ISO 11907 refers only to copper corrosion, the test can be applied to other metallic and non-metallic materials in a variety of detector forms.

NOTE 2 Additives and fillers have been shown to influence the test results.

1.3 It is emphasized that this standard method of test is intended to be used to evaluate the relative degree of possible corrosive effects of fire gases to exposed materials and products under controlled laboratory conditions, and should not be considered, or used, for describing or appraising the corrosion risk of materials, products or systems under actual fire conditions.

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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO/IEC Guide 52:1990, *Glossary of fire terms and definitions.*

IEC 695-5-1:1993, *Fire hazard testing — Part 5: Assessment of potential corrosion damage by fire effluent — Section 1: General guidance.*

3 Definitions

For the purposes of this part of ISO 11907, the definitions given by ISO/IEC Guide 52 apply, together with the following definitions.

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

3.2 corrosion damage: The extent of the physical damage or impaired function caused by the chemical action of fire effluents.

3.3 fire effluent decay characteristics: The chemical and physical changes in fire effluents with time and during transport.

3.4 fire model: Means for the decomposition and/or combustion of test specimens under defined conditions to represent (a) known stage(s) of fire in order to generate fire effluents for corrosivity assessments. (This term is also used by the fire science community in the mathematical simulation of fire characteristics.)

3.5 corrosion detector: A standardized test piece made up to represent an electrical circuit or other material which can fail by corrosive effects.

4 Principles

4.1 One of the basic principles of the test is expressed by the sequence: combustion — condensation — corrosion, which is observed in real fires when humid effluents condense out on to cooler structures and components which may have escaped direct thermal damage. This may produce conditions conducive to corrosion. The test chamber for measuring the corrosiveness of effluents has been designed in order to reproduce the above conditions in a small-scale test.

The method primarily measures corrosion under conditions such that the sample is subject to condensation of water from the atmosphere and from the combustion products. It is also able to measure corrosion produced under conditions of low relative humidity, or where the detector temperature is above the dew point.

4.2 Under high relative humidity conditions, the combustion products are made to condense out on to a target substrate. This corrosion detector is a printed wiring board (PWB) (see 5.3), which is typically kept at constant temperature below the dew point of the effluents in the chamber.

4.3 In order to induce condensation, it is necessary to have a certain minimum relative humidity within the test chamber. Moreover, for a given atmosphere at a given temperature and relative humidity, condensation on a surface will occur only if its temperature lies at or below the dew point for that atmospheric condition.

NOTE 4 Moisture may be adsorbed on to the detector surface at temperatures above the dew point if hygroscopic chemical species have been deposited on to the surface of the corrosion detector.

4.4 If the desired temperature and relative humidity are known, it is possible to calculate the absolute humidity, i.e. the number of grams of water per cubic metre, necessary to produce condensation on the corrosion detector.

4.5 Constant temperature and relative humidity in the test chamber are established at the time of ignition (although the combustion of the specimen produces a temporary increase in the temperature of the test atmosphere). Once ignition has taken place, the repeatability of the test results depends on having a constant temperature of condensation at the detector. The detector temperature determines the residual relative humidity in the test chamber and also the reliability of the measurements of the electrical resistance of the detector, since this varies with temperature.

5 Apparatus

5.1 Test chamber

The test chamber shall consist of a cylindrical sealed enclosure with a total volume of approximately 20 litres and a diameter of approximately 300 mm (a typical test apparatus is described in annex A). The chamber shall be maintained at a constant temperature, e.g. by heating elements around its external surface.

The test chamber doors shall be designed to provide a gas-tight seal adequate for the purpose of maintaining a constant relative humidity within the test chamber for the duration of the test.

The materials of construction of the test chamber shall have sufficiently low permeability to water vapour to permit the maintenance of a constant relative humidity of (65 ± 2) % at the test temperature of 50 °C ± 2 °C for the duration of the test.

All materials within the chamber, with the exception of the corrosion target, shall be chemically inert to the combustion products or shall be protected from contact with the combustion products by means of a surface coating or other device.

The interior surfaces of the chamber are thermostatted uniformly at 50 °C ± 2 °C to avoid formation of cold spots.

5.2 Ignition source

5.2.1 The ignition source consists of an inert crucible containing a resistance wire heater: a typical arrangement is shown in figure A.4.

5.2.2 A dispensing device shall be provided, capable of placing the specimen in the crucible after the required ignition temperature has been reached.

5.3 Corrosion detector

The corrosion detector shall be a resistance etched on a copper-plated laminated base. The detector shall be constructed of 36 conductor tracks each 52 mm ±

1 mm long, 0,3 mm ± 0,02 mm wide and 17 µm ± 1 µm thick, on a laminated epoxy-resin base-plate (see figure A.5).

Dimensions: PWB (40 ± 1) mm × (80 ± 1) mm
Base-plate (70 ± 2) mm × (140 ± 2) mm
Resistance: 8,0 Ω ± 0,5 Ω

Condensed products react with the copper if they are corrosive and the corrosivity shall be assessed by measurement of the resistance variation due to attack on the copper circuit.

6 Test specimens

A minimum sample quantity of 3 g shall be provided to allow five tests to be carried out on individual specimens of 600 mg ± 2 mg of material. The specimens shall be in the form of granules or chips to ensure intimate contact with the ignition source.

Prior to use, new equipment shall be calibrated for relative humidity and temperature within the exposure chamber, and for the temperature of the ignition source, in accordance with the procedures recommended by the equipment manufacturer. These calibrations shall be carried out for maintenance purposes after every 50 runs at least.

7 Procedure

7.1 Condensing mode

7.1.1 Place a test specimen in the dispensing device (5.2.2).

7.1.2 Prepare the PWB by cleaning with a pumice-based acidic powder formulation. Rub the PWB with a piece of moist cotton wool containing the cleaning powder. Thoroughly wash the PWB with excess deionized water and carefully wipe it dry.

NOTE 5 By this method, copper surfaces are cleaned chemically as well as physically. The acidic chemical cleaning action removes oxides and wets the PWB surface. No residual film remains after rinsing and drying. Circuits which have been subjected to this treatment show no sign of corrosion in simple condensation tests.

7.1.3 Ensure that the internal surface of the test chamber (5.1) as well as other parts which might have been in contact with combustion products have been suitably cleaned as described in clause 8.

7.1.4 Connect up the PWB corrosion detector (5.3) and position it on the holder (see figure A.5).

7.1.5 Adjust the temperature in the chamber to 50 °C ± 2 °C and the relative humidity to (65 ± 2) % (see A.5).

7.1.6 Circulate water to the corrosion detector holder to maintain it at 40 °C ± 2 °C and measure the initial resistance R_i to ± 0,02 Ω.

7.1.7 Heat the ignition source to 800 °C ± 5 °C.

7.1.8 Place the specimen in the crucible and maintain at 800 °C ± 5 °C for 3 min.

7.1.9 60 min after the specimen was placed in the crucible, measure the final resistance R_f to ± 0,02 Ω.

In order to avoid electrochemical corrosion of the PWB, the resistance shall not be measured continuously over the 60 min interval.

7.1.10 If required, remove the corrosion detector from the test chamber and post-condition for a specified time (see clause 10).

7.2 Non-condensing mode

7.2.1 Place a test specimen in the dispensing device (5.2.2).

7.2.2 Prepare the PWB as described in 7.1.2.

7.2.3 Ensure that the internal surface of the test chamber (5.1) as well as other parts which might have been in contact with combustion products have been suitably cleaned as described in clause 8.

7.2.4 Connect up the PWB corrosion detector (5.3) and position it on the holder (see figure A.5).

7.2.5 Adjust the temperature in the chamber to 50 °C ± 2 °C and the relative humidity to (65 ± 2) %.

7.2.6 Measure the initial resistance R_i of the corrosion detector to ± 0,02 Ω.

7.2.7 Heat the ignition source to 800 °C ± 5 °C.

7.2.8 Place the specimen in the crucible and maintain at 800 °C ± 5 °C for 3 min.

7.2.9 60 min after the specimen was placed in the crucible, measure the final resistance R_f to ± 0,2 Ω.

In order to avoid electrochemical corrosion of the PWB, the resistance shall not be measured continuously over the 60 min interval.

7.2.10 If required, remove the corrosion detector from the test chamber and post-condition for a specified time (see clause 10).

8 Cleaning procedure

Evacuate the combustion products from the chamber by purging with air before opening the chamber doors. Carry out this operation in a fume cupboard or using a filter at the chamber exit in order to remove combustion products.

Open the test chamber, taking the necessary precautions to avoid operator exposure to combustion products. Clean the soiled surface using the following procedure:

- — Use suitable gloves.
- — Remove the corrosion detector from its base-plate.
- — Wipe all exposed surfaces with a suitable solvent.
- — Remove the ignition source and clean it with a suitable solvent. If necessary, use a non-metallic brush to remove adhering residues.
- — Remove the heating element from the crucible and clean it with a suitable solvent. If necessary, use a non-metallic brush to remove adhering residues. Reassemble the heating element, making sure that the thermal insulation is unimpaired at the top of the crucible by using mineral wool. Seal off the two little orifices at the bottom of the crucible with mineral wool to avoid material flowing out when melted.
- — Clean the dispensing-device components and apply silicone lubricant to the slide joint to improve movement and the seal.

9 Expression of results

Express the corrosivity R_{COR} as the percentage change in the PWB resistance relative to the original value of the resistance, using the following equation:

$$R_{\mathrm{COR}} = 100 \left(\frac{R_{\mathrm{i}} R_{\mathrm{f}}}{8 \left(R_{\mathrm{f}} - R_{\mathrm{i}} \right)} - 1 \right)^{-1}$$

where

R_{i} is the initial resistance at 40 °C ± 2 °C (condensing mode) or 50 °C ± 2 °C (non-condensing mode);

R_{f} is the final resistance at 40 °C ± 2 °C (condensing mode) or 50 °C ± 2 °C (non-condensing mode).

NOTE 6 The results are expressed in this way in order to normalize the initial resistance of the standard corrosion detector to 8 Ω. This accommodates variations in the resistance of the printed wiring board inherent in the manufacturing process.

10 Post-exposure of test specimen (optional)

At the end of the 1 h period following combustion of the specimen and the simultaneous condensation of the combustion products on to the PWB detector, remove the PWB detector from the chamber after recording the final resistance value R_{f}.

Store the PWB detector at ambient temperature and 75 % R.H. in an enclosed chamber containing a saturated aqueous sodium chloride solution in an open basin. At intervals of 24 h after removal from the test chamber, measure the resistance of the PWB detector as R_x, where x equates to the number of hours after removal from the test chamber.

11 Precision

Only preliminary precision data are available at present. Results and conclusions from an interlaboratory trial of limited scope are given in annex B, which is informative.

12 Test report

The test report shall include the following information:

a) a reference to this part of ISO 11907;

b) all details necessary for full identification of the product tested, including the manufacturer's name, number or code;

c) the name and location of the testing laboratory;

d) the test values for each specimen, including:

  1) a number identifying the test specimen;

  2) the physical form of the specimen;

  3) the combustion temperature;

  4) the temperature of the test chamber and the PWB detector;

  5) the relative humidity in the test chamber;

  6) the increase in resistance of the PWB detector, $R_{\mathrm{f}} - R_{\mathrm{i}}$;

  7) the corrosivity, expressed as the percentage change in resistance, R_{COR};

  8) if required, the increase in resistance of the PWB detector after post-exposure, $R_x - R_{\mathrm{i}}$;

e) the date of the test and, if applicable, the date of completion of post-exposure (see clause 10).

Annex A
(informative)

Typical test apparatus

A.1 General

A typical test apparatus consists of an external hexagonal housing comprising two hinged sections of total overall dimensions 1 600 mm × 700 mm × 700 mm. Within the housing are two separate compartments as follows:

a) a forward, thermally insulated compartment comprising the temperature-controlled enclosure for the test chamber;

b) a rear compartment containing the equipment necessary for control of humidification and effluent condensation.

The purpose of the test chamber enclosure is to stabilize the temperature and humidity regime within the chamber so as to ensure good control of the combustion-product condensation process, and to eliminate thermal gradients and cold spots on the chamber walls. To accomplish this, three heating elements are arranged at 120° intervals around the external surface. The temperature control of the climatically controlled enclosure is further ensured by preheating the humid air in circulation. Temperature regulation of the enclosure is carried out using a probe placed in the air flow at fan level.

A.2 Test chamber

The test chamber consists of a 300 mm ± 6 mm diameter stainless-steel container of 20 litres ± 0,4 litres volume, closed with a PMMA door, where the interior metallic surface is protected from corrosion by a laminated film of PTFE of 80 µm thickness. Vapour-tight penetration devices make it possible to insert the combustion, condensation and temperature control equipment in the chamber.

Control of relative humidity and temperature (up to 70 °C ± 0,1 °C) within the test chamber is automatic. The start-up and the duration of the humidification phase are completely programmable by microprocessor.

The humid atmosphere is obtained by sweeping the test chamber with air previously saturated with water by bubbling through a thermostat bath at the required temperature. Two electrovalves make it possible to set up and to cut off circulation of the humid air while ensuring the vapour-tightness of the test chamber.

The increase and control of the temperature of the test chamber are obtained through a set of three electrical resistances which are fixed around and close to the outside surface of the chamber. The temperature control probe is situated within the test chamber, which is placed within a ventilated oven where circulation of warm air at constant temperature is maintained.

The temperature of the test chamber can be controlled by means of a thermocouple in the centre of a test chamber and by regulating the potentiometer after the temperature reading has stabilized. It is simpler to move up rather than down the temperature scale, as the system must be allowed to reach equilibrium after each change in setting.

The temperature-conditioning of the chamber can be checked by following the change in resistance of the PWB detector prior to ignition, as its resistance is a function of the ambient temperature.

A.3 Ignition source

Combustion of the sample is carried out by means of an ignition source 55 mm in height and 63 mm in diameter consisting of a heating element placed within an insulated crucible, typically made of aluminium alloy, and wrapped in ceramic wool. The heating element consists of Ni-Cr resistance wire in an Inconel jacket formed into connecting spirals using a laser soldering device. The temperature is typically controlled using a thermocouple, also in an Inconel jacket, soldered to the external surface. The insulated crucible minimizes heat transfer with the test chamber atmosphere.

The test specimen is placed in the crucible by means of a dispensing device which is activated when the crucible temperature has reached the temperature chosen for combustion.

In order to measure the temperature of the heating element, a thermocouple covering a range from 200 °C ± 20 °C to 1 000 °C ± 20 °C is required;

stainless-steel-jacketed thermocouples are suitable for this purpose. To ensure good contact between the thermocouple tip and the heating-element surface, the thermocouple is formed into a loop which is then inserted in between the spirals of the heating element.

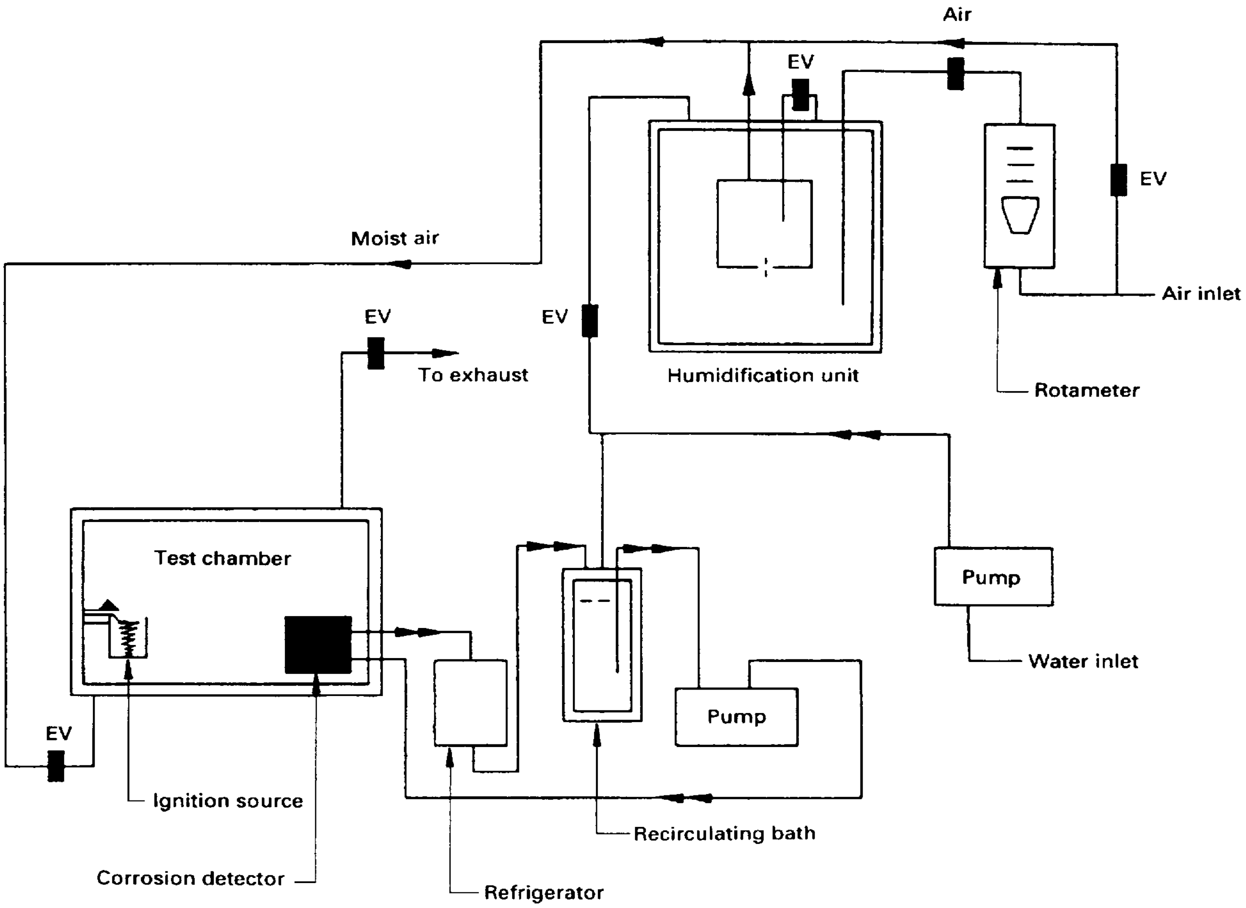
A.4 Condensation device

The combustion products are condensed out on to the PWB corrosion detector which is cooled by circulation of water provided by a thermostat bath maintained at a temperature equal to or below the dew point of the test atmosphere. The water is in contact with the interior surface of the corrosion detector, and the tightness of the system is guaranteed by means of elastomeric seals. As the thickness of the glass-filled epoxy base-plate is 0,8 mm, the active detector surface can be kept at a constant temperature whatever variations occur in the temperature of the test chamber.

The combustion products are condensed out on to the corrosion detector by cooling the copper holder using circulating cold water. This is the only surface inside the test chamber on which condensation occurs. The cooling water is provided by a magnetically stirred, thermostatted recirculating bath of 2 litres capacity (stainless steel, cylindrical of 110 mm diameter, temperature range up to 70 °C with control to ± 0,2 °C, with provision for automatic level control and manual draining).

A.5 Humidification unit

Reproducibility of the results depends on adequate humidity control within the test chamber. This is obtained by passing air at a controlled flow rate through a thermostatically controlled water tank (150 mm diameter, 3 litres capacity, stainless steel with magnetic stirring, temperature range up to 70 °C with control to ± 0,2 °C). The humidified air stream is then freed from suspended droplets in a separator unit before being reheated within the climatically controlled enclosure and circulated into the test chamber. The temperature of the humidification bath determines the absolute humidity of the air in the test chamber prior to combustion of the sample. The unit, which has an automatic level control, can also be drained manually.



Key:

EV Electrovalve

—◄— Moist air

—◄◄— Cooling water

Figure A.1 — General layout of test apparatus

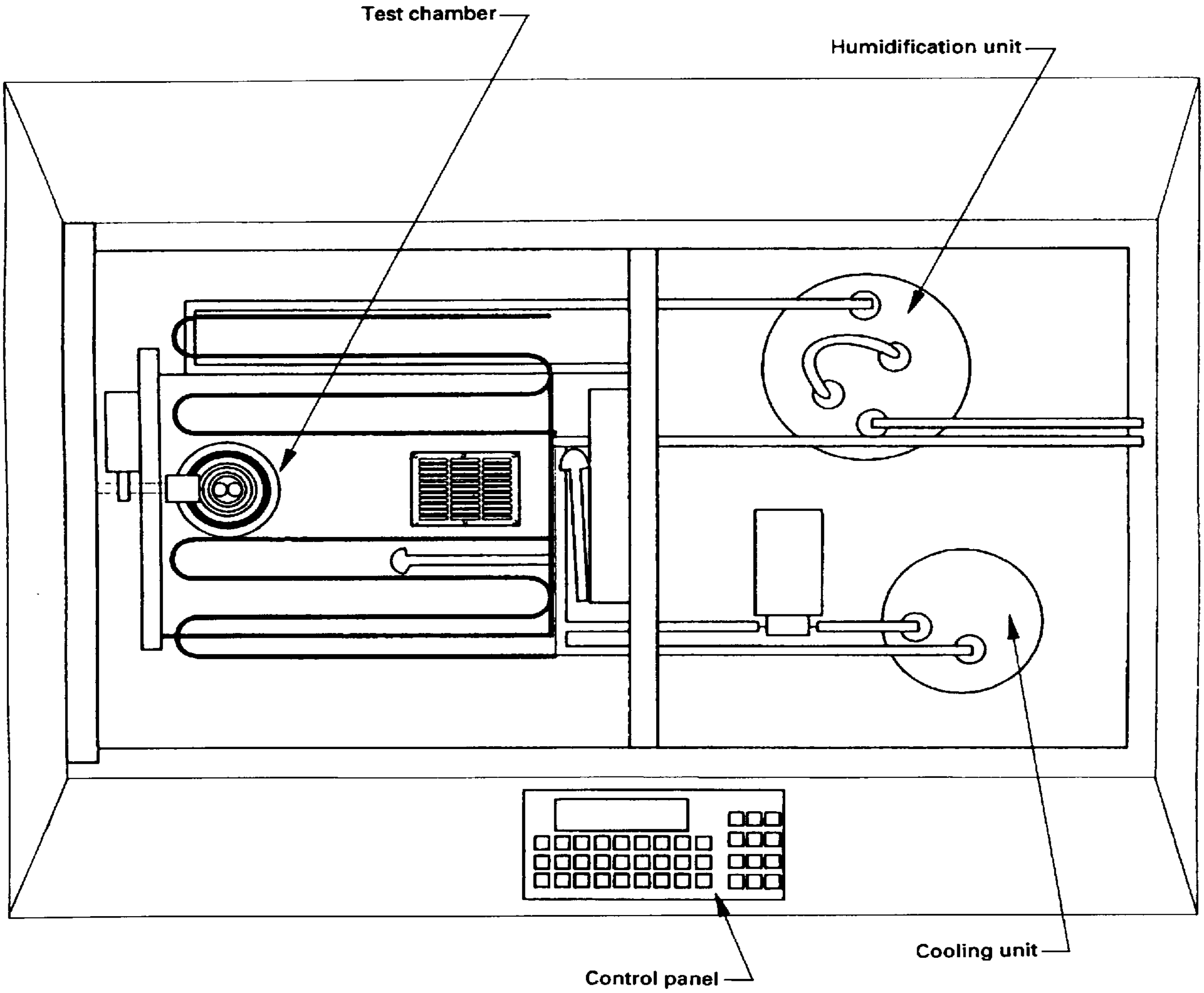


Figure A.2 — Plan view of typical test apparatus

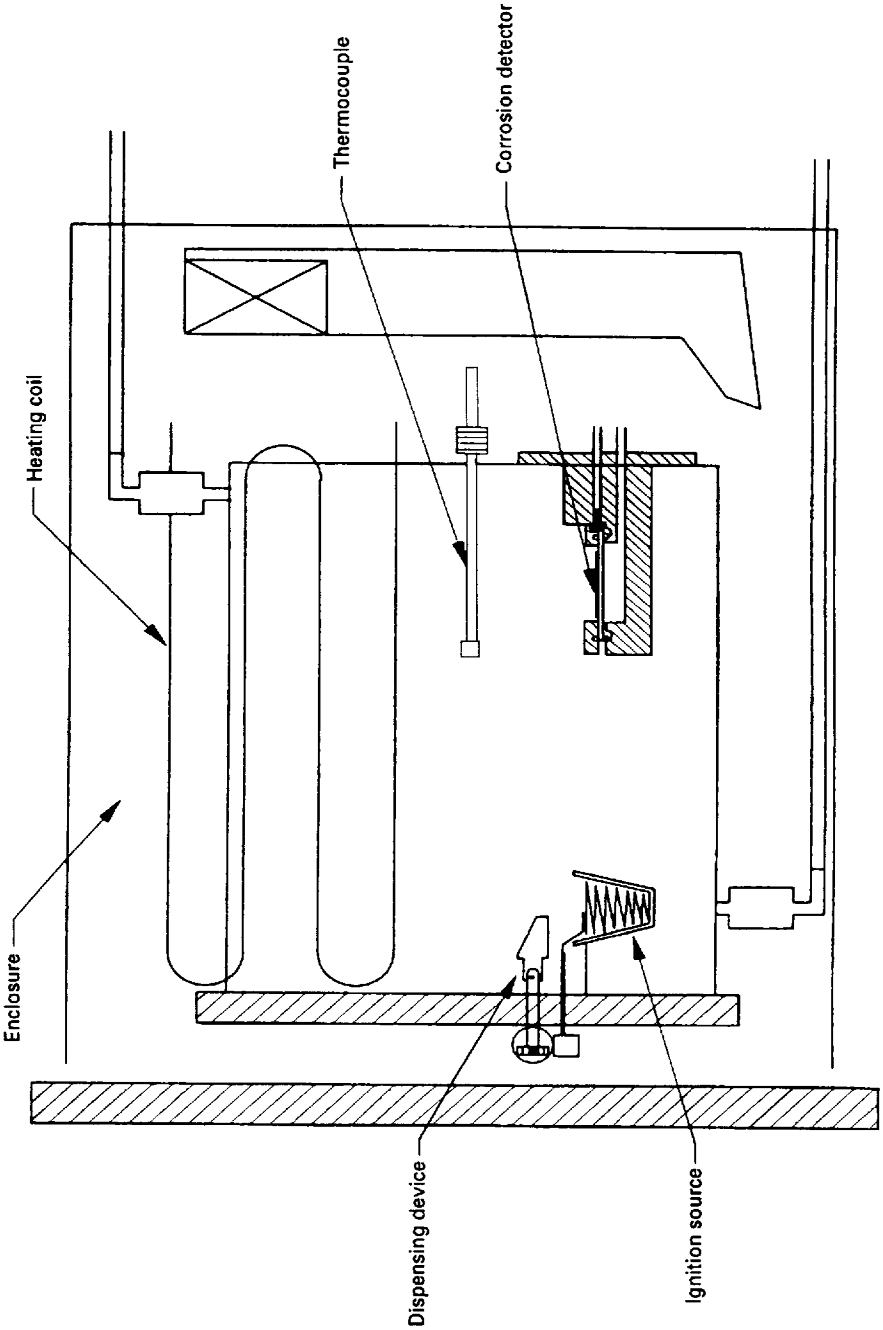


Figure A.3 — Typical test chamber arrangement

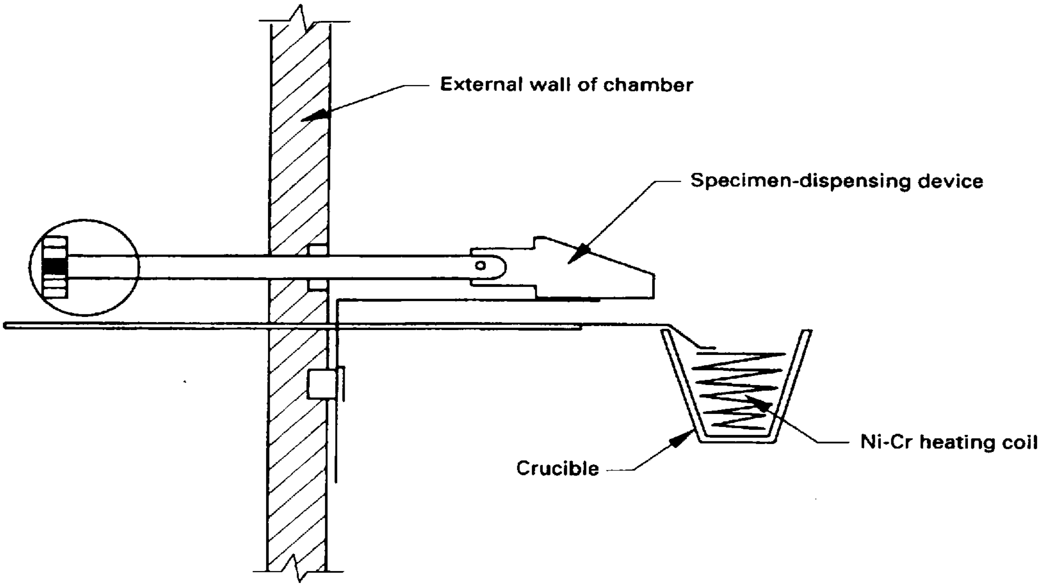


Figure A.4 — Typical ignition source

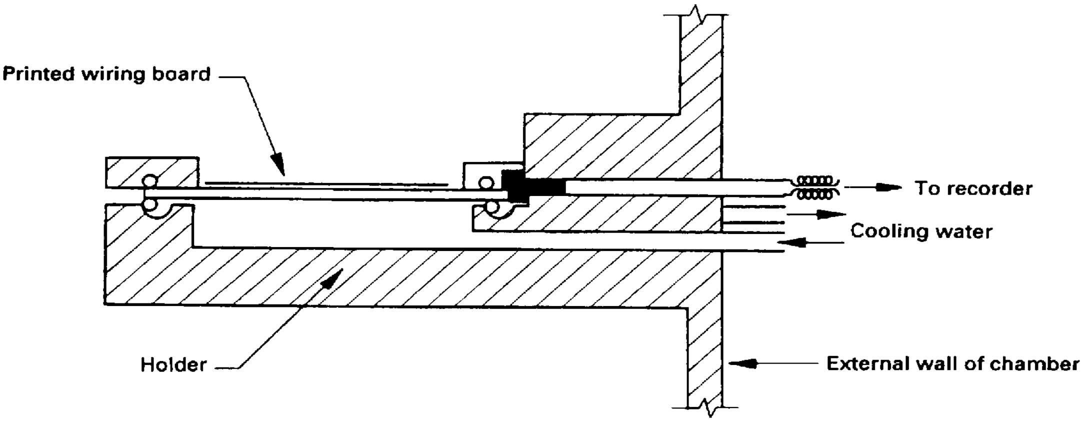


Figure A.5 — Typical PWB corrosion detector holder with cooling system attached

Annex B
(informative)

Variability in the corrosivity values (R_{COR}) measured by the static procedure

During 1991, a limited interlaboratory test programme was conducted for a preliminary assessment of the precision of the test method.

The design of the experiment was a fully crossed arrangement of four laboratories and five materials, with the materials acting as separate levels. No randomization scheme was used for the distribution of the materials to the laboratories, or for the order of testing within each laboratory. The corrosion targets were supplied to each laboratory from a single source of supply.

Four laboratories in four different countries burnt five different polymer compounds under controlled conditions in the apparatus specified in clause 5. Sufficient material was provided to do five replicate tests on each material. A written test protocol was provided which specified the combustion and condensation conditions for the fire effluent in the condensing mode (see 7.1). The effects of the different effluents generated by burning the different materials were determined by recording changes in the electrical resistance of the PWB detector.

The precision calculations based on the results of this exercise are given in table B.1. These figures, which were derived using ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* [now withdrawn[1]], have to be considered preliminary and indicative for two major reasons. The number of participating laboratories was below the ISO 5725 recommendation of 8. Only two laboratories were able to comply fully with the test protocol; the other two laboratories had small deviations from the specified humidity conditions in the chamber.

The definitions of repeatability and reproducibility given in ISO 5725:1986 were as follows:

— **repeatability,** r: The value below which the absolute difference between two single test results obtained with the same method on identical test material under the same conditions (same operator, same apparatus, same laboratory and a short interval of time) may be expected to lie with a specified probability; in the absence of other indications, the probability is 95 %.

— **reproducibility,** R: The value below which the absolute difference between two single test results obtained with the same method on identical test material under different conditions (different operators, different apparatus, different laboratories and/or different times) may be expected to lie with a specified probability; in the absence of other indications the probability is 95 %.

Table B.1 — Repeatability and reproducibility of corrosivity values (R_{COR})

Material	Mean R_{COR} value	Repeatability		Reproducibility	
		r	% of mean	R	% of mean
A	0,74	1,63	220	2,21	299
B	5,31	5,80	109	6,58	124
C	2,73	3,36	123	8,96	328
D	7,05	6,42	91	8,47	120
E	43,84	26,98	61	54,16	124

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[1] Replaced by ISO 5725, parts 1 to 4, published in 1994.

The interlaboratory trial highlighted certain aspects of the method which required improvement, and a number of these have already been incorporated into the apparatus and procedure described in this part of ISO 11907. A further interlaboratory trial is planned in order to determine the precision of the method with these improvements incorporated.

ICS 13.220.40; 83.080

Descriptors: plastics, combustion products, smoke generation, gaseous effluents, smoke, corrosion, tests, corrosion tests, static tests.

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