INTERNATIONAL STANDARD

ISO 19095-6

First edition
2021-06

Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies —

Part 6: Accelerated degradation test

Plastiques — Évaluation des performances de l'interface d'adhérence dans les assemblages plastique-métal —

Partie 6: Essai de dégradation accéléré

Reference number
ISO 19095-6:2021(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

A list of all parts in the ISO 19095 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Structures of heterogeneous materials are being manufactured in the automotive and aerospace industry sectors, where higher safety margins are required. The existing test methods are not appropriate because the evaluation of the adhesive interface is difficult, as the polymer material has a relatively low mechanical strength and therefore fractures outside the joints. Therefore, it is necessary to develop a methodology for the evaluation of the adhesive interfaces. A test method to accurately evaluate the adhesion interface performance or standardization of long-term evaluation under harsh environments is also necessary. The method in ISO 19095 is intended to ensure that the integrity of the joint is realized through the interface and that traceability of the value improves the data comparison. This document defines the conditions to evaluate the long-term durability which cannot be evaluated using ISO 19095-4.

Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies —

Part 6: Accelerated degradation test

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices. It is recognized that some of the materials permitted in this document might have a negative environmental impact. As technological advances lead to more acceptable alternatives for such materials, they will be eliminated to the greatest extent possible. At the end of the test, care should be taken to dispose of all waste in an appropriate manner.

1 Scope

This document specifies the environmental conditions to evaluate the accelerated degradation for the adhesion interface performance in plastic-metal assemblies.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

To evaluate the degradation of plastic-metal assemblies under severe conditions in a shorter period of time, the assemblies are subjected to a higher temperature and a higher humidity conditions such as 120 °C and 0,2 MPa of saturated water vapour pressure without air (oxygen) gas (see Annex B).

This method should be used at temperature at least 20 °C lower than softening temperature in order to avoid deformation (see Annex C).

5 Apparatus

5.1 Oven, capable of heating to 200 °C for test samples or sealed pressure vessels connected to pressure gauge and purge lines settled outside oven, with temperature control system (±1 °C).

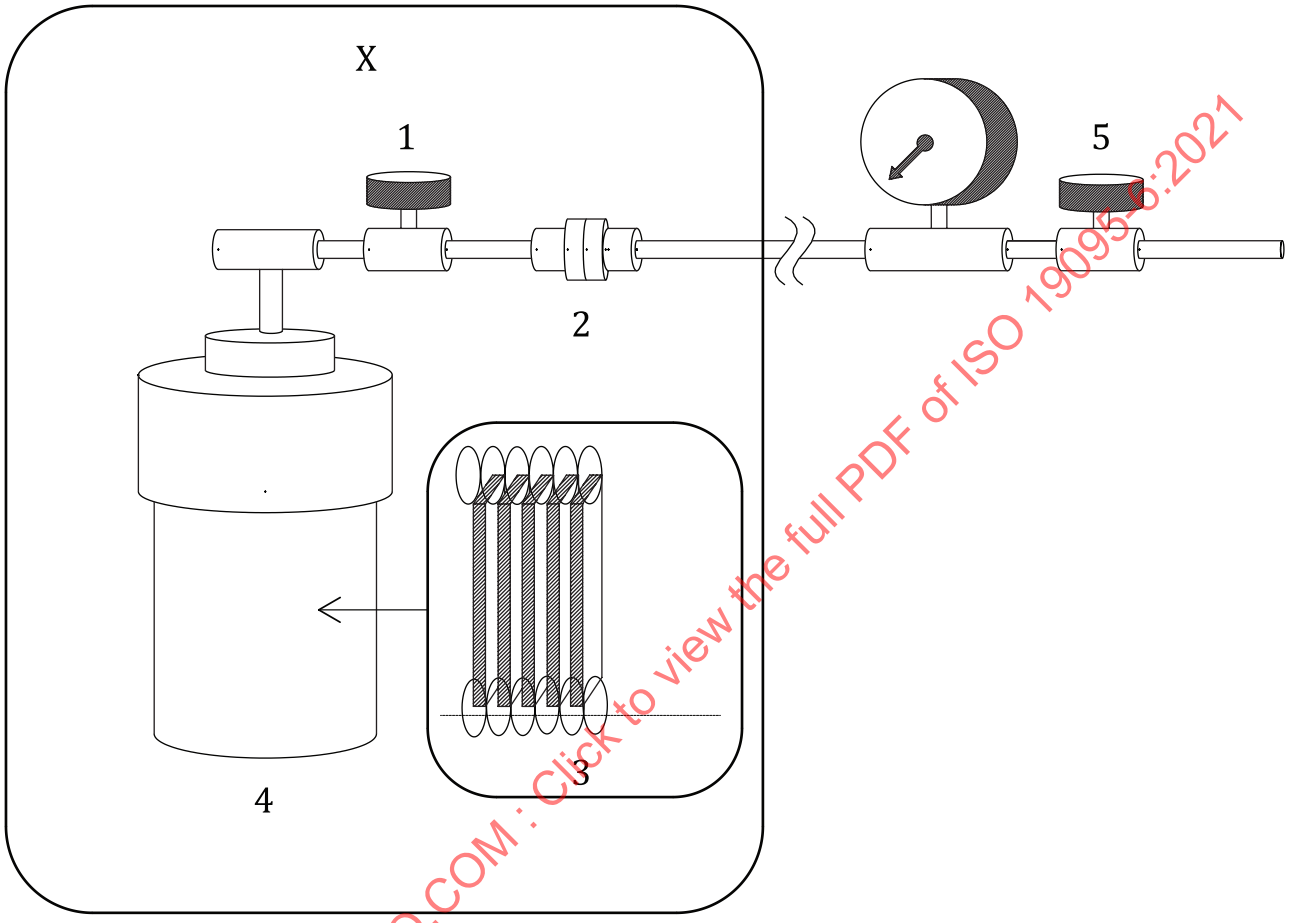
To control the temperature uniformly inside the oven, an air circulation heating system should be used.

If conditions of 120 °C and 0,2 MPa of saturated water vapor pressure is used, a sterilizer (see 5.5) can be used instead of oven and sealed pressure vessels.

5.2 Sealed pressure vessel, capable of high inside pressures (up to 2,0 MPa) and connected to pressure gauge and purge line as indicated in Figure 1.

The size of the vessel is dependent on the size and number of plastic-metal assemblies. For 10 samples with 10,0 mm × 100,0 mm × 2,0 mm, a 300 ml stainless steel vessel (autoclave) should be used.

An inner setting part should be used to avoid that sample surfaces do not touch each other and inside liquid water.



Key

X oven
1 valve A
2 connector
3 water
4 sealed pressure vessel
5 valve B

Figure 1 — Severe condition obtaining system for plastic-metal assemblies

5.3 Pressure gauge (e.g. 0 MPa to 1,0 MPa), to indicate the pressure in the vessel with resolution of 0,05 MPa.

5.4 Relief valve or bursting disc, operating at 3,5 MPa and installed in the line.

5.5 Sterilizer, capable of heating to 105 °C to 135 °C with saturated water vapour pressure.

Air releasing system shall be installed at 100 °C during heating from room temperature to 120 °C.

5.6 Vacuum oven, capable of heating to 200 °C under reduced pressure with temperature controlling (±1 °C).

5.7 Balance, capable of weighing the sample with a resolution of at least 0,1 mg.

6 Procedure

6.1 Test specimens of specimens plastic-metal assemblies

Test specimens of plastic-metal assemblies should be prepared according to ISO 19095-2.

6.2 Conditioning of specimens plastic-metal assemblies

Conditioning of plastic-metal assemblies before and after degradation by this document should be performed according to ISO 19095-1.

6.3 Weighing plastic-metal assemblies

Weigh the plastic-metal assemblies with a balance.

6.4 Plastic-metal assemblies subjected to severe conditions

Set the plastic-metal assemblies inside the sealed pressure vessel in a way to avoid sample's surfaces touching each other or the liquid water.

Pour deionized water (10 ml to 20 ml in the case of 300 ml vessel) into vessel.

Seal the pressure vessel and settle inside the oven as indicated in Figure 1. Open valves A and B.

Set the oven temperature above 100 °C, for example 120 °C. Heating is started. At around 100 °C, release steam (water vapour) at valve B for a short time (1 min to 2 min) to release inside air. Then close valve B. Inside water vapour pressure is confirmed by a pressure gauge at determined temperature. Continue heating throughout the determining period, such as 24 h. Examples of the conditions of temperature and test period are shown in Annex A. After leaving a test specimen in the pressure vessel with selected conditions, cool the test specimen in the pressure vessel and take it out from the pressure vessel, then leave it for more than 2 h under the condition described in ISO 19095-1 and carry out the test.

6.5 Evaluation of plastic-metal assemblies

Evaluations of plastic-metal assemblies after or before severe conditions environment by this document should be performed according to ISO 19095-3.

7 Test reports

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 19095-6:2021;

b) all information necessary for complete identification of the test specimen;

c) conditions of heating temperature and heating period;

d) operation date of this degradation procedure.

Annex A
(informative)

Example of severe condition tests of plastic-metal assemblies

A.1 Heating temperature

The vapour pressure of water is determined as a function of temperature. Table A.1 shows the vapour pressure at temperatures above 100 °C.

Table A.1 — Vapour pressure of water at high temperature than 100 °C

Temperature °C	Vapour pressure KPa
105	120,80
110	143,27
120	198,54
130	270,13
135	313,07
140	361,38
150	476,00
160	618,06

A.2 Test period

Examples of periods for degradation are shown in Table A.2.

Table A.2 — Examples of test period for degradation

Temperature °C	Test period h
120	96, 192, 288
135	192, 384
160	336

Annex B
(informative)

Example of tests of plastic-metal assemblies in the pressure vessel

B.1 Tensile tests of plastic-metal assemblies in the pressure vessel

Test samples as examples of plastic-metal assemblies were made of aluminium plate and polyphenylenesulfide (PPS). These specimens were degraded with heating and moisture in the pressure vessel. After degradation in the pressure vessel, these specimens were measured by tensile test. The strength of the specimen is determined as the maximum stress on the tensile test stress-strain curve. The results of tensile tests of specimens degraded in a sterilizer are shown in Table B.1. The averaged values of five specimens for each condition are shown in the table.

Table B.1 — Results of tensile tests of specimens degraded in a sterilizer

Temperature °C	Test period hour	Tensile strength MPa
Room temperature	0	45,2
120	192	37,42
135	192	32,19

The results of tensile tests of specimens degraded in a stainless steel vessel are shown in Table B.2. The averaged values of three specimens for each condition are shown in the table.

Table B.2 — Results of tensile tests of specimens degraded in a stainless steel pressure vessel

Temperature °C	Test period hour	Tensile strength MPa
Room temperature	0	34,4
160	336	28,3

Annex C
(informative)

Example of effect of heating on ordinary plastics

C.1 Example of ordinary plastics

Five kinds of plastics, ploymethyl metacrylate (PMMA), polyethylene terephthalate (PET), polycarbonate (PC), polypropylene (PP), and polyethylene (PE), were employed as examples of ordinary plastics.

C.2 Deformation ordinary plastics by heating

Deformations of five kinds of plastics after heating at 100 °C, 120 °C, 140 °C, and 160 °C for 1 h in an electric oven were observed. Photographs of deformations of ordinary plastics are shown in Figure C.1 to Figure C.5. These results, softening temperatures and melting temperatures are shown in Table C.1. In the table, the symbol ○ shows no deformation, the symbol Δ shows a small deformation, the symbol × shows clear deformation. Deformations are observed the specimens heating at temperature 20 °C greater than the softening temperature.

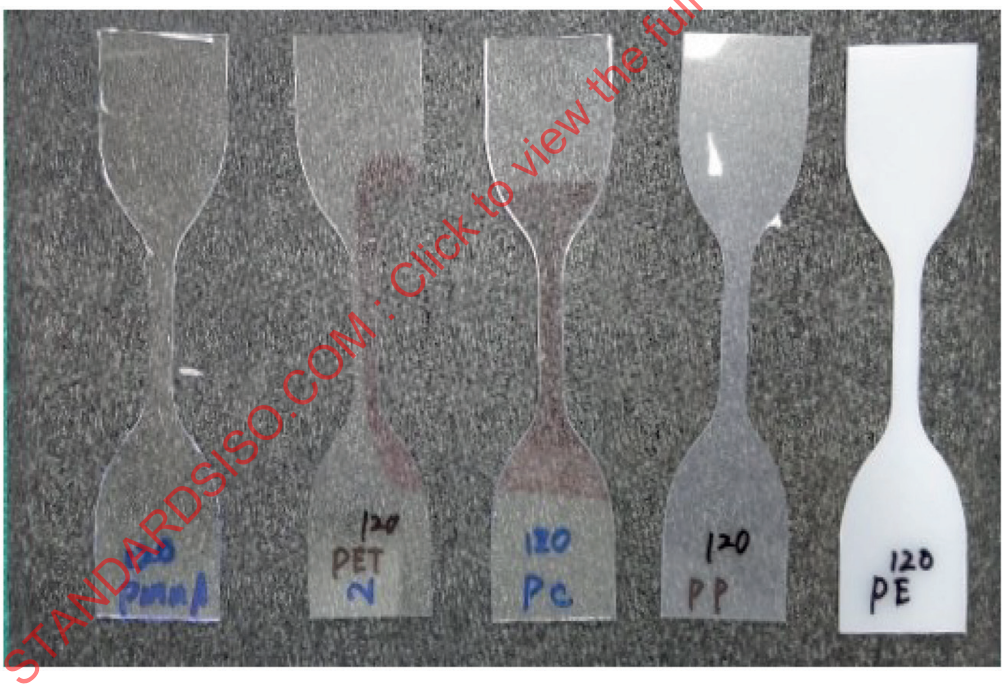


Figure C.1 — Dumbbell shape specimens of PMMA, PET, PC, PP and PE

Figure C.2 — Deformations of PMMA, PET, PC, PP and PE heating at 100 °C for 1 h

Figure C.3 — Deformations of PMMA, PET, PC, PP and PE heating at 120 °C for 1 h

Figure C.4 — Deformations of PMMA, PET, PC, PP and PE heating at 140 °C for 1 h

Figure C.5 — Deformations of PMMA, PET, PC, PP and PE heating at 140 °C for 1 h