
**Adhesives — Determination of the
solubility of water-soluble or alkali-
soluble pressure-sensitive adhesives**

*Adhésifs — Détermination de la solubilité des adhésifs sensibles à la
pression, solubles dans l'eau ou dans les alcalis*

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 25179 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

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Adhesives — Determination of the solubility of water-soluble or alkali-soluble pressure-sensitive adhesives

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It is recognized that some of the materials permitted in this document may 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 in accordance with local regulations.

1 Scope

This International Standard specifies a test method for the determination of the solubility of a water-soluble pressure-sensitive adhesive in water or the solubility of an alkali-soluble pressure-sensitive adhesive in alkali by measuring the loss in mass of the adhesive when immersed in water or alkali. The solubility of the adhesive is one of the key factors in assessing the possibility of recycling products to which labels are fixed using a pressure-sensitive adhesive.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

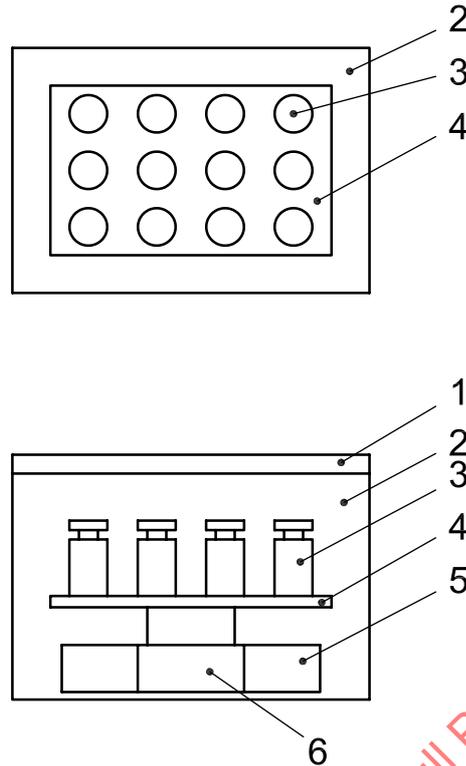
ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

3 Principle

This method measures the solubility of a water-soluble or an alkali-soluble adhesive, coated on kraft paper, as a function of time by measuring the loss in mass of an adhesive-coated test specimen after shaking the specimen in water or alkali solution.

4 Apparatus

4.1 Orbital shaker, enclosed in a chamber capable of being heated by air at a temperature in the range 40 °C to 80 °C, accurate to within ± 2 °C, and having a motor capable of producing a shaking speed of 10 r/min to 100 r/min, for shaking the bottles (4.2) containing the test specimens (see Figure 1).



Key

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| 1 shaker chamber cover | 3 bottle | 5 heater |
| 2 shaker chamber | 4 shaker plate | 6 motor |

Figure 1 — Example of a shaker

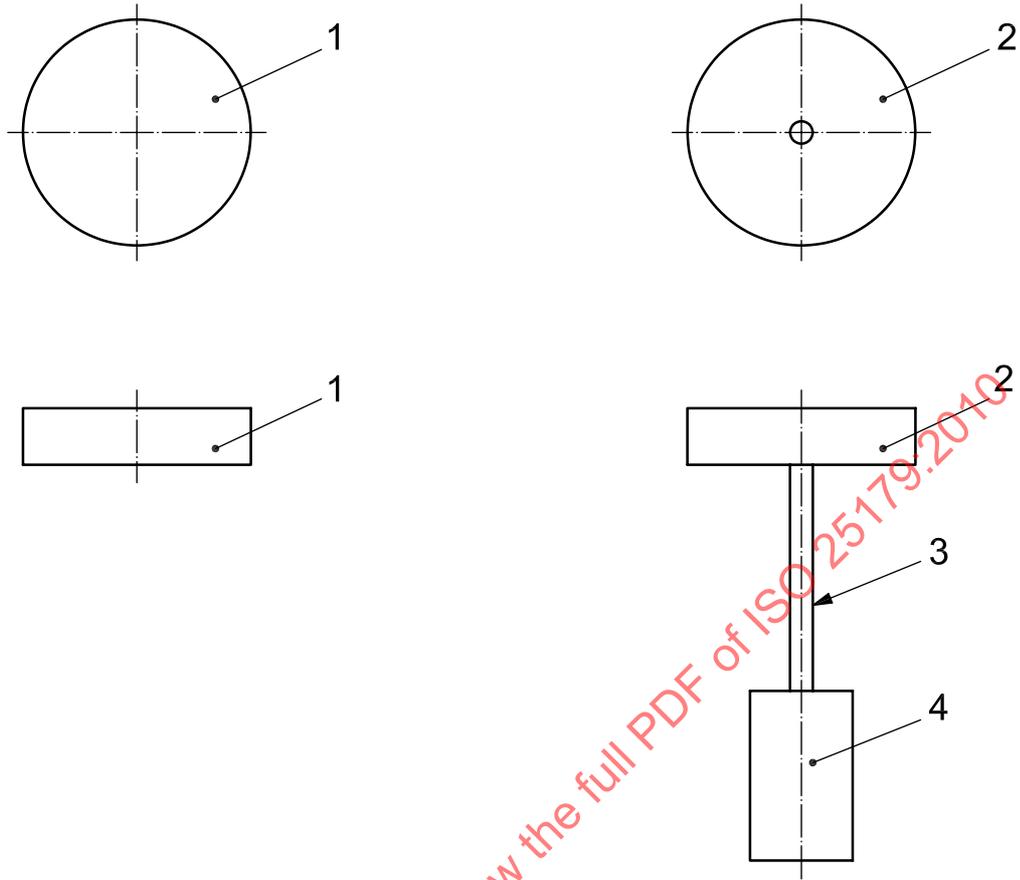
4.2 Bottles, made of glass, with a volume of 500 ml and capable of being sealed with a cap to prevent the escape of any vapour.

NOTE The method uses two types of bottle cap, an ordinary bottle cap and a prepared bottle cap (see Figure 2) to which the test specimen holder (see Figure 3) is connected.

4.3 Test specimen holder, comprising a cylindrical drum, two specimen-clamping bars and a connecting rod made of AISI 316 stainless steel, as shown in Figures 3, 4 and 5.

4.4 Drying oven, with forced-air convection, capable of being maintained at a temperature in the range from 50 °C to 120 °C to an accuracy of ± 2 °C.

4.5 Balance, capable of weighing the test specimen substrate and the coated test specimen to an accuracy of 0,000 1 g.

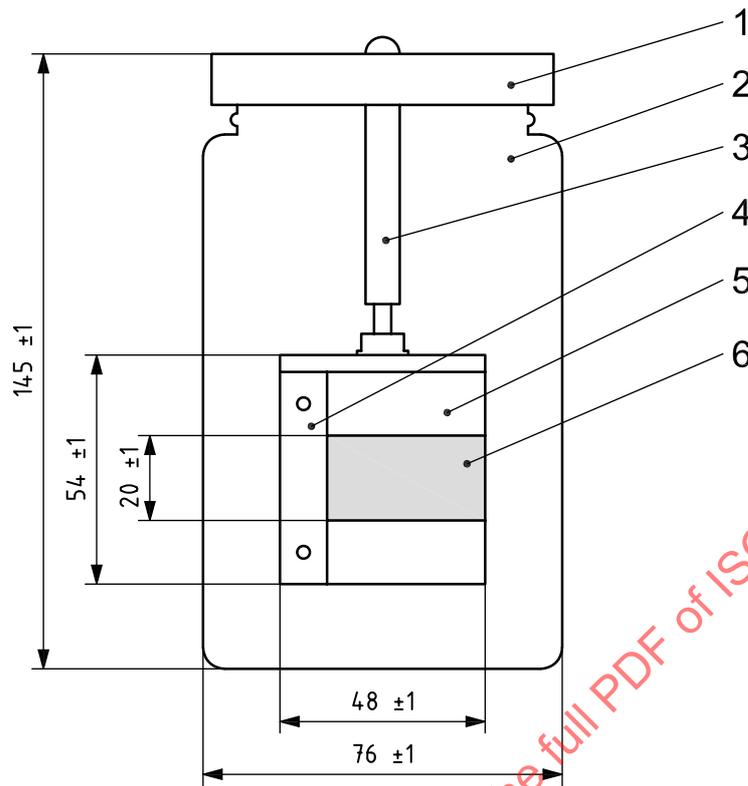


Key

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| 1 ordinary bottle cap | 3 connecting rod |
| 2 prepared bottle cap | 4 cylindrical drum of test specimen holder |

Figure 2 — Examples of ordinary and prepared bottle caps

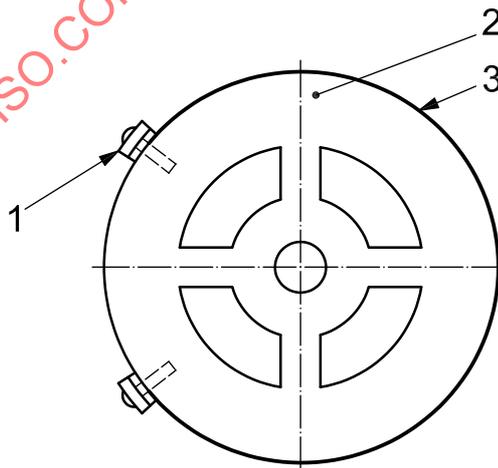
Dimensions in millimetres



Key

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|--------------|-------------------------|--|
| 1 bottle cap | 3 connecting rod | 5 cylindrical drum of test specimen holder |
| 2 bottle | 4 specimen-clamping bar | 6 test specimen |

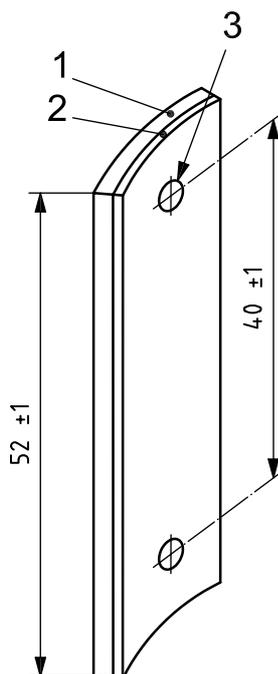
Figure 3 — Example of a bottle prepared for testing



Key

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|-------------------------|-----------------|
| 1 specimen-clamping bar | 3 test specimen |
| 2 cylindrical drum | |

Figure 4 — Example of a test specimen holder, viewed from above, showing the test specimen and the two specimen-clamping bars

**Key**

- | | | | |
|---|--|---|----------------|
| 1 | AISI 316 stainless steel, thickness 1,5 mm | 3 | fastening hole |
| 2 | polytetrafluoroethylene (PTFE), thickness 1 mm | | |

Figure 5 — Example of a specimen-clamping bar**5 Solutions and materials**

5.1 Distilled or deionized water, for dissolving water-soluble adhesives.

5.2 Aqueous alkali solution, made by dissolving $(1 \pm 0,01)$ parts by mass of reagent-grade sodium hydroxide (NaOH) in (99 ± 1) parts by mass of water, for dissolving alkali-soluble adhesives.

5.3 Repulpable kraft paper, with a mass per unit area between 80 g/m^2 and 100 g/m^2 , for use as the test specimen substrate.

6 Preparation of test specimens

6.1 Cut from the kraft paper (5.3) a sheet measuring 400 mm in length and 300 mm in width.

6.2 Place the sheet on a clean glass plate and coat with the adhesive being tested, using a laboratory-scale coating machine to produce a wet-film thickness of about $40 \mu\text{m}$.

6.3 Dry the coated sheet on the glass plate in the drying oven for 30 min at $80 \text{ }^\circ\text{C}$. Then remove the sheet from the drying oven and allow to cool to room temperature.

6.4 Remove the coated sheet from the glass plate and cut from it ten or more test specimens measuring 110 mm in length and 20 mm in width.

6.5 Draw lines across each test specimen at a distance of 5 mm from each end and condition the specimens using one of the sets of conditions specified in ISO 291.

6.6 Test specimens can also be prepared by cutting them from ready-made adhesive tape.

7 Procedure

7.1 Determine, by weighing, the initial mass of each test specimen and calculate the mass of the central 100 mm × 20 mm section [the section between the lines (see 6.5)] by multiplying by 100/110.

7.2 Clamp each test specimen on its respective test specimen holder with the adhesive-coated side facing away from the holder. Position the ends of the specimen under the specimen-clamping bars (see Figure 4) so that each clamping bar covers 5 mm at each end of the test specimen (see 6.5). Connect each test specimen holder to a prepared bottle cap using a connecting rod.

7.3 Fill the required number of bottles with 350 ml of distilled water (5.1) or 350 ml of alkali solution (5.2). Seal each bottle tightly using an ordinary bottle cap (see Figure 2). Place the bottles on the shaker plate inside the shaker chamber (see Figure 1). Close the chamber with the cover and set the inside temperature to 50 °C ± 2 °C and the shaker speed to 50 r/min. Other temperatures may be used if the solubility at a specific temperature is of interest. In addition, to accelerate the test the shaker speed and temperature can be increased.

7.4 90 min from the time all the bottles were placed in the shaker chamber, check the temperature in one of the bottles using a glass thermometer. If the temperature in the bottle has reached the test temperature, replace every ordinary bottle cap with a prepared bottle cap (see Figure 2) to which a test specimen holder carrying a test specimen has been attached, inserting the test specimen holder into the bottle and tightening the cap.

If the temperature in the bottle which was checked has not reached the desired test temperature, wait an additional 30 min and recheck a bottle. Continue to do this until the bottles are at the desired test temperature.

7.5 Continue to shake the bottles (with the test specimens in them). After a predetermined length of time (see next paragraph), stop the shaker and remove one of the bottles. Take off the cap, take out the test specimen holder and remove the test specimen. Dry the test specimen in the oven at 80 °C until there is no change in mass. Cut the 5 mm strip marked as described in 6.5 from each end of the test specimen.

The predetermined length of time is 5 min for fast-dissolving adhesives, 10 min for adhesives which dissolve at medium speed and 15 min to 20 min for slow-dissolving adhesives. This length of time may be changed by agreement between the adhesive manufacturer and testing agency.

7.6 Reweigh the test specimen (now measuring 100 mm × 20 mm) and record its mass m_t and the time t after which the bottle was removed from the shaker.

7.7 Repeat 7.5 and 7.6 for further successive lengths of time with the bottles remaining in the shaker. When the loss in mass of the test specimen removed becomes constant, dissolution of the adhesive has reached an equilibrium state and the test is terminated.

If the equilibrium state is reached before 50 % of the bottles have been removed, a shorter length of time is recommended to improve the accuracy of the results. If the equilibrium state has not been reached when all the bottles have been removed, a longer time is recommended.

7.8 Record the pattern of dissolution of the adhesive from the surface of the test specimen.

8 Expression of results

For each specimen removed from the shaker, calculate the amount of adhesive lost, expressed as a mass percentage, at time t using Equation (1).

$$\text{Amount of adhesive lost (\%)} \text{ at time } t = 100 \times (m_i - m_t) / (m_i - m_s) \quad (1)$$

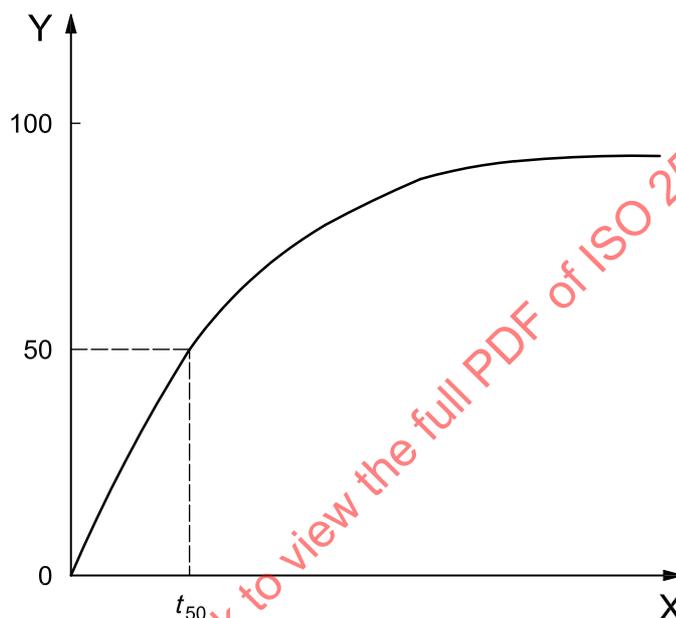
where

m_i is the initial (calculated) mass of the central 100 mm × 20 mm section of the test specimen;

m_t is the mass of the central 100 mm × 20 mm section of the test specimen (after cutting off the ends covered by the specimen-clamping bars) at time t ;

m_s is the mass of the substrate in the central 100 mm × 20 mm section of the test specimen (calculated from the mass per unit area of the kraft paper used).

Plot the amount of adhesive lost as the ordinate (Y) versus time as the abscissa (X), as shown in Figure 6.



Key

X time, t

Y amount of adhesive lost (mass percent)

t_{50} time at which 50 % of adhesive has been lost

Figure 6 — Plot of amount of adhesive lost versus time

9 Test report

The test report shall include the following information:

- a reference to this International Standard;
- all details necessary for identification of the adhesive tested, including type, coated quantity in g/m², source and manufacturer's code number;
- all details necessary for complete identification of the kraft paper used, including type, thickness, mass per unit area in g/m² and details of any surface treatment;
- details of the test specimen conditioning carried out before testing;
- the liquid used to dissolve off the adhesive (water or aqueous alkali solution);
- the temperature inside the shaker chamber, in °C, and the shaker speed, in r/min;