INTERNATIONAL STANDARD

ISO 1409

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Plastics/rubber — Polymer dispersions and rubber latices (natural and synthetic) — Determination of surface tension by the ring method

Plastiques/caoutchouc — Dispersions de polymères et latex de caoutchouc (naturel et synthétique) — Détermination de la tension superficielle par la méthode de l'anneau

Reference number
ISO 1409:1995(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 1409 was prepared jointly by Technical Committees ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry* and ISO/TC 35, *Paints and varnishes* and ISO/TC 61, *Plastics*.

This fourth edition cancels and replaces the third edition (ISO 1409:1983), the scope of which has been broadened to cover polymer dispersions in addition to rubber latices.

Annexes A and B of this International Standard are for information only.

International Organization for Standardization
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Plastics/rubber — Polymer dispersions and rubber latices (natural and synthetic) — Determination of surface tension by the ring method

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies a ring method for the determination of the surface tension of polymer dispersions and rubber latices (natural and synthetic).

The method is valid for polymer dispersions and rubber latices with a viscosity less than 200 mPa·s. To achieve this, the dispersion or latex is diluted with water to a total solids content of 40 % (m/m). If necessary, the solids content is further reduced to ensure that the viscosity is under the specified limit.

The method is also suitable for prevulcanized latices and compounded materials.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard 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 123:1985, *Rubber latex — Sampling.*

ISO 124:—[1], *Latex, rubber — Determination of total solids content.*

ISO 705:1994, *Rubber latex — Determination of density between 5 °C and 40 °C.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 1625:1977, *Plastics — Aqueous dispersions of polymers and copolymers — Determination of residue at 105 °C.*

ISO 1652:1985, *Rubber latex — Determination of viscosity.*

ISO 2555:1989, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method.*

ISO 3219:1993, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 8962:1987, *Plastics — Polymer dispersions — Determination of density.*

1) To be published. (Revision of ISO 124:1992)

3 Principle

A horizontally suspended ring of thin wire is attached to a du Nouy type tensiometer and immersed in the liquid under test, then slowly pulled out. Just before the ring detaches itself from the surface of the liquid, the force required reaches a maximum. This force is measured by a torsion balance, inductive pick-up or some other suitable measuring device.

4 Reagents

4.1 Distilled water, carbon-dioxide-free, or water of equivalent purity (grade 3 as defined in ISO 3696).

4.2 Toluene, of recognized analytical purity.

5 Apparatus

5.1 Tensiometer (du Nouy type), with a platinum or platinum-iridium alloy ring of either 60 mm or 40 mm nominal circumference (corresponding to 9,55 mm or 6,37 mm internal radius, respectively) and made of wire having a nominal radius of 0,185 mm.

5.2 Glass dish or vessel, of 50 cm^3 capacity, with an internal diameter of at least 45 mm.

5.3 Thermostatic bath, or some other means of adjusting the temperature of the test sample to 23 °C ± 1 °C (27 °C ± 1 °C for tropical countries).

NOTE 1 The temperature coefficient of rubber latices over the temperature range 20 °C to 30 °C is − 0,1 mN/m per degree Celsius.

6 Sampling

Carry out the sampling in accordance with one of the methods specified in either ISO 123 or ISO 842, as appropriate.

7 Procedure

7.1 Preparation of apparatus

Clean the dish or vessel (5.2) carefully, since any contamination may lead to variable results. Clean the ring of the tensiometer (5.1) by washing in water (4.1) and then heating in the oxidizing section of a Bunsen or methanol flame. Take extreme care to avoid touching or distorting the tensiometer ring when handling it; ensure that the ring remains parallel to the surface of the liquid during the determination. Failure to observe these precautions will lead to inaccurate results.

7.2 Calibration

Carefully calibrate the tensiometer scale against a standard mass or a reference liquid such as distilled water (4.1) or toluene (4.2) in accordance with the manufacturer's instructions, so that the scale will read in millinewtons per metre.

NOTE 2 Calibration is generally carried out against a standard mass. It should be noted that the calculation of the results (see clause 8) requires different correction factors depending on the method of calibration.

7.3 Preparation of the test sample

7.3.1 If the total solids content of the test sample of latex or polymer dispersion is not known, determine it in accordance with ISO 124 or ISO 1625, respectively. If the solids content is greater than 40 % (*m*/*m*), dilute the test sample to a total solids content of 40 % (*m*/*m*) ± 1 % (*m*/*m*) with water (4.1). If it is suspected that the viscosity of the diluted test sample is still greater than 200 mPa·s, determine it by the appropriate method (ISO 1652, ISO 2555 or ISO 3219). If necessary, dilute the test sample further until the viscosity is less than 200 mPa·s noting the final solids content.

NOTE 3 Dilution to 40 % (*m*/*m*) has a negligible effect on the surface tension of polymer dispersions and rubber latices. In some cases, the surface tension at a higher solids content may be required, in which case this may be measured by the method as specified with little loss of accuracy, provided that the viscosity is less than 200 mPa·s.

If the density of the diluted test sample of latex or polymer dispersion is not known, determine it in accordance with ISO 705 or ISO 8962.

7.3.2 Using the thermostatic bath (5.3), adjust the temperature of the diluted test sample to 23 °C ± 1 °C (or 27 °C ± 1 °C in tropical countries).

7.3.3 Remove approximately 25 cm^3 of the diluted test sample, using a pipette with its tip well below the surface of the liquid, and transfer to the dish or vessel (5.2). Remove any air bubbles from the surface of the sample by wiping with a piece of hard filter paper and measure the surface tension immediately to avoid errors due to the formation of surface skin.

7.4 Determination

With the tensiometer protected from air currents, place the dish or vessel containing the diluted test sample on the adjustable platform of the instrument beneath the ring of the tensiometer. Adjust the instrument, following the manufacturer's guidelines, so that the beam is in the balance position when the ring is dry and the scale reading is zero. Raise the platform until the liquid makes contact with the ring and then immerse the ring to a depth of approximately 5 mm.

Slowly lower the platform by means of the platform-adjusting screw, and simultaneously increase the torsion in the wire, proportioning these two adjustments so that the beam remains exactly in its balance position. As the film adhering to the ring approaches its breaking point, proceed very slowly with adjustments.

For determinations with an automatic tensiometer, regulate the penetration depth of the ring to approximately 5 mm below the surface of the liquid and adjust the rate of movement of the dish or vessel to 10 mm/min.

Record the maximum scale reading just prior to the point at which the ring separates from the dispersion or latex (this is of particular importance with undiluted latices or polymer dispersions of high viscosity).

Immediately raise the dish again before the film breaks, re-submerging the ring. Repeat the determination three times for a total of four determinations.

Should the film break, clean the ring as described in 7.1 and repeat the determination.

Discount the first reading and record the average of the next three readings, which should agree to within ± 0,5 mN/m of the median value.

8 Expression of results

8.1 Calibration against a standard mass

If (as is usual) the tensiometer has been calibrated against a standard mass, the reading must be corrected by a factor depending on the dimensions of the ring and the density of the liquid.

$$\sigma = M \times F$$

where

M is the scale reading, in millinewtons per metre;

F is the factor calculated from the equation

$$F = 0,725 + \sqrt{\frac{0,036\ 78M}{R^2\rho} + P}$$

in which

R is the mean radius, in millimetres, of the ring;

ρ is the density, in megagrams per cubic metre, of the liquid;

P is a constant calculated from the equation

$$P = 0,045\ 34 - \frac{1,679r}{R}$$

r being the radius, in millimetres, of the wire.

NOTES

4 Some formulae for calculating F include the gravitational constant G. This has been incorporated directly into the constant 0,036 78 to avoid possible confusion over units.

5 For standard rings of 60 mm or 40 mm nominal circumference (R = 9,55 mm or 6,37 mm, respectively) and wire of nominal radius 0,185 mm, P will be given by

$P_{60} = +\ 0,012\ 82$

or

$P_{40} = -\ 0,003\ 43$

6 "Surface tension" and "surface free energy" are synonymous and have equal numerical values when expressed in millinewtons per metre and millijoules per metre squared, respectively.

7 Since it may not be practical to calculate the factor F separately for each sample tested, it is convenient to create tables for a correction index $\Delta\sigma$ depending on the wire dimensions (see annex B).

8.2 Calibration against a standard liquid

If the calibration has been carried out using a reference liquid, then the surface tension σ', expressed in millinewtons per metre, is given by:

$$\sigma' = M' \times F'$$

where

M' is the reading for the diluted test sample, in millinewtons per metre, on the scale calibrated with the reference liquid;

F' is a factor calculated from the equation

$$F' = F \times \sigma''/M''$$

in which

F is calculated as shown in 8.1;

σ'' is the known surface tension of the reference liquid;

M'' is the actual scale reading recorded for the reference liquid.

NOTE 8 It is particularly important to read the equipment manufacturer's instructions carefully if using this procedure, as some corrections may have been built into the equipment. For this reason, calculated corrections are not given in annex B.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the identification of the sample tested;

c) the temperature at which the test was carried out;

d) the solids content of the test sample originally, and as tested after dilution;

e) the results and the units in which they have been expressed;

f) any unusual features noted during the determination;

g) the date and place where the determination was carried out;

h) any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional.

Annex A
(informative)

Precision of the test method

With accurate operation and control, i.e. correctly following the details of the procedure, it is possible to obtain the following:

repeatability: 1,0 mN/m

reproducibility: 2,0 mN/m

NOTE 9 The work to generate the precision data was initiated before the publication of ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards.* Consequently the results are not expressed in the recommended format.

Annex B
(informative)

Correction factors

The calculation of correction factors for each individual evaluation is time-consuming. Where tensiometers with electronic data processing are used, the results may be automatically corrected. If this is not practical, it is suggested that it is more convenient to express the correction factors F as correction indices $\Delta\sigma$ that can be subtracted from the scale reading M:

$$\sigma = M - \Delta\sigma$$

rather than

$$\sigma = M \times F$$

Table B.1 and table B.2 contain such correction indices for rings with 40 mm and 60 mm circumference and a mean radius of the wire of 0,185 mm.

Table B.1 — Correction index $\Delta\sigma$, for 40-mm-circumference tensiometer ring, to be subtracted from the scale reading M

Scale reading, M	**20**	**30**	**40**	**45**	**50**	**55**	**60**	**65**	**70**	**72**
Density, ρ (Mg/m³)	**Correction index, $\Delta\sigma$**									
0,85	2,8	3,2	3,1	2,9	2,6	2,2	1,7	1,2	0,6	0,3
0,95	3,0	3,5	3,5	3,4	3,2	2,9	2,6	2,1	1,6	1,4
1,05	3,2	3,8	3,9	3,9	3,8	3,6	3,3	3,0	2,5	2,4
1,15	3,3	4,0	4,3	4,3	4,3	4,1	3,9	3,7	3,3	3,2
1,25	3,4	4,2	4,6	4,7	4,7	4,6	4,5	4,3	4,0	3,9

Table B.2 — Correction index $\Delta\sigma$, for 60-mm-circumference tensiometer ring, to be subtracted from the scale reading M

Scale reading, M	**20**	**30**	**40**	**45**	**50**	**55**	**60**	**65**	**70**	**72**
Density, ρ (Mg/m³)	**Correction index, $\Delta\sigma$**									
0,85	2,5	3,3	3,9	4,1	4,2	4,3	4,3	4,3	4,2	4,2
0,95	2,6	3,5	4,1	4,3	4,5	4,7	4,8	4,8	4,8	4,8
1,05	2,6	3,6	4,3	4,6	4,8	5,0	5,1	5,2	5,3	5,3
1,15	2,7	3,7	4,4	4,8	5,0	5,3	5,5	5,6	5,7	5,8
1,25	2,7	3,8	4,6	4,9	5,2	5,5	5,7	5,9	6,1	6,1

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