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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 1409:2006(E)

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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 1409 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 1409:1995), of which it constitutes a minor revision, primarily concerning updating of references and the deletion of references to the withdrawn standards ISO 842, ISO 1625 and ISO 8962.

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 mass fraction of total solids of 40 %. 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 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 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

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

ISO 1652, *Rubber latex — Determination of apparent viscosity by the Brookfield test method*

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

ISO 3219, *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*

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 3696:1987).

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 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 ISO 123.

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 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. If the mass fraction of solids is greater than 40 %, dilute the test sample to a mass fraction of total solids of 40 % ± 1 % 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 Dilution to a mass fraction of 40 % 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.

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 it 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. 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 standard mass

If (as is usual) the tensiometer has been calibrated against a standard mass, the reading (σ) 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;

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

P is a constant calculated from the equation

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

where r is the radius, in millimetres, of the wire.

NOTE 1 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.

NOTE 2 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

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

NOTE 3 “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.

NOTE 4 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 in 8.1;

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

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

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 usual 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.