

ISO/TC 45/SC 3

Secretariat: AFNOR

Voting begins on:
2020-05-20

Voting terminates on:
2020-07-15

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

*Plastiques/caoutchouc — Dispersions de polymères et latex de
caoutchouc (naturel et synthétique) — Détermination de la tension
superficielle*

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
<https://standards.iteh.ai/catalog/standards/sic/5533aa20-f853-4e27-ab4c-c96aa5ee5bd8/iso-fdis-1409>

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.



Reference number
ISO/FDIS 1409:2020(E)

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
<https://standards.iteh.ai/catalog/standards/sist/58e3aa2d-f853-4e27-ab4c-c96aa5ee5bd8/iso-fdis-1409>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2020

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
4.1 Method A	2
4.2 Method B	2
5 Reagents	2
6 Apparatus	2
7 Sampling	2
8 Procedure	3
8.1 Method A	3
8.1.1 Preparation of apparatus	3
8.1.2 Calibration	3
8.1.3 Preparation of the test sample	3
8.1.4 Determination	3
8.2 Method B	4
8.2.1 Preparation of apparatus	4
8.2.2 Calibration	4
8.2.3 Preparation of the test sample	4
8.2.4 Determination	5
9 Expression of results	6
9.1 Method A	6
9.1.1 Calibration against standard mass	6
9.1.2 Calibration against a standard liquid	7
9.2 Method B	8
10 Precision	8
11 Test report	8
Annex A (informative) Precision	9
Annex B (informative) Correction factors of method A	11
Bibliography	12

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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This sixth edition cancels and replaces the fifth edition (ISO 1409:2006), which has been technically revised. The main changes compared to the previous edition are as follows:

- extension of [Clause 1](#) and [Clause 4](#);
- inclusion in [Clause 1](#) that method A is the preferred method in case of dispute;
- inclusion of the tensiometer (Wilhelmy type) and glass dish or vessel in [6.2](#) and [6.3](#);
- inclusion of the procedure of the new method B in [8.2](#);
- expression of the test results according to the new method B in [9.2](#);
- inclusion of the precision data for method B in [Annex A](#).

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.

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

1 Scope

This document specifies two methods for the determination of the surface tension of polymer dispersions and rubber latices (natural and synthetic).

- Method A is the ring method (Du Noüy ring method).
- Method B is the plate method (Wilhelmy plate method).

Method A is suitable valid for polymer dispersions and rubber latices with a viscosity less than 200 mPa·s.

Method B is not suitable for polymer dispersions and rubber latices containing cationic surfactants.

Methods A and B are also suitable for prevulcanized latices and compounded material.

In case of dispute, the preferred method is method A (the ring method).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 degrees C and 40 degrees 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 using a single cylinder type rotational viscometer 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*

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 <http://www.electropedia.org/>

4 Principle

4.1 Method A

Method A is suitable 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 solid content is further reduced to ensure that the viscosity is under the specified limit.

A horizontally suspended ring of thin wire is attached to a "Du Noüy" 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.

4.2 Method B

When a liquid is brought into contact with the edge of the plate which is suspended from the "Wilhelmy type" tensiometer, the plate is pulled down into the liquid due to the surface tension. The instrument applies an upward vertical force to the plate so that the plate reaches the force balance. This vertical force is measured and used to calculate the surface tension of the liquid.

5 Reagents

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

5.2 Toluene, of recognized analytical purity.

6 Apparatus

6.1 Tensiometer (Du Noüy 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.

6.2 Tensiometer (Wilhelmy type), with a flat, thin plate made of platinum or platinum-iridium alloy, and the size suitable for the tensiometer.

6.3 Glass dish or vessel, of 50 cm³ capacity, with an internal diameter of at least 45 mm or suitable for the thermostat of the tensiometer.

6.4 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.

6.5 Bunsen or ethanol lamp, or some other means of heating and cleaning the ring or plate.

7 Sampling

Carry out the sampling in accordance with one of the methods specified in ISO 123.

8 Procedure

8.1 Method A

8.1.1 Preparation of apparatus

Clean the dish or vessel (6.3) carefully, since any contamination may lead to variable results. Clean the ring of the tensiometer (6.1) by washing in water (5.1) and then heating in the oxidizing section of a Bunsen or ethanol flame (6.5). 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.

8.1.2 Calibration

Carefully calibrate the tensiometer scale against a standard mass or a reference liquid such as distilled water (5.1) or toluene (5.2) in accordance with the manufacturer's instructions, so that the scale will read in mN/m.

NOTE Calibration is generally carried out against a standard mass. It should be noted that the calculation of the results (see Clause 9) requires different correction factors depending on the method of calibration.

8.1.3 Preparation of the test sample

8.1.3.1 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 $40 \% \pm 1 \%$ with water (5.1). 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 given in 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 can be required, in which case this can 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.

8.1.3.2 Using the thermostatic bath (6.4), adjust the temperature of the diluted test sample to $23\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ (or $27\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ in tropical countries).

8.1.3.3 Take approximately 25 cm³ 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 (6.3). 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.

8.1.4 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 8.1.1 and repeat the determination.

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

8.2 Method B

8.2.1 Preparation of apparatus

Clean the dish or vessel (6.3) carefully. The surface of the plate (6.2) should be roughened enough to obtain a perfect wetting and with no bright spots on the plate. Rinse the plate thoroughly by washing in water (5.1), then heating in the oxidizing section of a gas burner or Bunsen (6.5) until the plate is clean. Hang the plate on the instrument after cooling to room temperature. Make sure that the lower edge of the plate is exactly straight by checking whether the light gap between the edge of the plate and the surface of the liquid is parallel and not wedge-shaped. Ensure that the plate remains parallel to the surface of the liquid during the determination.

NOTE Any contamination can lead to variable results and failure to observe these precautions will lead to inaccurate results.

8.2.2 Calibration

8.2.2.1 Calibrate the tensiometer (6.2) scale against standard mass or distilled water (5.1) in accordance with the manufacturer's instructions, so that the scale will read in mN/m.

8.2.2.2 If the calibration is carried out against distilled water, determine the surface tension of distilled water; the result should be $72,28 \text{ mN/m} \pm 0,30 \text{ mN/m}$ at 23°C or $71,66 \text{ mN/m} \pm 0,30 \text{ mN/m}$ at 27°C .

8.2.3 Preparation of the test sample

8.2.3.1 Using the thermostatic bath (6.4), adjust the temperature of the test sample to $23^\circ\text{C} \pm 1^\circ\text{C}$ (or $27^\circ\text{C} \pm 1^\circ\text{C}$ in tropical countries).

8.2.3.2 Rinse the dish or vessel (6.3) with a small amount of the test sample, and then transfer approximately 30 cm^3 of the test sample to the dish or vessel by a pipette with its tip well below the surface of the liquid. Make sure the depth of the test sample is at least 10 mm. 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.

NOTE For the polymer dispersions or rubber latices with high viscosity, the equilibration time of the surface tension can be longer, and it can be shortened by diluting the test sample to a mass fraction of total solids of $40\% \pm 1\%$ with water.

8.2.4 Determination

8.2.4.1 General

Two alternative plate methods are specified, which can be chosen according to the instruments:

- method B1: carry out the measurement when the pre-wetted plate is suddenly caught by the liquid;
- method B2: immerse the plate in the liquid to a certain depth, and then carry out the measurement when the plate is pulled back into contact with the surface of the liquid.

8.2.4.2 Method B1

Adjust the instrument following the manufacturer's instructions.

Place the dish or vessel containing the test sample on the adjustable platform of the instrument beneath the plate of the tensiometer and tare the reading to zero.

After pre-wetting the plate in the liquid to a depth of 2 mm to 5 mm according to the manufacturer's instructions, pull the plate out of the liquid.

Raise the platform slowly until the plate is suddenly caught by the liquid. Record the stable reading from the tensiometer. Lower the platform until the plate separates from the liquid. Repeat the determination of contact separation another two times without taring the reading to zero and cleaning the plate.

Discount the first reading and record the average of the next two readings, which difference should be within $\pm 0,5$ mN/m.

8.2.4.3 Method B2

Adjust the instrument following the manufacturer's instructions.

Place the dish or vessel containing the test sample on the adjustable platform of the instrument beneath the plate of the tensiometer and tare the reading to zero.

Raise the platform until the lower edge of the plate is just submerged and mark the position of the platform according to the manufacturer's instructions.

Raise the platform another 2 mm to 5 mm to ensure the wetting of the plate.

Lower the platform until it is located exactly to the marked position again, that is the lower edge of the plate comes into contact with the surface of the liquid again. Record the stable reading from the tensiometer.

After lowering the platform until the plate separates from the liquid, tare the reading to zero and repeat the determination without cleaning the plate.

Make at least three measurements until the difference between two successive readings is not greater than $\pm 0,5$ mN/m. Discount the first reading and record the average of the last two readings.

For method B1 or method B2, if an automatic tensiometer that can display the change of surface tension in real time is used for measurement, adjust the instrument and set the parameters following the manufacturer's instructions. Some recommended values are given in [Table 1](#) and the typical surface tension curve is given in [Figure 1](#). For [Table 1](#), the test result is the mean value of the last 5 readings when the maximum measuring time is reached.

NOTE [Table 1](#) shows only the recommended maximum measuring time. Before the formation of the surface skin, the maximum measuring time for different test sample can be determined according to the surface tension curve.