



DPC: 24/30457124 DC

BSI Group Headquarters

389 Chiswick High Road London W4 4AL

Tel: +44 (0)20 8996 9000

Fax: +44 (0)20 8996 7400

www.bsigroup.com

Date: 22 January 2024

Origin: International

Latest date for receipt of comments: 16 March 2024

Project No. 2022/01290

Responsible committee: PRI/21 Testing of plastics

Interested committees:

Title: Draft BS ISO 6721-10 Plastics - Determination of dynamic mechanical properties. Part 10: Complex shear viscosity using a parallel-plate and a cone-and-plate oscillatory rheometer.

Please notify admin.start@bsigroup.com if you are aware of any keywords that might assist in clarifying or identifying the standard or of the content of this standard:

- i) has any issues related to 3rd party IPR, patent or copyright
- ii) affects other national standard(s)
- iii) requires additional national guidance or information

WARNING: THIS IS A DRAFT AND MUST NOT BE REGARDED OR USED AS A BRITISH STANDARD. THIS DRAFT IS NOT CURRENT BEYOND 16 March 2024

This draft is issued to allow comments from interested parties; all comments will be given consideration prior to publication. No acknowledgement will normally be sent. **See overleaf for information on the submission of comments.**

No copying is allowed, in any form, without prior written permission from BSI except as permitted under the Copyright, Designs and Patent Act 1988 or for circulation within a nominating organization for briefing purposes. Electronic circulation is limited to dissemination by e-mail within such an organization by committee members.

Further copies of this draft may be purchased from BSI Shop <http://shop.bsigroup.com> or from BSI Customer Services, Tel: +44(0) 20 8996 9001 or email cservices@bsigroup.com. British, International and foreign standards are also available from BSI Customer Services.

Information on the co-operating organizations represented on the committees referenced above may be obtained from <https://standardsdevelopment.bsigroup.com/>.

Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

For national standards, comments will be reviewed by the relevant UK national committee and the resulting standards published as a British Standard.

UK Vote

Please indicate whether you consider the UK should submit a negative (with supporting technical reasons) or positive vote on this draft. Please indicate if you are aware of any reason why this draft standard should not be published as a British Standard.

Submission of Comments

- **Annotated drafts are not acceptable and will be rejected.**
- All comments should be submitted online at <https://standardsdevelopment.bsigroup.com/>. You will need to register in order to comment.

Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
------------------	------------------------

1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	

DRAFT INTERNATIONAL STANDARD

ISO/DIS 6721-10

ISO/TC 61/SC 5

Secretariat: DIN

Voting begins on:
2024-01-22

Voting terminates on:
2024-04-15

Plastics — Determination of dynamic mechanical properties —

Part 10:

Complex shear viscosity using a parallel-plate and a cone-and-plate oscillatory rheometer

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 10: Viscosité complexe en cisaillement à l'aide d'un rhéomètre à oscillations à plateaux parallèles ou à géométrie cône/plan

ICS: 83.080.01

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

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.

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.

This document is circulated as received from the committee secretariat.



Reference number
ISO/DIS 6721-10:2024(E)

AVA Documents
Document Sharing Hub
© ISO 2024



COPYRIGHT PROTECTED DOCUMENT

© ISO 2024

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
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
5 Apparatus	3
5.1 Measurement apparatus.....	3
5.2 Temperature-controlled enclosure.....	4
5.3 Temperature measurement and control.....	4
5.4 Measurement geometry.....	4
5.4.1 Parallel plates geometry.....	4
5.4.2 Cone and plate geometry.....	5
5.5 Calibration.....	5
6 Sampling	5
7 Procedure	6
7.1 Test temperature.....	6
7.2 Zeroing the gap.....	6
7.3 Introducing the test specimen.....	6
7.4 Conditioning the test specimen.....	6
7.5 Test mode (controlled stress or controlled strain).....	6
7.5.1 Controlled strain mode.....	7
7.5.2 Controlled stress mode.....	7
7.6 Determination of thermal stability of sample material.....	7
7.7 Determination of region of linear-viscoelastic behaviour.....	7
7.7.1 In the controlled-strain mode.....	7
7.7.2 In the controlled-stress mode.....	8
7.7.3 Confirmation of linear-viscoelastic behaviour.....	8
7.8 Frequency sweep.....	8
7.9 Temperature sweep.....	8
7.10 Air entrapment.....	9
8 Expression of results	9
8.1 Symbols used.....	9
8.2 Calculation of complex shear modulus and complex shear viscosity.....	10
9 Precision	11
10 Test report	12
Annex A (informative) Uncertainty limits	14
Annex B (informative) Verification of rheometer performance	17
Bibliography	21

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 61, *Plastics*, Subcommittee SC 5, Physical-chemical properties.

This fourth edition cancels and replaces the third edition (ISO 6721-10:2015), which has been technically revised.

The main changes compared to the previous edition are as follows:

- rheometer geometry is described in detail for both parallel-plate and cone-and-plate geometry;
- in the test mode, controlled stress mode and controlled strain mode are described in separate sections.

A list of all parts in the ISO 6721 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.

Plastics — Determination of dynamic mechanical properties —

Part 10:

Complex shear viscosity using a parallel-plate and a cone-and-plate oscillatory rheometer

1 Scope

This document specifies the general principles of a method for determining the dynamic rheological properties of polymer melts at angular frequencies typically in the range of $0,01 \text{ rad}\cdot\text{s}^{-1}$ to $100 \text{ rad}\cdot\text{s}^{-1}$ by means of an oscillatory rheometer with a parallel-plate or a cone-and-plate geometry. Angular frequencies outside this range can also be used (see Note 1). The method is applicable to determine values of the following dynamic rheological properties: complex shear viscosity η^* , dynamic shear viscosity η' , the out-of-phase component of the complex shear viscosity η'' , complex shear modulus G^* , shear loss modulus G'' , shear storage modulus G' , phase angle δ , and loss factor $\tan\delta$. It is suitable for measuring complex shear viscosity values typically up to $\sim 10 \text{ MPa}\cdot\text{s}$ (see Note 2).

NOTE 1 The angular-frequency measurement range is limited by the specification of the measuring instrument and also by the response of the specimen. When testing using angular frequencies lower than $0,1 \text{ rad}\cdot\text{s}^{-1}$, the test time can increase significantly as the time taken to obtain a single measurement is proportional to the reciprocal of the angular frequency. Consequently, when testing at low angular frequencies, degradation or polymerization of the specimen is more likely to occur and have an effect on the results. At high angular frequencies, the specimen can distort or fracture at the edge, consequently invalidating the test results.

NOTE 2 The range of complex shear viscosity values that can be measured is dependent on the specimen dimensions determined by the diameter of the geometry used and also the specification of the measuring instrument. For a specimen of given dimensions, the upper limit of the range is limited by the machine's torque capacity, angular-displacement resolution, motor inertia, and compliance. However, corrections can be made for compliance effects.

NOTE 3 The shear loss modulus G'' is sometimes also called viscous shear modulus and the shear storage modulus G' is sometimes also called elastic shear modulus.

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 472, *Plastics — Vocabulary*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 6721-1, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6721-1, ISO 5725-1, ISO 472, and the following apply.

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/>

3.1

controlled-strain mode

testing by applying a sinusoidal angular displacement of constant amplitude

3.2

controlled-stress mode

testing by applying a sinusoidal torque of constant amplitude

3.3

complex shear viscosity

η^*

ratio of dynamic stress, given by $\sigma(t) = \sigma_0 \exp(i\omega t)$, and dynamic rate of strain where the shear strain $\gamma(t)$ is given by $\gamma(t) = \gamma_0 \exp\{i(\omega t - \delta)\}$, of a viscoelastic material that is subjected to a sinusoidal vibration, where σ_0 and γ_0 are the amplitudes of the stress and strain cycles, ω is the angular frequency, δ is the phase angle between the stress and strain, and t is time

Note 1 to entry: It is expressed in pascal seconds.

3.4

dynamic shear viscosity

η'

real part of the complex shear viscosity

Note 1 to entry: It is expressed in pascal seconds.

3.5

out-of-phase component of the complex shear viscosity

η''

imaginary part of the complex shear viscosity

Note 1 to entry: It is expressed in pascal seconds.

4 Principle

The specimen is held between two concentric, circular parallel plates or cone-and-plate (see [Figure 1](#) and [2](#)). The thickness of the specimen is small compared with the diameter of the plates.

One of the plates or cone is subjected to either a sinusoidal torque or a sinusoidal angular displacement of constant angular frequency. These are referred to as “controlled-stress” or “controlled-strain” test modes, respectively. When using the controlled-stress mode, the resultant displacement and the phase shift between the torque and displacement are measured. When using the controlled-strain mode, the resultant torque and the phase shift between the displacement and torque are measured.

The complex shear modulus G^* , shear storage modulus G' , shear loss modulus G'' , phase angle δ , and corresponding shear viscosity terms (see [Clause 3](#)) are determined from the measured torque and displacement and the specimen dimensions. In deriving these values, it is assumed that the specimen exhibits a linear-viscoelastic response.

The mode of oscillation used is designated as oscillatory mode I (see ISO 6721-1).

5 Apparatus

5.1 Measurement apparatus

The measurement apparatus shall consist of two concentric, rigid, circular parallel plates (see [Figure 1](#)) or cone-and-plate (see [Figure 2](#)) between which the specimen is placed. One of these plates or one side of cone-and-plate shall be made to oscillate at a constant angular frequency while the other remains at rest.

The requirements on the apparatus are that it shall permit measurement of the amplitudes of the torque and the angular displacement and the phase difference between them for a specimen subjected to either a sinusoidal torque or a sinusoidal displacement of constant angular frequency.

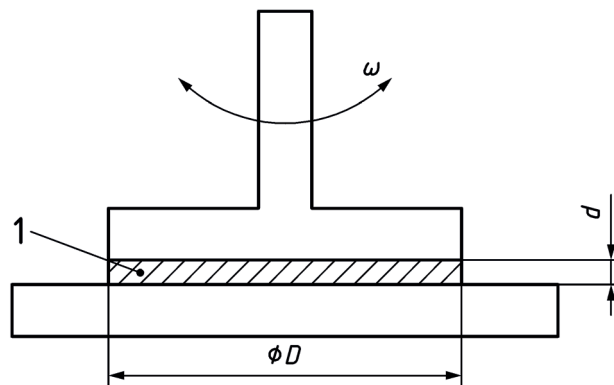
The torque required to overcome the viscoelastic resistance of the specimen shall be determined, for example, by connecting a torque-measuring device to one of the plates or one of cone and plate.

An angular-displacement measuring device shall be fitted to one of the plates or one of cone or plate, thus permitting determination of its angular displacement and angular frequency.

The apparatus shall be capable of measuring the torque to within $\pm 2\%$ of the minimum torque amplitude used to determine the dynamic properties.

The apparatus shall be capable of measuring the angular displacement to within $\pm 20 \times 10^{-6}$ rad.

The apparatus shall be capable of measuring the angular frequency to within $\pm 2\%$ of the absolute value.



Key

- 1 test specimen
- ω angular frequency
- d specimen thickness
- D diameter of plate

Figure 1 — Parallel-plate rheometer geometry