

INTERNATIONAL STANDARD

NORME INTERNATIONALE



Insulating liquids – Test methods for oxidation stability
Test method for evaluating the oxidation stability of insulating liquids in the delivered state

Isolants liquides – Méthodes d'essai de la stabilité à l'oxydation
Méthode d'essai pour évaluer la stabilité à l'oxydation des isolants liquides tels que livrés



THIS PUBLICATION IS COPYRIGHT PROTECTED

Copyright © 2018 IEC, Geneva, Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either IEC or IEC's member National Committee in the country of the requester. If you have any questions about IEC copyright or have an enquiry about obtaining additional rights to this publication, please contact the address below or your local IEC member National Committee for further information.

Droits de reproduction réservés. Sauf indication contraire, aucune partie de cette publication ne peut être reproduite ni utilisée sous quelque forme que ce soit et par aucun procédé, électronique ou mécanique, y compris la photocopie et les microfilms, sans l'accord écrit de l'IEC ou du Comité national de l'IEC du pays du demandeur. Si vous avez des questions sur le copyright de l'IEC ou si vous désirez obtenir des droits supplémentaires sur cette publication, utilisez les coordonnées ci-après ou contactez le Comité national de l'IEC de votre pays de résidence.

IEC Central Office
3, rue de Varembe
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
info@iec.ch
www.iec.ch

About the IEC

The International Electrotechnical Commission (IEC) is the leading global organization that prepares and publishes International Standards for all electrical, electronic and related technologies.

About IEC publications

The technical content of IEC publications is kept under constant review by the IEC. Please make sure that you have the latest edition, a corrigenda or an amendment might have been published.

IEC Catalogue - webstore.iec.ch/catalogue

The stand-alone application for consulting the entire bibliographical information on IEC International Standards, Technical Specifications, Technical Reports and other documents. Available for PC, Mac OS, Android Tablets and iPad.

IEC publications search - webstore.iec.ch/advsearchform

The advanced search enables to find IEC publications by a variety of criteria (reference number, text, technical committee,...). It also gives information on projects, replaced and withdrawn publications.

IEC Just Published - webstore.iec.ch/justpublished

Stay up to date on all new IEC publications. Just Published details all new publications released. Available online and also once a month by email.

Electropedia - www.electropedia.org

The world's leading online dictionary of electronic and electrical terms containing 21 000 terms and definitions in English and French, with equivalent terms in 16 additional languages. Also known as the International Electrotechnical Vocabulary (IEV) online.

IEC Glossary - std.iec.ch/glossary

67 000 electrotechnical terminology entries in English and French extracted from the Terms and Definitions clause of IEC publications issued since 2002. Some entries have been collected from earlier publications of IEC TC 37, 77, 86 and CISPR.

IEC Customer Service Centre - webstore.iec.ch/csc

If you wish to give us your feedback on this publication or need further assistance, please contact the Customer Service Centre: sales@iec.ch.

A propos de l'IEC

La Commission Electrotechnique Internationale (IEC) est la première organisation mondiale qui élabore et publie des Normes internationales pour tout ce qui a trait à l'électricité, à l'électronique et aux technologies apparentées.

A propos des publications IEC

Le contenu technique des publications IEC est constamment revu. Veuillez vous assurer que vous possédez l'édition la plus récente, un corrigendum ou amendement peut avoir été publié.

Catalogue IEC - webstore.iec.ch/catalogue

Application autonome pour consulter tous les renseignements bibliographiques sur les Normes internationales, Spécifications techniques, Rapports techniques et autres documents de l'IEC. Disponible pour PC, Mac OS, tablettes Android et iPad.

Recherche de publications IEC - webstore.iec.ch/advsearchform

La recherche avancée permet de trouver des publications IEC en utilisant différents critères (numéro de référence, texte, comité d'études,...). Elle donne aussi des informations sur les projets et les publications remplacées ou retirées.

IEC Just Published - webstore.iec.ch/justpublished

Restez informé sur les nouvelles publications IEC. Just Published détaille les nouvelles publications parues. Disponible en ligne et aussi une fois par mois par email.

Electropedia - www.electropedia.org

Le premier dictionnaire en ligne de termes électroniques et électriques. Il contient 21 000 termes et définitions en anglais et en français, ainsi que les termes équivalents dans 16 langues additionnelles. Egalement appelé Vocabulaire Electrotechnique International (IEV) en ligne.

Glossaire IEC - std.iec.ch/glossary

67 000 entrées terminologiques électrotechniques, en anglais et en français, extraites des articles Termes et Définitions des publications IEC parues depuis 2002. Plus certaines entrées antérieures extraites des publications des CE 37, 77, 86 et CISPR de l'IEC.

Service Clients - webstore.iec.ch/csc

Si vous désirez nous donner des commentaires sur cette publication ou si vous avez des questions contactez-nous: sales@iec.ch.

INTERNATIONAL STANDARD

NORME INTERNATIONALE



Insulating liquids – Test methods for oxidation stability
Test method for evaluating the oxidation stability of insulating liquids in the delivered state

Isolants liquides – Méthodes d’essai de la stabilité à l’oxydation
Méthode d’essai pour évaluer la stabilité à l’oxydation des isolants liquides tels que livrés

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

COMMISSION
ELECTROTECHNIQUE
INTERNATIONALE

ICS 29.040.10

ISBN 978-2-8322-5210-9

Warning! Make sure that you obtained this publication from an authorized distributor.
Attention! Veuillez vous assurer que vous avez obtenu cette publication via un distributeur agréé.

CONTENTS

FOREWORD.....	5
1 Scope.....	7
2 Normative references	7
3 Terms and definitions	8
4 Apparatus.....	9
4.1 General principle of the method	9
4.2 Equipment	9
4.2.1 Heating arrangement	9
4.2.2 Test vessels	10
4.2.3 Absorption tubes.....	10
4.2.4 Filtering crucibles	10
4.2.5 Porcelain vessels.....	11
4.2.6 Flowmeter.....	11
4.2.7 Timer	11
4.2.8 Gas supply	11
4.2.9 Analytical balance	11
4.2.10 Burette	11
4.2.11 Volumetric pipette.....	11
4.2.12 Volumetric flask.....	12
4.2.13 Graduated measuring cylinder	12
4.2.14 Thermometer	12
4.2.15 Erlenmeyer flask.....	12
4.3 Reagents	12
4.3.1 Normal heptane	12
4.3.2 Alkali blue 6B indicator according to IEC 62021-2.....	12
4.3.3 Phenolphthalein indicator	12
4.3.4 Potassium hydroxide according to IEC 62021-2	12
4.3.5 Oxidant gas	12
4.3.6 Acetone	12
4.4 Cleaning of test vessels	12
4.5 Catalyst	13
4.6 Insulating liquid sample conditioning.....	13
4.7 Preparation of the test	13
4.8 Determinations on the oxidized insulating liquid.....	13
4.8.1 Sludge formation	13
4.8.2 Soluble acidity (SA)	14
4.8.3 Volatile acidity (VA)	14
4.8.4 Total acidity (TA)	15
4.8.5 Dielectric dissipation factor (DDF)	15
4.8.6 Oxidation rate with air.....	15
4.8.7 Induction period with air (IP with air) (optional)	15
4.9 Report.....	15
4.10 Precision.....	16
4.10.1 General	16
4.10.2 Repeatability (<i>r</i>) (95 % confidence)	16
4.10.3 Reproducibility (<i>R</i>) (95 % confidence).....	16

Annex A (normative) Thermometer specifications	20
Annex B (informative) Method for evaluating the oxidation stability of inhibited insulating liquids in the delivery state by measurement of the induction period with oxygen.....	21
B.1 Outline of the method.....	21
B.2 Reagents and test conditions	21
B.3 Procedure	21
B.3.1 General	21
B.3.2 Preparation of the test	21
B.3.3 Oxidation.....	22
B.3.4 Determination of the induction period with oxygen	22
B.3.5 Determinations on the oxidized oil (optional).....	22
B.4 Report.....	23
B.5 Precision.....	23
B.5.1 General	23
B.5.2 Relative repeatability (<i>r</i>) (95 % confidence).....	23
B.5.3 Relative reproducibility (<i>R</i>) (95 % confidence)	23
Annex C (informative) Method for evaluation of thermo-oxidative behaviour of unused ester insulating liquids	24
C.1 Outline of the method.....	24
C.2 Equipment	24
C.2.1 Heating arrangement	24
C.2.2 Test vessels	24
C.2.3 Reagents.....	24
C.3 Test procedure.....	24
C.3.1 Sample conditioning and preparation	24
C.3.2 Ageing procedure	25
C.4 Determination of the oxidized insulating liquid.....	25
C.4.1 Soluble acidity	25
C.4.2 Dielectric dissipation factor (DDF) at 90 °C.....	25
C.4.3 Appearance	25
C.4.4 Kinematic viscosity	25
C.5 Report.....	25
C.6 Precision.....	26
Bibliography.....	27
Figure 1 – Typical 8 hole (4 x 2) aluminium heating block	17
Figure 2 – Aluminium alloy temperature measuring block.....	17
Figure 3 – Position of the tube in the oil bath	18
Figure 4 – Oxidation tube or absorption tube	18
Figure 5 – Oxidation tube and absorption tube assembly	19
Figure C.1 – Headspace vial with copper catalyst	25
Table 1 – Repeatability and reproducibility of the oxidation stability test of uninhibited mineral oil in the delivered state for 164 h at 120 °C.....	16
Table A.1 – Thermometer specifications	20
Table B.1 – Precision data for induction time with oxygen for the oxidation test for mineral oil according to Annex B.....	23

Table C.1 – Precision data for headspace procedure according to Annex C26

INTERNATIONAL ELECTROTECHNICAL COMMISSION

INSULATING LIQUIDS – TEST METHODS FOR OXIDATION STABILITY**Test method for evaluating the oxidation stability of insulating liquids in the delivered state**

FOREWORD

- 1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
- 2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
- 3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
- 4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
- 5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
- 6) All users should ensure that they have the latest edition of this publication.
- 7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
- 8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
- 9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

International Standard IEC 61125 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications.

This second edition cancels and replaces the first edition published in 1992 and Amendment 1:2004. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

- a) the title has been modified to include insulating liquids different from mineral insulating oils (hydrocarbon);
- b) the method applies for insulating liquids in the delivered state;
- c) former Method C is now the main normative method;
- d) precision data of the main normative method has been updated concerning the dissipation factor;

- e) former Method A has been deleted;
- f) former Method B has been transferred to Annex B;
- g) a new method evaluating the thermo-oxidative behaviour of esters is included in Annex C.

The text of this standard is based on the following documents:

FDIS	Report on voting
10/1047/FDIS	10/1052/RVD

Full information on the voting for the approval of this International Standard can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under "<http://webstore.iec.ch>" in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INSULATING LIQUIDS – TEST METHODS FOR OXIDATION STABILITY

Test method for evaluating the oxidation stability of insulating liquids in the delivered state

1 Scope

This document describes a test method for evaluating the oxidation stability of insulating liquids in the delivered state under accelerated conditions regardless of whether or not antioxidant additives are present. The duration of the test can be different depending on the insulating liquid type and is defined in the corresponding standards (e.g. in IEC 60296, IEC 61099, IEC 62770). The method can be used for measuring the induction period, the test being continued until the volatile acidity significantly exceeds 0,10 mg KOH/g in the case of mineral oils. This value can be significantly higher in the case of ester liquids.

The insulating liquid sample is maintained at 120 °C in the presence of a solid copper catalyst whilst bubbling air at a constant flow. The degree of oxidation stability is estimated by measurement of volatile acidity, soluble acidity, sludge, dielectric dissipation factor, or from the time to develop a given amount of volatile acidity (induction period with air).

In informative Annex B, a test method for evaluating the oxidation stability of inhibited mineral insulating oils in the delivered state by measurement of the induction period with oxygen is described. The method is only intended for quality control purposes. The results do not necessarily provide information on the performance in service. The oil sample is maintained at 120 °C in the presence of a solid copper catalyst whilst bubbling through a constant flow of oxygen. The degree of oxidation stability is estimated by the time taken by the oil to develop a determined amount of volatile acidity (induction period with oxygen). Additional criteria such as soluble and volatile acidities, sludge and dielectric dissipation factor can also be determined after a specified duration.

In informative Annex C, a test method intended to simulate the thermo-oxidative behaviour of ester insulating liquids (headspace of air at 150 °C for 164 h) is described.

Additional test methods such as those described in IEC TR 62036 based on differential scanning calorimetry can also be used as screening tests, but are out of the scope of this document.

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.

IEC 60247, *Insulating liquids – Measurement of relative permittivity, dielectric dissipation factor ($\tan \delta$) and d.c. resistivity*

IEC 62021-2, *Insulating liquids – Determination of acidity – Part 2: Colorimetric titration*

IEC 62021-3, *Insulating liquids – Determination of acidity – Part 3: Test methods for non-mineral insulating oils*