PD IEC TR 63025:2021

BSI Standards Publication

Insulating liquids — Quantitative determination of methanol and ethanol in insulating liquids

Documents
Document Sharing Hub

Full document details: PD IEC TR 63025:2021

National foreword

This Published Document is the UK implementation of IEC TR 63025:2021.

The UK participation in its preparation was entrusted to Technical Committee GEL/10, Fluids for electrotechnical applications.

A list of organizations represented on this committee can be obtained on request to its committee manager.

Contractual and legal considerations

This publication has been prepared in good faith, however no representation, warranty, assurance or undertaking (express or implied) is or will be made, and no responsibility or liability is or will be accepted by BSI in relation to the adequacy, accuracy, completeness or reasonableness of this publication. All and any such responsibility and liability is expressly disclaimed to the full extent permitted by the law.

This publication is provided as is, and is to be used at the recipient's own risk.

The recipient is advised to consider seeking professional guidance with respect to its use of this publication.

This publication is not intended to constitute a contract. Users are responsible for its correct application.

This publication is not to be regarded as a British Standard.

© The British Standards Institution 2021
Published by BSI Standards Limited 2021

ISBN 978 0 580 97284 3

ICS 29.040.10

Compliance with a Published Document cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 31 August 2021.

Amendments/corrigenda issued since publication

Date	Text affected

Documents
Document Sharing Hub

Full document details: PD IEC TR 63025:2021

IEC TR 63025

Edition 1.0 2021-07

TECHNICAL REPORT

colour inside

Insulating liquids – Quantitative determination of methanol and ethanol in insulating liquids

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 29.040.10

ISBN 978-2-8322-9990-6

Warning! Make sure that you obtained this publication from an authorized distributor.

® Registered trademark of the International Electrotechnical Commission

Documents
Document Sharing Hub

Full document details: PD IEC TR 63025:2021

CONTENTS

FOREWORD 4
INTRODUCTION 6
1 Scope 8
2 Normative references 8
3 Terms and definitions 8
4 Symbols and abbreviated terms 9
5 Sampling 10
6 Principle of the methods 10
7 Method A – HS-GC-MS 10
7.1 General 10
7.2 Apparatus 10
7.2.1 Analytical balance 10
7.2.2 Headspace sampler 10
7.2.3 Gas chromatograph coupled with mass spectrometry detector 11
7.3 Reagents and materials 11
7.3.1 Laboratory equipment and glassware 11
7.3.2 Standard chemicals 11
7.3.3 GC carrier gases 12
7.4 Preparation of standard solutions 12
7.4.1 General 12
7.4.2 Degassed insulating liquid 12
7.4.3 Internal standard stock solution 12
7.4.4 Standard solutions of methanol and ethanol 13
7.5 Sample preparation 14
7.6 Headspace sampler parameters 15
7.7 Gas chromatograph parameters 15
7.7.1 Injector 15
7.7.2 Carrier gas 15
7.7.3 Temperature ramp 15
7.8 Mass spectrometer parameters 16
7.9 Analysis procedure 16
7.10 Internal standard calibration 17
7.10.1 General 17
7.10.2 Response factor determination 18
7.11 Expression of the results 18
8 Method B – HS-GC-FID 18
8.1 General 18
8.2 Apparatus 18
8.2.1 Analytical balance 18
8.2.2 Headspace sampler 18
8.2.3 Gas chromatograph with flame ionization detector 19
8.3 Reagents and materials 19
8.3.1 Laboratory equipment and glassware 19
8.3.2 Standard chemicals 19
8.3.3 GC carrier gases 19
8.4 Preparation of standard solutions 20

AVA Documents
Document Sharing Hub

8.4.1 General 20
8.4.2 Degassed insulating liquid 20
8.4.3 Standard solutions of methanol and ethanol 20
8.5 Sample preparation 21
8.6 Headspace sampler parameters 21
8.7 Gas chromatograph parameters 22
8.7.1 Injector 22
8.7.2 Carrier gas 22
8.7.3 Temperature ramp 22
8.7.4 FID parameters 22
8.8 Analysis procedure 22
8.9 Calibration 23
8.10 Expression of the results 23
9 Test report 23
10 Precision 24
10.1 Verification of the entire analytical system 24
10.2 General 24
10.3 Detection limits of Method A and Method B 24
10.4 Repeatability 24
10.5 Reproducibility 25
Bibliography 26

Figure 1 – Comparison of methanol and 2-furfural production in mineral oil versus cellulose scission number 7
Figure 2 –Typical chromatogram with selected ion (m/z = 31) mass spectrum 17
Figure 3 – Typical GC-FID chromatogram 23

Table 1 –Method A – Example of GC temperature ramp parameters 16
Table 2 – Method A – m/z values of internal standard ions 16
Table 3 – Method B – Examples of FID parameters reported in literature 22
Table 4 – Detection limits of Method A and Method B, in mineral oil 24
Table 5 – Repeatability (r) in % for Method A (HS-GC-MS), in mineral oil 24
Table 6 – Reproducibility (R) in % for Method A (HS-GC-MS), in mineral oil 25

Documents
Document Sharing Hub
Full document details: PD IEC TR 63025:2021

INTERNATIONAL ELECTROTECHNICAL COMMISSION

INSULATING LIQUIDS – QUANTITATIVE DETERMINATION OF METHANOL AND ETHANOL IN INSULATING LIQUIDS

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.

IEC TR 63025 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications. It is a Technical Report.

The text of this Technical Report is based on the following documents:

Draft	Report on voting
10/1112/DTR	10/1131/RVDTR

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Report is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

Documents
Document Sharing Hub

Full document details: PD IEC TR 63025:2021

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under 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 document 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.

Documents
Document Sharing Hub

Full document details: PD IEC TR 63025:2021

INTRODUCTION

It has been demonstrated over several years that the ageing of impregnated paper in insulating liquid, which results in cellulose degradation, produces molecules of light alcohols, methanol (MeOH) and ethanol (EtOH). In laboratory experiments, a good correlation has been established between the increase of the methanol content in insulating liquid and the decrease of the degree of polymerization of the cellulose, irrespective of the type of paper, standard kraft or thermally upgraded. Further, at the early stages of paper ageing, i.e. of cellulose degradation, the methanol content is always higher than that of furanic compounds (mainly 2-furfural), so this behaviour suggests that methanol could be a relevant in-oil marker to detect early paper ageing in transformers and to assess its evolution (see Figure 1).

Ethanol is a second light alcohol of interest that these methods would be able to detect.

It should be emphasized that in a real transformer the situation is much more complicated than in laboratory setups, so the relationship between in situ paper degradation and tracer concentration (MeOH, EtOH, as well as 2-FAL) is much more complex and hard to establish.

In order to address the growing interest of industry in using these alcohols as tracers of cellulosic material ageing in operating equipment, there is a need for the development of a document describing analytical methods to quantify methanol and ethanol in the different types of insulating liquids. The objective is for one of these methods to remain as simple and affordable as possible, and for the other to be more sophisticated and more accurate.

The principle of this Technical Report was brought up and discussed during the IEC TC 10 plenary meeting held in Vienna in November 2013. A project team was set up to prepare test methods for the unambiguous quantitative determination of methanol and ethanol in unused and used insulating liquids.

WARNING – Health and safety

This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

The insulating liquids which are the subject of this document should be handled with due regard to personal hygiene. Direct contact with eyes may cause slight irritation. In the case of eye contact, irrigation with copious quantities of clean running water should be carried out and medical advice sought.

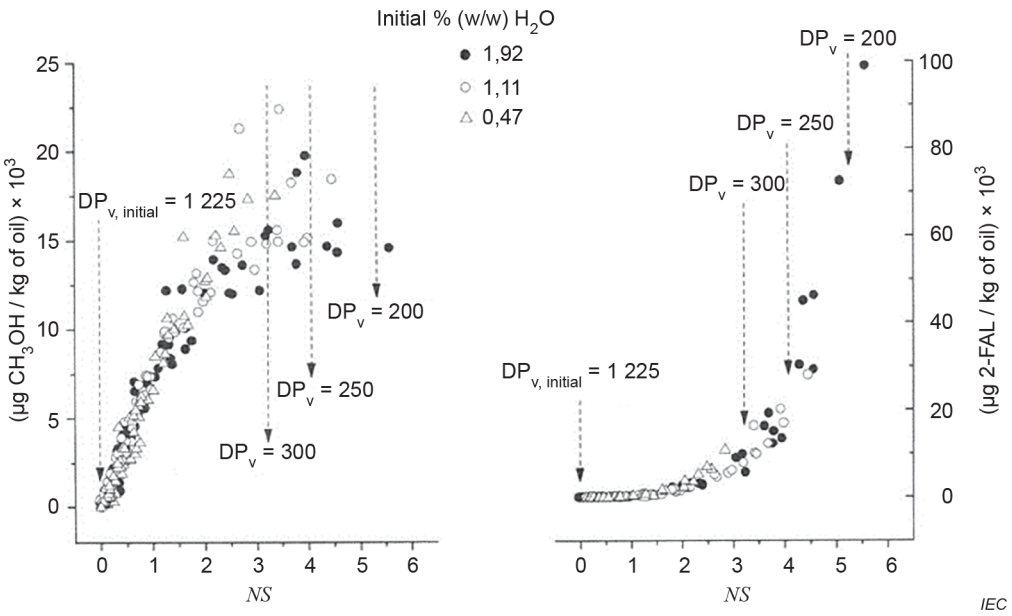
Some of the tests specified in this document involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

WARNING – Environment

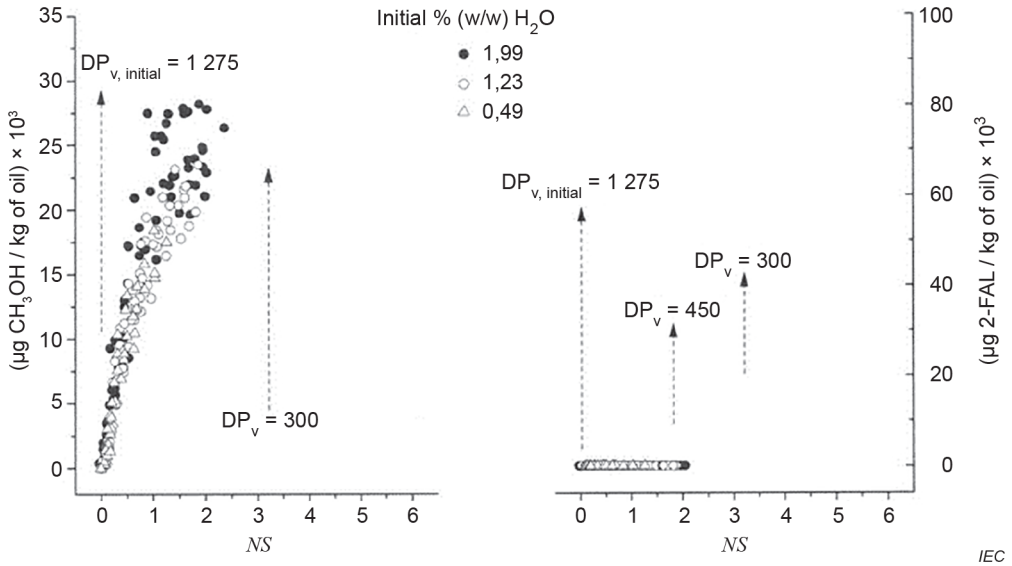
This document involves mineral oils, ester liquids, chemicals and used sample containers. The disposal of these items should be carried out in accordance with current national legislation with regard to their impacts on the environment. Every precaution should be taken to prevent the release into the environment of the chemicals used during the test.

Documents
Document Sharing Hub

Full document details: PD IEC TR 63025:2021



a) Clupak HD75 specimens



b) Manning 220 mannitherm D specimens

Key

NS: number of scissions, inversely proportional to the polymerization degree (DPv)

a): standard kraft paper

b): thermally upgraded paper

NOTE See Jalbert J., Gilbert R., Tétreault P., Morin B. and Lessard-Déziel D. (2007) in the Bibliography.

Figure 1 – Comparison of methanol and 2-furfural production in mineral oil versus cellulose scission number

Documents
Document Sharing Hub
Full document details: PD IEC TR 63025:2021