



BSI Standards Publication

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

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## National foreword

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

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# TECHNICAL REPORT



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

INTERNATIONAL  
ELECTROTECHNICAL  
COMMISSION

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## 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 |

|       |   |    |
|-------|---|----|
| 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 |

## INTERNATIONAL ELECTROTECHNICAL COMMISSION

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

## FOREWORD

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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](http://www.iec.ch/members_experts/refdocs). The main document types developed by IEC are described in greater detail at [www.iec.ch/standardsdev/publications](http://www.iec.ch/standardsdev/publications).

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- withdrawn,
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- amended.

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## 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**

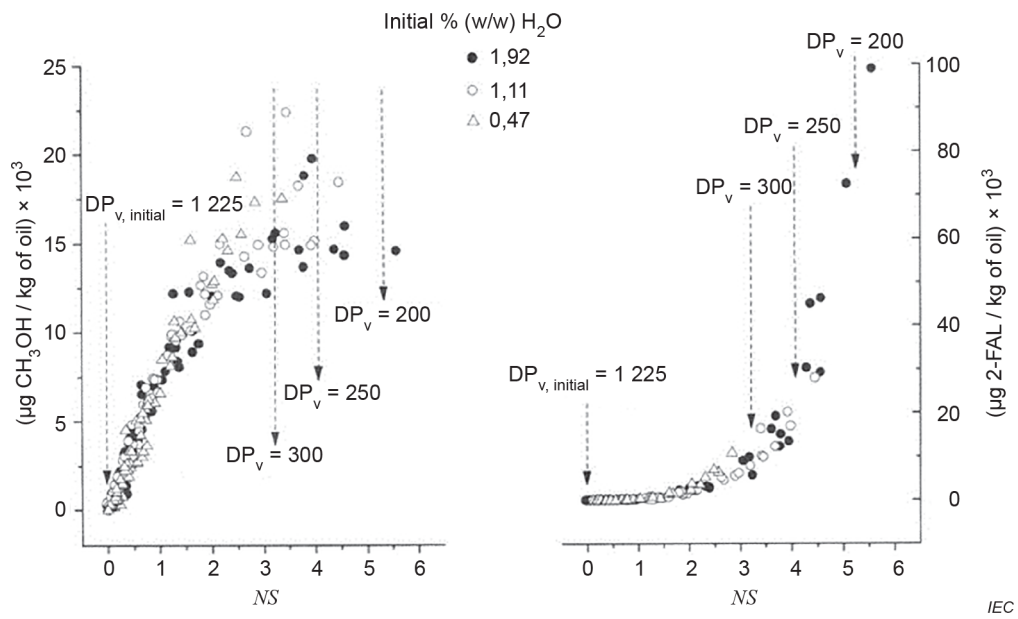
This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

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.

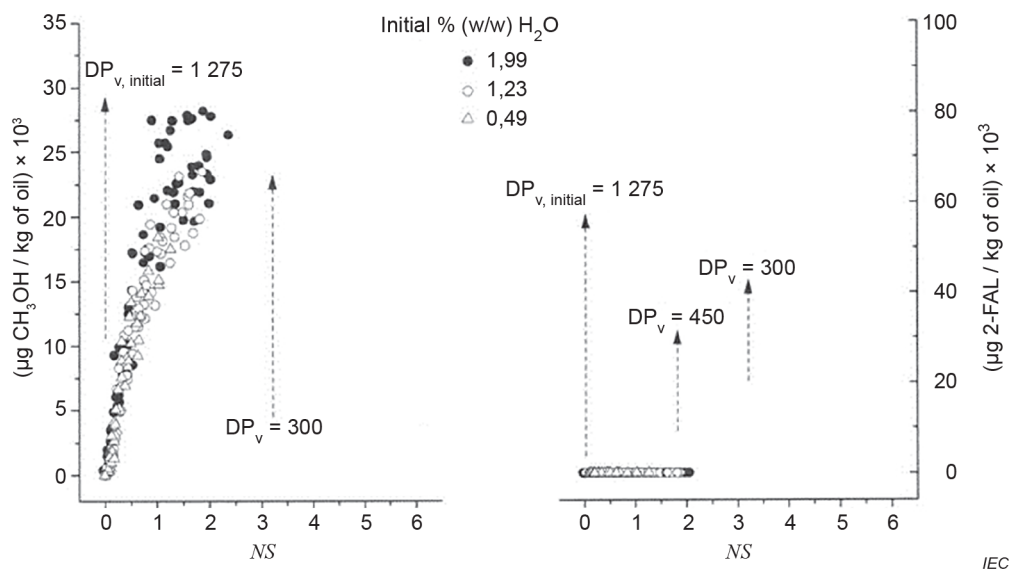
Some of the tests specified in this document involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

### **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.



**a) Clupak HD75 specimens**



**b) Manning 220 mannitherm D specimens**

**Key**

*NS*: number of scissions, inversely proportional to the polymerization degree ( $DP_v$ )

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**

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

## 1 Scope

This document specifies two test methods for methanol and ethanol determination in insulating liquids.

Methanol (MeOH) and ethanol (EtOH) are two light alcohols generated during the degradation process of cellulosic materials. They are soluble in insulating liquids so they can be regarded as ageing tracers whose concentrations in oil reflect the degradation of insulating cellulosic materials in liquid-impregnated transformers.

## 2 Normative references

There are no normative references in this document.

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

### 3.1

#### **flame ionization detector**

device in which hydrocarbons are burned in a hydrogen-air flame and the electrical current caused by the resulting ions is measured between two electrodes

Note 1 to entry: The flame ionization detector is used in gas chromatography mainly to detect hydrocarbon compounds.

[SOURCE: ISO 14532:2014, 2.4.8, modified – "detector" replaced with "device".]

### 3.2

#### **gas chromatograph**

#### **GC**

device used to determine complex mixture components that can be vaporized without decomposition then separated by differential migration with a carrier gas through a stationary phase in a column

Note 1 to entry: The method used is called "gas chromatography" (GC).

[SOURCE: IEC 62697-1:2012, 3.1.14, modified – "used for separating volatile and semi-volatile compounds in mixtures" replaced with "used to determine complex mixture components", "through differential migration" replaced with "then separated by differential migration" and "a stationary phase" and Note 1 to entry added.]

### 3.3

#### **headspace extraction**

procedure for collecting the volatile compounds emitted by a specimen enclosed in an airtight vial under controlled conditions