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INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

Test methods for quantitative determination of corrosive sulfur compounds in unused and used insulating liquids – Part 1: Test method for quantitative determination of dibenzyldisulfide (DBDS)

Méthodes d'essai pour la détermination quantitative des composés de soufre corrosif dans les liquides isolants usagés et neufs – Partie 1: Méthode d'essai pour la détermination quantitative du disulfure de dibenzyle (DBDS)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

TEST METHODS FOR QUANTITATIVE DETERMINATION OF CORROSIVE SULFUR COMPOUNDS IN UNUSED AND USED INSULATING LIQUIDS –

Part 1: Test method for quantitative determination of dibenzyldisulfide (DBDS)

FOREWORD

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

FDIS	Report on voting
10/887/FDIS	10/891/RVD

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

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

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

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INTRODUCTION

Sulfur can be present in insulating liquids in various forms, including elemental sulfur, inorganic sulfur compounds and organic sulfur compounds. The number of diverse sulfur species comprised of different isomers and homologous can run into hundreds. The total sulfur(TS) concentration in insulating liquids depends on the origin of the liquid, refining processes and the degree of refining and formulation including addition of additives to the base oils. Base oils include mineral based paraffinic and naphthenic oils, synthetic iso-paraffins obtained through gas to liquid conversion process (GTL-Fischer-Tropsch), esters, poly alpha olefins, poly alkylene glycols, etc. Additives can be comprised of electrostatic discharge depressants, metal deactivators, metal passivators, phenolic and sulfur containing antioxidants such as the polysulfides, disulfides, dibenzyl disulfide (DBDS), etc.

Certain sulfur compounds present in the insulating liquids exhibit antioxidant and metal deactivating properties without being corrosive, whereas other sulfur compounds have been known to react with metal surfaces. Specifically, sulfur compounds such as mercaptans are very corrosive to metallic components of electrical devices. Presence of these corrosive sulfur species has been linked to failures of electrical equipment used in generation, transmission and distribution of electrical energy for several decades. Therefore, the IEC standard for mineral insulating oils states that corrosive sulfur compounds shall not be present in unused and used insulating liquids (see IEC 60296) [5][1].

Recently, the serious detrimental impact of corrosive sulfur has been linked to the presence of a specific highly corrosive sulfur compound, DBDS. This compound has been found in certain mineral insulating oils [1, 14, 15, 16]; presence of this compound has been shown to result in copper sulfide formation on the surfaces of copper conductors under normal operating conditions of transformers [2].

Current standard test methods for detection of corrosive sulfur (ASTM D1275, methods A and B, and DIN 51353) and potentially corrosive sulfur in used and unused insulating oil (IEC 62535) are empirical and qualitative. These methods rely on visual and subjective perception of colour profiles. The methods do not yield quantitative results in regard to the concentration of DBDS or other corrosive sulfur compounds present in insulating liquids.

Furthermore, methods for corrosive sulfur and potentially corrosive sulfur in insulating liquids (ASTM D1275, method B and IEC 62535) are applicable only to mineral insulating oils that do not contain a metal passivator additive, the methods otherwise can yield negative results even when corrosive sulfur compounds are present in the insulating liquids – thus providing a false negative test result. On the other hand, the test method when used with aged insulating oils (e.g. those with relative high acidity), may give ambiguous results and lead to a false positive test result. Further analysis of insulating liquids is stipulated, e.g. IEC 62535 specifies that if there are any doubts in the interpretation of the results of inspection of paper, the composition of precipitate should be analyzed by other methods (for example by SEM-EDX).

For this reason, IEC TC 10 WG 37 was set up to prepare test methods for the unambiguous quantitative determination of corrosive sulfur compounds in unused and used insulating liquids. Because of the complexity of such determinations, the test methods are divided into three parts:

Part 1 – Test method for quantitative determination of dibenzyldisulfide (DBDS).

Part 2 – Test methods for quantitative determination of total corrosive sulfur (TCS).

Part 3 – Test methods for quantitative determination of total mercaptans and disulfides (TMD) and other targeted corrosive sulfur species.

[1] Figures in square brackets refer to the bibliography.

Health and safety

This part of IEC 62697 does not purport to address all the safety problems associated with its use. It is the responsibility of the user of the standard 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 standard 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 standard involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

Environment

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

TEST METHODS FOR QUANTITATIVE DETERMINATION OF CORROSIVE SULFUR COMPOUNDS IN UNUSED AND USED INSULATING LIQUIDS –

Part 1: Test method for quantitative determination of dibenzyldisulfide (DBDS)

1 Scope

This part of IEC 62697 specifies a test method for the quantitative determination of corrosive sulfur compounds-dibenzyl disulfide (DBDS) in used and unused insulating liquids over a 5 – 600 mg kg^{-1} concentration range.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60475, *Method of sampling liquid dielectrics*

IEC 62535:2008, *Insulating liquids – Test method for detection of potentially corrosive sulfur in used and unused insulating oil*

3 Terms, definitions and abbreviations

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

3.1 Terms and definitions

3.1.1
accuracy
closeness of agreement between test result and the accepted reference value

3.1.2
additive
a suitable chemical substance that is deliberately added to insulating liquid in order to improve certain characteristics

Note 1 to entry: Examples include antioxidants, pour-point depressants, electrostatic charging tendency depressant such as benzotriazol (BTA) metal passivator or deactivators, antifoam agent, refining process improver, etc.

3.1.3
atomic emission detector
AED
simultaneously monitors emissions of radiation resulting from atomic species excited in a microwave-induced plasma and permits quantitative determination of selected heteroatoms in compounds that elute from a GC column

Note 1 to entry: AED thus provides heteroatom profiles, i.e. “fingerprints” of complex samples such as insulating liquids.