IEC 60296

Edition 5.0 2020-06

INTERNATIONAL STANDARD

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Fluids for electrotechnical applications – Mineral insulating oils for electrical equipment

Fluides pour applications électrotechniques – Huiles minérales isolantes pour matériel électrique

IEC 60296:2020-06(en-fr)

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

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

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Fluids for electrotechnical applications – Mineral insulating oils for electrical equipment

Fluides pour applications électrotechniques – Huiles minérales isolantes pour matériel électrique

INTERNATIONAL
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ICS 29.040.10

ISBN 978-2-8322-8377-6

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FLUIDS FOR ELECTROTECHNICAL APPLICATIONS – MINERAL INSULATING OILS FOR ELECTRICAL EQUIPMENT

FOREWORD

International Standard IEC 60296 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications.

This fifth edition cancels and replaces the fourth edition published in 2012. This edition constitutes a technical revision.

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

– This International Standard is applicable to specifications and test methods for unused and recycled mineral insulating oils in the delivered state.

– Within the transformer insulating oils, two groups, Type A and Type B, are defined, based on their performance.

– A new method for stray gassing under thermo-oxidative stress of mineral insulating oils, which has been tested in a joint round robin test (RRT) between CIGRE D1 and IEC technical committee 10, has been included.

The text of this International Standard is based on the following documents:

FDIS	Report on voting
10/1117/FDIS	10/1118/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 web site 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- withdrawn,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.

INTRODUCTION

WARNING – 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 mineral insulating oils which are the subject of this document should be handled in compliance with local regulations and suppliers safety data-sheets.

This document is applicable to mineral insulating oils, chemicals and used sample containers. The disposal of these items should be carried out according to local regulations with regard to their impact on the environment.

FLUIDS FOR ELECTROTECHNICAL APPLICATIONS – MINERAL INSULATING OILS FOR ELECTRICAL EQUIPMENT

1 Scope

This document provides specifications and test methods for unused and recycled mineral insulating oils (see Clause 3 for definitions). It applies to mineral oil delivered according to the contractual agreement, intended for use in transformers, switchgear and similar electrical equipment in which oil is required for insulation and heat transfer. Both unused oil and recycled oil under the scope of this document have not been used in, nor been in contact with electrical equipment or other equipment not required for manufacture, storage or transport.

Unused oils are obtained by refining, modifying and/or blending of petroleum products and other hydrocarbons from virgin feedstock.

Recycled oils are produced from oils previously used as mineral insulating oils in electrical equipment that have been subjected to re-refining or reclaiming (regeneration) by processes employed offsite. Such oils will have originally been supplied in compliance with a recognized unused mineral insulating oil specification. This document does not differentiate between the methods used to recycle mineral insulating oil. Oils treated on-site (see IEC 60422) are not within the scope of this document.

Oils with and without additives are both within the scope of this document.

This document does not apply to mineral insulating oils used as impregnating medium in cables or capacitors.

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 60156, *Insulating liquids – Determination of the breakdown voltage at power frequency – Test method*

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

IEC 60422:2013, *Mineral insulating oils in electrical equipment – Supervision and maintenance guidance*

IEC 60475, *Method of sampling liquid dielectrics*

IEC 60567:2011, *Oil-filled electrical equipment – Sampling of gases and analysis of free and dissolved gases – Guidance*

IEC 60628:1985, *Gassing of insulating liquids under electrical stress and ionization*

IEC 60666:2010, *Detection and determination of specified additives in mineral insulating oils*

IEC 60814, *Insulating liquids – Oil-impregnated paper and pressboard – Determination of water by automatic coulometric Karl Fischer titration*

IEC 60970*, Insulating liquids – Methods for counting and sizing particles*

IEC 61125:2018, *Insulating liquids – Test methods for oxidation stability – Test method for evaluating the oxidation stability of insulating liquids in the delivered state*

IEC 61198, *Mineral insulating oils – Methods for the determination of 2-furfural and related compounds*

IEC 61619, *Insulating liquids – Contamination by polychlorinated biphenyls (PCBs) – Method of determination by capillary column gas chromatography*

IEC 61620, *Insulating liquids – Determination of the dielectric dissipation factor by measurement of the conductance and capacitance – Test method*

IEC 61868, *Mineral insulating oils – Determination of kinematic viscosity at very low temperatures*

IEC 62021-1*, Insulating liquids – Determination of acidity – Part 1: Automatic potentiometric titration*

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

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

IEC 62697-1, *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)*

IEC 62961, *Insulating liquids – Test methods for the determination of interfacial tension of insulating liquids – Determination with the ring method*

ISO 2049, *Petroleum products, Determination of colour (ASTM scale)*

ISO 2719, *Determination of flash point – Pensky-Martens closed cup method*

ISO 3016, *Petroleum and related products from natural or synthetic sources – Determination of pour point*

ISO 3104, *Petroleum products – Transparent and opaque liquids – Determination of kinematic viscosity and calculation of dynamic viscosity*

ISO 3675, *Crude petroleum and liquid petroleum products – Laboratory determination of density – Hydrometer method*

ISO 3819, *Laboratory glassware –Beakers*

ISO 8754, *Petroleum products – Determination of sulphur content – Energy-dispersive X-ray fluorescence spectrometry*

ISO 12185, *Crude petroleum and petroleum products – Determination of density – Oscillating U-tube method*

ISO 14596, *Petroleum products – Determination of sulphur content – Wavelength-dispersive X-ray fluorescence spectrometry*

ASTM D971, *Standard Test Method for Interfacial Tension of Oil Against Water by the Ring Method*

ASTM D1500, *Standard Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)*

ASTM D6591, *Standard Test Method for Determination of Aromatic Hydrocarbon Types in Middle Distillates – High Performance Liquid Chromatography Method with Refractive Index Detection*

ASTM D7042, *Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)*

ASTM D7896, *Standard Test Method for Thermal Conductivity, Thermal Diffusivity and Volumetric Heat Capacity of Engine Coolants and Related Fluids by Transient Hot Wire Liquid Thermal Conductivity Method*

DIN 51353, *Testing of insulating oils; detection of corrosive sulphur; Silver strip test*

IP 346, *Determination of polycyclic aromatics in unused lubricating base oils and asphaltene free petroleum fractions – Dimethyl sulfoxide extraction refractive index method*

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
mineral insulating oil
insulating liquid for transformers and similar electrical equipment (e.g. switchgear, tap-changers), derived from petroleum products and/or other hydrocarbons

Note 1 to entry: Mineral insulating oils include unused (3.8) and recycled (3.9) mineral insulating oils.

[SOURCE: IEC 60050-212:2010, 212-17-02, modified – "for transformers and similar electrical equipment, (e.g. switchgear, tap-changers)" added, "crudes" replaced with "petroleum products and/or other hydrocarbons" and note to entry added.]

3.2
low temperature switchgear oil
mineral insulating oil for oil-filled switchgear for outdoor applications in very cold climatic conditions

3.3
additive
chemical substance that is added to mineral insulating oil in order to improve certain characteristics

EXAMPLES Antioxidants, metal passivators, metal deactivators, electrostatic charging tendency depressants, gassing tendency modifier, pour point depressants, anti-foam agents and refining process improvers.

[SOURCE: IEC 60050-212:2010, 212-17-13, modified – "specific" replaced with "chemical", "an insulating material or liquid in small proportion" replaced with "mineral insulating oil" and examples added.]

3.4
antioxidant
oxidation inhibitor
additive incorporated in mineral insulating oil that improves oxidation stability

Note 1 to entry: DBPC = 2,6-di-tert-butyl-para-cresol; DBP = 2,6-di-tert-butyl-phenol.

Note 2 to entry: For the purposes of this document, the oxidation inhibitor is a synthetic chemical substance of the phenolic type, such as DBPC and DBP described in IEC 60666.

[SOURCE: IEC 60050-212:2010, 212-17-14, modified – "an insulating material to reduce or delay degradation by oxidation" replaced with "mineral insulating oil that improves oxidation stability" and note replaced with notes to entry.]

3.4.1
other antioxidant additive
antioxidant additive of the sulphur-, amine- or phosphorous- type

Note 1 to entry: Sulphur-type additives do not include dibenzyldisulphide (DBDS) or other potentially corrosive sulphur compounds.

3.4.2
passivator
additive used primarily as corrosion deactivator and sometimes as electrostatic charging depressant

Note 1 to entry: It can also improve the oxidation stability, by reducing the catalytic effect of copper on oxidation of the oil

3.5
uninhibited oil (U)
mineral insulating oil containing no oxidation inhibitor or other antioxidant additives

Note 1 to entry: No inhibitor means that the total inhibitor content is below the detection limit of 0,01 % indicated in IEC 60666.

[SOURCE: IEC 60050-212:2010, 212-17-19, modified – In the term, deletion of "insulating", in the definition "antioxidant, but which may contain other additives" replaced with "oxidation inhibitor or other antioxidant additives" and note replaced with the note to entry.]

3.6
trace inhibited oil (T)
mineral insulating oil containing minimum 0,01 % and less than 0,08 % of total inhibitor content as measured in IEC 60666

3.7
inhibited oil (I)
mineral insulating oil containing a minimum of 0,08 % and a maximum of 0,40 % of total inhibitor content as measured in IEC 60666

3.8
unused mineral insulating oil (V)
mineral insulating oil, obtained by refining, modifying and/or blending of petroleum products and other hydrocarbons and that has not been used in, nor been in contact with electrical equipment or other equipment not required for manufacture, storage or transport

Note 1 to entry: In some countries unused mineral oil is described as virgin oil.

Note 2 to entry: The manufacturer and supplier of unused mineral insulating oil shall take reasonable precautions to ensure that there is no contamination with polychlorinated biphenyls or terphenyls (PCB, PCT) or other contaminants.

3.9
recycled mineral insulating oil (R)
mineral insulating oil previously used in electrical equipment that has been subjected to re-refining or reclaiming (regeneration) after removal from the electrical equipment

Note 1 to entry: Any blend of unused and recycled oils is to be considered as recycled.

Note 2 to entry: The characteristics of recycled oil are heavily dependent on the oil from which it was recycled, the original refining technique, the service history and the type of recycling process.

Note 3 to entry: Natural or added antioxidants originally present in the oil might have been depleted in service or removed by the recycling process. The oxidation stability therefore needs to be restored/improved and is usually achieved by the addition of an oxidation inhibitor.

Note 4 to entry: Such recycled oils are often produced from mixtures of mineral insulating oils of different origins. The manufacturer and supplier of recycled mineral insulating oil shall take reasonable precautions to ensure that there is no contamination with polychlorinated biphenyls or terphenyls (PCB, PCT) or other contaminants.

3.10
reclaimed mineral insulating oil
regenerated mineral insulating oil
recycled mineral insulating oil used in electrical equipment, which has been subjected after removal from the electrical equipment to chemical and physical processing to reduce soluble and insoluble contaminants

3.11
re-refined mineral insulating oil
recycled mineral insulating oil used in electrical equipment that has been removed from service and subjected to a process similar to that used for the production of unused mineral insulating oil from virgin feedstock, in order to reduce the level of undesired compounds

Note 1 to entry: Such re-refined oils are often produced from mixtures of mineral insulating oils of different origins including processes such as distillation and hydrogenation.

4 Properties of oil

4.1 General

Oil characteristics are listed in Table 2, Table 3 and Table 4 and in Clause 6 and Clause 7.

4.2 Functional properties

These are properties of oil that have an impact on its function as a liquid for insulation and heat transfer.

NOTE Functional properties include viscosity, density, pour point, water content, breakdown voltage, dielectric dissipation factor, as well as specific heat capacity, thermal conductivity and expansion coefficient.

4.3 Production and stability

These are properties of oil that are influenced by the quality and type of refining and additives.

NOTE 1 These can include appearance, interfacial tension, sulphur content, acidity, corrosive sulphur, potentially corrosive sulphur, 2-furfural and related compounds content and stray gassing.

NOTE 2 Properties like aromatic content, refractive index or/and distribution of aromatic type of compounds can provide valuable information on consistency of a certain oil product.

4.4 Performance

These are properties that are related to the long-term behaviour of oil in service and/or its reaction to high electrical or thermal stresses. In terms of performance, transformer insulating oils are divided into Type A (Table 3) and Type B (Table 4).

4.5 Health, safety and environment (HSE) properties

These are oil properties related to safe handling and environmental protection.

NOTE Examples can include flash point, density, PCA (polycyclic aromatics) and PCB/PCT (polychlorinated biphenyls/ terphenyls) content.

5 Classification, labelling, identification, general delivery requirements and sampling

5.1 Classification and labelling

5.1.1 Classes

For the purposes of this document, mineral insulating oils are classified into two classes:

– transformer oils;
– low temperature switchgear oils.

Within the transformer oils two groups of oils are defined: Type A (Table 3) and Type B (Table 4).

Type A insulating oils are fully inhibited ("I" according to 5.1.2) and deliver higher oxidation stability than Type B.

Type B insulating oils can be uninhibited ("U"), trace inhibited ("T") or fully inhibited ("I") and deliver good resistance to oil degradation and provide good oxidation stability.

Inhibitor concentration for inhibited oil in service needs to be monitored and eventually maintained. This is described in IEC 60422.

NOTE During base oil refining some components such as aromatic and polycyclic aromatic compounds are removed depending on the severity and type of refining process.

Uninhibited oils are typically made from base oil(s) with the aim to retain a balance of removable components, some of which are easily oxidized, while others provide some protection against the normal oxidation process. The refining process is optimized to retain certain sulphur and aromatic compounds which act as natural antioxidants. However, since the natural antioxidants are not as effective as synthetic antioxidants, the uninhibited oils will exhibit less oxidative stability compared to inhibited oils.

Uninhibited oil contains a certain amount of so called natural antioxidants, some of them present from the beginning (mostly sulphur-containing acting as secondary antioxidants), others being formed as intermediates by oxidative processes (mostly oxidation of aromatic compounds then acting as radical scavengers). Inhibited oil is a blend of base oil(s) with a synthetic antioxidant. The additive response and the resulting oxidation stability of the inhibited oil depends very much on the refining severity. The antioxidant is added to control the oxidation processes. The inhibitor acts as radical scavenger and protects the base oil hydrocarbons – depending on the degree of refining – from oxidation. Oils with very high oxidative stability are inhibited oils and can be achieved by blending very severely treated base oil and antioxidant.

5.1.2 Antioxidant (oxidation inhibitor) content

Mineral insulating oils are classified into three groups, according to the content of antioxidant additive:

– uninhibited mineral insulating oils: marked with U;
– trace inhibited mineral insulating oils: marked with T;

– inhibited mineral insulating oils: marked with I.

5.1.3 Lowest cold start energizing temperature (LCSET)

LCSET shall be −30 °C unless otherwise specified. If a different LCSET is specified it shall be chosen from values of Table 2.

5.1.4 Labelling and ordering designation

For the purpose of declaration, mineral insulating oils shall be labelled as:

V: unused mineral insulating oil as defined in 3.8.

R: recycled mineral insulating oil as defined in 3.9.

The ordering designation for insulating oil according to IEC 60296 shall follow the order: Equipment/Declaration/Type/Antioxidant according to the scheme in Table 1.

Table 1 – Meaning of the identifying letter codes in the ordering designation of mineral oil according to IEC 60296

First Letter = Equipment	**T** – Transformer	**S** – Switchgear	
Second Letter = Declaration	**V** – Unused (Virgin)	**R** – Recycled	
Third Letter = Type	**A** – Specification Type A	**B** – Specification Type B	
Fourth Letter = Antioxidant	**I** – inhibited	**U** – uninhibited	**T** – trace inhibited

EXAMPLE 1 For order for inhibited high grade recycled oil for transformers: **TRAI.**

EXAMPLE 2 For order for uninhibited unused oil for transformers: **TVBU.**

EXAMPLE 3 For order for inhibited high grade unused oil for switchgear: **SVAI.**

EXAMPLE 4 For order for trace inhibited recycled oil for switchgear: **SRBT.**

Mineral insulating oils with non-standard specification such as LCSET, pour point etc. shall be declared separately.

5.2 Requirements

General requirements of this document are given in Table 3 and Table 4.

Additional properties are defined under Clause 7.

5.3 Miscibility and compatibility

Mineral oils according to this document are generally considered miscible and compatible if the characteristics of their mixture are not less favourable than those of the worst individual oil.

All mineral insulating oils according to this document are physically miscible with each other and are considered to result, after homogenization, in a single homogeneous phase and without precipitation of insoluble substances, or formation of turbidity. The mixture, however, can show different properties, for example density, viscosity, total sulphur content, oxidation stability or stray gassing from the original oils.

Mineral insulating oils of the same class and type (5.1.1), the same group (5.1.2), same LCSET (5.1.3) and containing the same types of additives are considered to be compatible with each other in mixtures up to 10 % with no need for additional testing.

If oils of different class or type (5.1.1), or group (5.1.2), or LCSET (5.1.3) or type of additives are mixed, the resulting mixture shall be classified and tested according to this document, see Table 3 and Table 4.

A procedure to perform miscibility tests in service and a set of recommended investigations are described in IEC 60422:2013, 5.12.

5.4 Identification and general delivery requirements

Identification and general delivery requirements are as follows:

a) Oil is normally delivered in bulk, rail tank cars, tank containers or packed in drums or intermediate bulk containers (IBC). These shall be clean and suitable for this purpose to avoid any contamination. The supplier shall take all precautions to ensure the delivery product will be in accordance with the requirements of this document.
b) All types of oil containers shall carry at least the following markings:
   - supplier's designation;
   - classification and labelling (see 5.1);
   - oil quantity.
c) As agreed between the supplier and purchaser each oil delivery may be accompanied by a document specifying the supplier’s designation, oil classification and labelling and compliance certificate.
d) The supplier shall declare the chemical family and function of all additives, and the concentrations in the cases of antioxidants and passivators.
e) A delivery shall be traceable to a manufactured batch.

5.5 Sampling

Sampling shall be carried out in accordance with the procedure described in IEC 60475.

6 Properties, their significance and test methods

6.1 Viscosity

Viscosity influences heat transfer and therefore the temperature rise of the equipment. Higher viscosities may impede the flow of oil and reduce the heat transfer performance. At low ambient temperatures, the resulting higher viscosity of oil is a critical factor for the cold start of transformers with poor or no circulation of oil and this may lead to overheating at hot spots in the transformer windings. Higher viscosity at low temperatures may also reduce the speed of moving parts in equipment such as power circuit breakers, switchgear, on-load tap-changer mechanisms, pumps and regulators. The viscosity at the lowest cold start energizing temperature (LCSET) for transformers shall not exceed 1 800 mm^2/s at −30 °C. This lowest cold start energizing temperature (LCSET) for mineral insulating oils is defined in this document as being −30 °C. Other LCSETs (see Table 2) can be agreed between supplier and purchaser.

Low temperature switchgear oil should have a lower viscosity at LCSET: maximum 400 mm^2/s. Standard LCSET of low temperature switchgear oil is defined at –40 °C but other LCSETs may be agreed between supplier and purchaser.

Table 2 – Maximum viscosity and pour point of mineral insulating oil

Application	LCSET °C	Maximum viscosity mm^2/s	Maximum pour point °C
Transformer	0	1 800	−10
Transformer	−20	1 800	−30
Transformer	−30	1 800	−40
Transformer low ambient temperature application	−40	2 500[a]	−50
Low ambient temperature switchgear	−40	400[a]	−60

[a] For low ambient temperature applications. In other cases the value shall be discussed between the user and manufacturer.

Viscosity shall be measured according to ISO 3104 (reference method) or ASTM D7042, and viscosity at −40 °C for low temperature oils according to IEC 61868.

Viscosity should also be taken into account when designing protection level, flow control and switching equipment.

6.2 Pour point

The pour point of mineral insulating oil is the lowest temperature at which the oil will just flow. It is recommended that the pour point should be at least 10 °C below the lowest cold start energizing temperature (LCSET). If a pour point depressant additive is used, this shall be declared by the supplier to the user. Pour point shall be measured in accordance with ISO 3016.

6.3 Water content

A low water content of mineral insulating oil is necessary to achieve adequate breakdown voltage and low dissipation losses. To avoid separation of free water, mineral insulating oil as delivered should have limited water content. Before filling the electrical equipment, the oil shall be treated to meet the requirements of IEC 60422 when inside the transformer. Water content shall be measured in accordance with IEC 60814.

6.4 Breakdown voltage

The breakdown voltage of mineral insulating oil indicates its ability to resist electrical stress in electrical equipment. Breakdown voltage shall be measured in accordance with IEC 60156. The supplier shall demonstrate after treatment to reduce particles, water and dissolved air by a vacuum procedure (see note), that the oil shall have a high dielectric strength (breakdown voltage > 70 kV).

NOTE This treatment referred to consists of filtration of the oil at 60 °C by vacuum (pressure below 2,5 kPa) through a sintered glass filter (with a maximum pore size of 2,5 μm).

6.5 Density

In cold climates, the density of the oil shall be low enough to prevent any ice resulting from the freezing of free water to float and possibly lead to fault conditions developing such as flashover of conductors. Density should also be taken into account when designing protection, flow and control equipment, for example devices which rely on buoyancy principles like the Buchholz relay. Density shall be measured in accordance with ISO 12185 (reference method), ISO 3675 and ASTM D7042 are acceptable.

6.6 Dielectric dissipation factor (DDF)

DDF is a measure for dielectric losses within the oil. DDF values above the requirements of Table 3 and Table 4 can indicate contamination of the oil by polar contaminants or poor refining quality. DDF shall be measured in accordance with IEC 60247 (reference method) or IEC 61620 at 90 °C.

NOTE By agreement between parties, DDF can be measured at temperatures other than 90 °C.

6.7 Colour and appearance

The colour of an insulating oil is determined in transmitted light and is expressed by a numerical value based on comparison with a series of colour standards. Colour shall be measured following ISO 2049 (reference method) or ASTM D1500.

A visual inspection of insulating oil (oil sample in transmitted light under a thickness of approximately 10 cm and at ambient temperature) will indicate the presence of visible contaminants, free water or suspended matter.

6.8 Acidity

Mineral insulating oil shall be free from any acidic compound. Acidity shall be measured according to IEC 62021-2 (reference method) or IEC 62021-1.

6.9 Interfacial tension (IFT)

Low IFT indicates the presence of polar compounds. IFT shall be measured in accordance with IEC 62961 (reference method) or ASTM D971.

6.10 Sulphur content

Different organo-sulphur compounds can be present in mineral oils, dependent on the crude oil origin and the degree and type of refining. Refining reduces the content of sulphur and aromatic hydrocarbons. As some naturally present sulphur compounds have an affinity to metals, they may act as natural oxidation inhibitors or they may promote corrosion.

Total sulphur content analysis is a requirement for mineral oils of Type A (Table 3).

Total sulphur content shall be measured following ISO 14596 (reference method) or ISO 8754.

6.11 Corrosive and potentially corrosive sulphur

Some sulphur compounds, for example mercaptans, are very corrosive to metal surfaces, i.e. steel, copper and silver and shall not be present in oil as delivered. This type of corrosive sulphur shall be detected following DIN 51353.

Some other sulphur compounds, for example dibenzyldisulphide (DBDS), may result in the deposition of copper sulphide (Cu_2S) in paper insulation, reducing its electrical insulation properties (see Annex B). This has resulted in several equipment failures in service.

IEC 62535, based on work performed by CIGRE WG A2.32, provides the best currently available method to detect potentially corrosive sulphur compounds in oil. It applies only to oils that do not contain a metal passivator additive (declared or undeclared).

. For passivator-containing oils, see Clause B.3

6.12 Additives (see 3.3)

6.12.1 General

The chemical family and function of all additives shall be declared in product data sheets and certificates of compliance. For antioxidant additives and passivators, their concentrations shall also be stated.

6.12.2 Antioxidants (see 3.4)

Antioxidants slow down the oxidation of oil and therefore the formation of degradation products such as sludge and acids. It is useful to know whether and in what proportion antioxidant additives have been added in order to monitor additive depletion during service.

Additives that slow down the oxidation of mineral insulating oils include:

– oxidation inhibitors such as phenols. The most widely used oxidation inhibitors are DBPC and DBP (see 3.4). Detection and measurement of DBPC and DBP shall be carried out in accordance with IEC 60666. IEC test methods are not available for other types of inhibitors.
– other antioxidant additives such as sulphur-, amine- and phosphor- containing compounds, for example organic polysulphides and dithiophosphates (see 3.4.1). An antioxidant additive of this type is DBDS (see 6.18), but it is forbidden as it is known to be potentially corrosive to copper and will likely result in the oil failing the potentially corrosive sulphur test of IEC 62535.
– metal passivators (see 3.4.2).

6.12.3 Metal passivators

Some of these additives form thin films on copper, preventing the catalytic effect of copper in oil and the formation of harmful copper sulphide deposits in paper generated by the reaction of corrosive sulphur compounds in the oil with copper. Some of them protect the oil from the catalytic action of metals and slow down the rate of oxidation of oil. Passivators therefore slow down the oxidation process in IEC 61125 as they passivate the surface of the catalysing copper-wire, thus leading to an optimistic result of the oxidation stability test. Some of them are also used to modify the electrostatic charging tendency of oils (see 7.2).

Three main types of benzotriazole derivatives are typically used as metal passivator additives: N-bis(2-ethylhexyl)-aminomethyl-tolutriazole (TTAA), benzotriazole (BTA) and 5-methyl-1H-benzotriazole (TTA). Detection and measurement of these additives shall be according to IEC 60666.

Several other compounds can be used as metal passivator additives, such as N,N-bis(2-ethylhexyl)-1H-1,2,4-triazole-1 methanamine (TAA), diamino-diphenyldisulphide, nicotinic acid, hydroquinoline and other sulphur-based compounds, for which no IEC test methods are available[1].

6.12.4 Pour point depressants

These additives are used to improve the pour point and viscosity of oils at very low temperatures. Detection and measurement of the two main types of pour point depressant additives used (polynaphthalenes and polymethacrylates) shall be carried out according to IEC 60666.

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[1] Examples of commercially available TTAA and TAA are Irgamet® 39 and Irgamet® 30, respectively. This information is given for the convenience of users of this document and does not constitute an endorsement by the IEC of these products.

6.13 Oxidation stability

Oxidation of oil gives rise to acidity and sludge formation. This can be reduced by using oils with a high oxidation stability thus minimizing sludge deposition and maximizing insulation life. Oxidation stability is tested in accordance with IEC 61125 and the limits are indicated in Table 3 and Table 4. There is an option for stricter limits for special applications. In some countries more stringent limits and/or additional requirements and tests may be requested.

Test durations for oils containing phenolic inhibitors shall be as indicated in Table 3 and Table 4. Test duration for oils containing other antioxidant additives and metal passivators shall be 500 h.

Passivator-containing oils shall be tested for oxidation stability before the passivator additive has been added to the oil, using the test durations of Table 3 and Table 4.

6.14 Flash point

The safe operation of electrical equipment requires an adequately high flash point that is measured in accordance with ISO 2719 (Pensky-Martens closed cup procedure).

6.15 Polycyclic aromatics (PCAs) and polyaromatic hydrocarbons (PAHs)

Some PCAs are classified as carcinogens and therefore need to be controlled to an acceptable level in mineral insulating oil. PCAs are evaluated under the conditions of IP 346 after extraction with dimethyl sulfoxide (DMSO). Mineral insulating oils shall be considered as non-carcinogenic if PCA content (measured by IP 346 method) is below 3 %.

Benzo(a)pyrene (BaP) and some other polyaromatic hydrocarbons (PAHs) have been classified as carcinogenic, mutagenic and toxic to reproduction and can be measured according to EN 16143.

NOTE Acceptable limits of total or individual PCAs and PAHs are specified in some national and local regulations, e.g. REACH (https://www.echa.europa.eu/candidate-list-table).

6.16 Polychlorinated biphenyl content (PCBs)

Mineral insulating oil shall be free from PCBs. The reference test method is IEC 61619.

NOTE Acceptable limits of total or individual PCBs are specified in national and local regulations. Further specifications are described in European Directive 96/59/EC and UN Guideline for the identification of PCBs and materials containing PCBs.

6.17 2-furfural (2-FAL) and related compounds content

2-FAL and related compounds in mineral insulating oils can result either from improper re-distillation after solvent extraction during refining or re-refining or from contamination with used oil.

Mineral insulating oils should have a low level of 2-FAL and related compounds; measurement shall be carried out according to IEC 61198.

NOTE Related compounds are: 5-hydroxymethyl-2-furfural (5HMF), 2-furfurylalcohol (2FOL), 2-acetylfuran (2ACF) and 5-methyl-2-furfural (5MEF).

Furanic compounds are by-products of insulating paper aging in oil filled transformers. Monitoring furanic content of oil samples taken from in-service transformers can be used as an indication of paper aging. Therefore it is important to have supplied insulating oil free from 2-furufural content.

6.18 DBDS content

This compound is potentially corrosive at normal transformer operating temperatures and can produce copper sulphide. It therefore shall not be present in mineral insulating oil. The test method for measuring DBDS shall be in accordance with IEC 62697-1.

6.19 Stray gassing under thermo-oxidative stress

Stray gassing under thermo-oxidative stress (called stray gassing in this document) describes the development of gases in an insulating liquid in-service under temperatures considered usual for normal operating conditions (IEC 60076-7), due to its constituents and not connected to an internal fault in the electrical equipment. Various kinds of gassing have been observed, for example hydrogen, methane, ethane or a combination of these gases. Stray gassing is accelerated by oxygen content and copper availability as well as by temperature. Nevertheless, it has been observed both in open breathing and sealed equipment.

Stray gassing can be caused for different reasons, for example refining, additives. The definition used here for stray gassing does not include the influence of incompatible materials on the gassing of oil. In reality, however, outgassing of paints or some types of cross-linked polyethylene (XLPE), as well as other incompatible materials can contribute to gas formation not related to internal faults. The method used in this document and described in Annex A does not consider this, since the compatibility of materials is a responsibility of the equipment manufacturer.

A dissolved gas analysis (DGA) has been developed as a tool recognizing faulty conditions in liquid insulated electrical equipment. The most common evaluation schemes, however, may not distinguish between this kind of stray gassing and certain kinds of fault and therefore can lead to misinterpretation.

It is therefore useful to have a method characterizing the stray gassing behaviour (under thermo-oxidative stress) of a certain oil. In practice, gas due to stray gassing only has not been proven to be harmful to the equipment and it usually levels off with time. The proposed method provides useful information to help users differentiate between genuine fault conditions in an electrical equipment and stray gassing due to thermo-oxidative stress. This characterization should be considered when users select an oil for equipment so that it forms part of the supporting information when DGA is done.

The method used in this document and described in Annex A is suitable for oil insulated electrical equipment with copper windings. The gas patterns may be somewhat different if copper windings are in reality enamelled or made out of aluminium conductors. It implements a temperature of 105 °C, which is the highest permissible top oil temperature at normal cyclic loading according to IEC 60076-7 for the duration of 48 h (it has been shown that longer incubation times do not increase the significance of results) in the presence of copper (copper enhances the radical formation and is a metal used for the windings in the majority of electrical equipment).

The incubation at 105 °C can be carried out with air or nitrogen saturated oil with and without the presence of copper. Testing under all these conditions can be beneficial for qualifying a new oil.

The results of the RRT showed that the most severe condition for gas formation is under air saturated oil in the presence of copper. The limits reported in Table 3 are based on the testing under this condition.

Table 3 – General specifications, Type A (fully inhibited high grade oils)

Property	Test method	Limits	
		Transformer oil	Low temperature switchgear oils
1 – Function			
Viscosity at 40 °C	ISO 3104[a] or ASTM D7042	Max. 12 mm^2/s	Max. 3,5 mm^2/s
Viscosity at –30 °C [b]	ISO 3104[a] or ASTM D7042	Max. 1 800 mm^2/s	–
Viscosity at –40 °C [c]	IEC 61868	–	Max. 400 mm^2/s
Pour point	ISO 3016	Max. –40 °C	Max. –60 °C
Water content	IEC 60814	Max. 30 mg/kg [d] / 40 mg/kg [e]	
Breakdown voltage	IEC 60156	Min. 30 kV / 70 kV [f]	
Density at 20 °C	ISO 12185 [a] or ISO 3675 or ASTM D7042	Max. 895 kg/m^3	
DDF at 90 °C	IEC 60247 [a] or IEC 61620	Max. 0,005	
2 – Refining/stability			
Colour	ISO 2049	L0,5 (less than 0,5)	
Appearance	–	Clear, free from sediment and suspended matter	
Acidity	IEC 62021-2[a] or 62021-1	Max. 0,01 mg KOH/g	
Interfacial tension	IEC 62961 [a] or ASTM D971	Min. 43 mN/m	
Total sulphur content	ISO 14596 [a] or ISO 8754	Max. 0,05 %	
Corrosive sulphur	DIN 51353	Not corrosive	
Potentially corrosive sulphur	IEC 62535	Not corrosive	
DBDS	IEC 62697-1	Not detectable (< 5 mg/kg)	
Inhibitors of IEC 60666	IEC 60666	(I) Inhibited oil: 0,08 % to 0,40 % (see 3.7)	
Metal passivator additives of IEC 60666	IEC 60666	Not detectable (< 5 mg/kg), or as agreed upon with the purchaser	
Other additives		See [g]	
2-furfural and related compounds content	IEC 61198	Not detectable (< 0,05 mg/kg) for each individual compound	
Stray gassing under thermo-oxidative stress	Procedure in Clause A.4 (oil saturated with air) in the presence of copper	Non stray gassing: < 50 µl/l of hydrogen (H_2) and < 50 µl/l methane CH_4) and < 50 µl/l ethane (C_2H_6)	
3 – Performance			
Oxidation stability	IEC 61125: Test duration (I) Inhibited oil: 500 h	For oils with other antioxidant additives and metal passivator additives, see 6.12.2	
– Total acidity [h]	4.8.4 of IEC 61125:2018	Max. 0,3 mg KOH/g	
– Sludge [h]	4.8.1 of IEC 61125:2018	Max. 0,05 %	
– DDF at 90 °C [h]	4.8.5 of IEC 61125:2018	Max. 0,050	
4 – Health, safety and environment (HSE)[i]			
Flash point	ISO 2719	Min. 135 °C	Min. 100 °C
PCA content[j]	IP 346	< 3 %	
PCB content	IEC 61619	Not detectable (< 2 mg/kg)	

[a] Reference method.

[b] This is the standard LCSET for a transformer oil (see 6.1) and can be modified depending on the climatic condition of each country. Pour point should be minimum 10 °C below LCSET.

[c] Standard LCSET for low temperature switchgear oil.

[d] For bulk supply.

[e] For delivery in drums and IBC.

[f] After laboratory treatment (see 6.4).

[g] The supplier shall declare the chemical family and function of all additives (3.3), and the concentrations in the cases of inhibitors, antioxidants and passivators (3.4).

[h] At the end of oxidation stability tests.

[i] In some countries there can be additional requirements, e.g. REACH in the EU.

[j] Some individual PAH compounds can be determined by EN 16143.

Table 4 – General specifications, Type B (uninhibited and inhibited standard grade oils)

Property	Test method	Limits	
		Transformer oil	Low temperature switchgear oils
1 – Function			
Viscosity at 40 °C	ISO 3104[a] or ASTM D7042	Max. 12 mm^2/s	Max. 3,5 mm^2/s
Viscosity at –30 °C [b]	ISO 3104[a] or ASTM D7042	Max. 1 800 mm^2/s	–
Viscosity at –40 °C [c]	IEC 61868	–	Max. 400 mm^2/s
Pour point	ISO 3016	Max. –40 °C	Max. –60 °C
Water content	IEC 60814	Max. 30 mg/kg [d]/ 40 mg/kg [e]	
Breakdown voltage	IEC 60156	Min. 30 kV / 70 kV [f]	
Density at 20 °C	ISO 12185 [a] or ISO 3675 or ASTM D7042	Max. 895 kg/m^3	
DDF at 90 °C	IEC 60247 [a] or IEC 61620	Max. 0,005	
2 – Refining/stability			
Colour	ISO 2049	Max. 1,5	
Appearance	–	Clear, free from sediment and suspended matter	
Acidity	IEC 62021-2[a] or 62021-1	Max. 0,01 mg KOH/g	
Interfacial tension	IEC 62961 [a] or ASTM D971	Min. 40 mN/m	
Corrosive sulphur	DIN 51353	Not corrosive	
Potentially corrosive sulphur	IEC 62535	Not corrosive	
DBDS	IEC 62697-1	Not detectable (< 5 mg/kg)	
Inhibitors of IEC 60666	IEC 60666	Uninhibited (U): not detectable (< 0,01 %) Trace inhibited (T): ≥ 0,01 < 0,08% Inhibited oil (I): 0,08 % to 0,40 % (see 3.5 to 3.7)	
Metal passivator additives of IEC 60666	IEC 60666	Not detectable (< 5 mg/kg), or as agreed upon with the purchaser	
Other additives		See [g]	
2-furfural and related compounds content	IEC 61198	Not detectable (< 0,05 mg/kg) for each individual compound[h]	
3 – Performance			
Oxidation stability	IEC 61125 Test duration [i] (U) Uninhibited oil: 164 h (T) Trace inhibited oil: 332 h (I) Inhibited oil: 500 h	For oils with other antioxidant additives and metal passivator additives, see 6.12.2	
– Total acidity [j]	4.8.4 of IEC 61125:2018	max. 1,2 mg KOH/g	
– Sludge [j]	4.8.1 of IEC 61125:2018	max. 0,8 %	
– DDF at 90 °C [j]	4.8.5 of IEC 61125:2018	max. 0,500	
4 – Health, safety and environment (HSE)[k]			
Flash point	ISO 2719	Min. 135 °C	Min. 100 °C
PCA content[l]	IP 346	< 3 %	
PCB content	IEC 61619	Not detectable (< 2 mg/kg)	

Stray gassing under thermo-oxidative stress (see 6.19) is not included as a normative test for mineral oils Type B, because there has been insufficient data to determine appropriate limits. The requirement for a stray gassing test, as well as the limit values, if stipulated, can be negotiated between the user and supplier.

[a] Reference method.

[b] This is the standard LCSET for a transformer oil (see 6.1) and can be modified depending on the climatic condition of each country. Pour point should be minimum 10 °C below LCSET.

[c] Standard LCSET for low temperature switchgear oil.

[d] For bulk supply.

[e] For delivery in drums and IBC.

[f] After laboratory treatment (see 6.4).

[g] The supplier shall declare the function and chemical family of all additives (3.3), and the concentrations in the cases of inhibitors antioxidants and passivators (3.4).

[h] In agreement with the customer, oils with a higher furfural content can be delivered, when these values do not jeopardize the application.

[i] In some countries there can be lower requirements for oxidation stability.

[j] At the end of oxidation stability tests.

[k] In some countries there can be additional requirements, e.g. REACH in the EU.

[l] Some individual PAH compounds can be determined by EN 16143.

7 Additional properties

7.1 General

Determination of properties such as electrostatic charging tendency, gassing tendency, thermal properties may be required for certain applications. Where required such measurements shall be performed according to a given standard and with specific limits, negotiated between supplier and user.

7.2 Electrostatic charging tendency (ECT)

ECT of oil is an important property for certain designs of high voltage (HV) and extra-high voltage (EHV) transformers which have oil pumping rates that can give rise to the build-up of electrostatic charge. This charge can result in energy discharge causing transformer failure.

NOTE A method to measure ECT is proposed by CIGRE Technical Brochure 170. It has been reported that ECT can be modified by using metal passivator additives such as BTA and TTA.

7.3 Gassing tendency

Gassing tendency describes oil capability to absorb or evolve gases when subjected to electrical stress and ionisation under specified laboratory conditions. A low gassing tendency is preferred by some users for special equipment such as HV instrument transformers and bushings. Gas absorption properties may be related to oil aromatic content. Gassing tendency shall be measured using Method A of IEC 60628:1985.

NOTE 1 Additives such as 1,2,3,4- tetrahydronaphtalene (tetralin), mono or dibenzyltoluene and others have been proposed to reduce the gassing tendency of some oils, but are not described in IEC 60666. Mono and dibenzyltoluene are described in IEC 60867.

NOTE 2 If requested by the purchaser, gassing tendency according to IEC 60628 can be agreed upon between the supplier and purchaser of the oil.

7.4 Thermal properties

The thermal performance of an oil-filled electrical equipment requires knowledge of the oil thermal characteristics like thermal conductivity, specific heat capacity, thermal expansion coefficient. They are, however, not to be considered as acceptance, but as design parameters. Thermal conductivity measures temperature change across a liquid sample for a known amount of energy input and can be tested according to ASTM D7896. Specific heat capacity measures the capacity of oil to absorb thermal energy and is tested according to ASTM E1269. Thermal expansion coefficient is determined according to ASTM D1903.

7.5 Properties connected with consistency (aromatic content, distribution of PAHs, refractive index)

Mineral oils contain different kinds of aromatic structures, mono-, di-, tri- and polyaromatic. The amount of aromatics, measured according to IEC 60590, as well as their distribution (mono-, di-, tri- and polyaromatics, to be measured according to ASTM D6591) is typical for a certain oil product and can provide valuable information on its constitutional continuity over time. The same is valid for the refractive index, which is related to the oil constitution and the refining process and can be determined according to DIN 51423-1 (reference method) or ASTM D1218.

7.6 Lubricating properties

Mechanical equipment, such as tap-changers immersed and operating in the insulating liquid, require sufficiently good lubricating properties of the liquid, which can be tested according to DIN 51350-3 (reference method) or ASTM D4172.

7.7 Particle content

Particles in mineral insulating oil may result from manufacturing, storage or handling of the oil, and may affect its breakdown voltage (see 6.4). Particles and moisture content act in a synergic way on the breakdown strength. Measurement shall be carried out according to IEC 60970.

NOTE Particle content in drums at delivery of oil can be agreed between supplier and customer, based on a statistical reference at delivery.

7.8 Foaming

Contaminations with silicone, phthalates or other surface-active chemicals or oils can cause undesired foaming – see Annex C.

7.9 Transformer oil test equivalents

. Some transformer oil test equivalents are presented in Annex D, Table D.1

Annex A
(normative)

Method for stray gassing under thermo-oxidative stress

A.1 Overview of the method

The procedure described according to Clauses A.2 to A.7 has been established by a joint work of CIGRE working group D1.70 and IEC technical committee 10 and submitted to a round robin test to evaluate its robustness. Eighteen laboratories participated in the RRT that was conducted on a set of mineral insulating oils of different nature and origin.

Clause A.8 provides a summary of the outcome of the RRT.

According to this procedure, a representative sample of mineral oil is subjected to the following incubation procedures:

– A first portion of sample oil saturated with air (as described in Clause A.4), a second portion of sample oil saturated with nitrogen (as described in Clause A.5) are used.
– 2 aliquots of both saturated samples are incubated, at 105 °C for 48 h, in glass syringes, one filled with oil only, the second with copper in contact with the oil.
– Finally, a DGA according to IEC 60567 is performed on each incubated sample.

A.2 Required materials

Performing this test procedure requires the following materials and/or chemical reagents:

– ventilated oven, able to maintain a temperature of (105 ± 2) °C;
– 100 ml syringes, suitable for DGA (see specific requirements in IEC 60475). They shall be of good quality and have a good gas tightness to avoid gas leakage during the incubation time and later;
– 3-way metal stopcock (chrome plated brass, stainless steel, or equivalent);
– beaker 250 ml according to ISO 3819;
– copper foil, 0,25 mm thickness, purity 99,89 % or higher;
– high vacuum silicone grease;
– dry air, free of hydrocarbons;

NOTE 1 Synthetic air (purity > 99,999 %) for gas chromatography is suitable.

– dry nitrogen, free of oxygen and hydrocarbons.

NOTE 2 Nitrogen (purity > 99,999 %) for gas chromatography is suitable.

A.3 Pretreatment of syringes

Syringes shall be clean and dried prior to this pretreatment.

Treat the upper part of the piston (close to the handle) with high vacuum silicone grease, dispersing a thin, uniform layer of silicone around the entire circumference of the piston.

Immerse the lower part of the piston in a sub-sample of the liquid to be tested, avoiding the contact between the oil sample and the grease. The sub-sample of the test liquid is not to be used further since it is likely to have been in contact with the grease.

A.4 Procedure A: stray gassing under oxidative conditions (high oxygen content)

A.4.1 Pretreatment of mineral oil

Place a suitable volume of the sample oil in a beaker, and purge it with air to ensure saturation. The prepared oil shall be sufficient to fill two syringes with 50 ml to 60 ml of oil, one with copper foils inside, the second without copper (oil only).

NOTE The oil sample is not filtered or vacuum treated in the laboratory prior to purging.

A.4.2 Filling syringes with mineral oil

Prepare two syringes according to Clause A.3. In one of the two, place two copper foils, cut and when required folded in a shape that allows their introduction into the syringe without occluding the ingress of the oil from the tip (see Figure A.1). The total surface of copper shall be 9,6 cm^2 to 10 cm^2. Copper shall be prepared before use, as described in the standard for oxidation stability IEC 61125.

Fill both syringes with 50 ml to 60 ml with oil.

Firmly close each syringe with the metal 3-ways stopcock.

IEC

Figure A.1 – Syringes with and without copper

A.4.3 Incubation procedure

Place the syringes in the ventilated oven, in vertical (or semi-vertical) position, with piston up and laying on the stopcock.

NOTE The vertical positioning limits the gas leakages from the tip of the syringe.

Keep the syringes at 105 °C, for a duration of (48 ± 0,5) h.

A.4.4 Dissolved gas analysis

After cooling the syringes to room temperature, perform the dissolved gas analysis (DGA, according to IEC 60567) on the tested oils.

A.5 Procedure B: stray gassing under inert conditions (low oxygen content)

. Perform all steps from A.4.1 to A.4.4, after purging the oil with nitrogen instead of air

A.6 Reporting

A.6.1 Test report

Report all gases measured in the applied conditions of incubation, indicating each gas to the nearest µl/l according to IEC 60567.

A.6.2 Evaluation of the stray gassing behaviour of the oil

For a general assessment and classification of a new type of oil, it is recommended to run the test with both procedures A and B, with and without copper. For a verification of a new batch of oil, testing with procedure A with copper can be sufficient.

The oil is evaluated as "non stray gassing" if, after incubation under all the tested conditions, none of the following gases exceeds 50 µl/l:

– hydrogen;
– methane;
– ethane.

Otherwise, the oil is evaluated as "stray gassing" if, after incubation under at least one of the tested conditions, at least one of the following gases exceeds 50 µl/l:

– hydrogen;
– methane;
– ethane.

NOTE Although carbon monoxide and carbon dioxide show trends in case of stray gassing as well, their evaluation is more difficult to assess, since a certain impact of these gases in real equipment is due to the solid insulation.

A.7 Precision data

A.7.1 General

Precision data were evaluated within CIGRE WG D1.70, with a round robin test.

A.7.2 Repeatability

In a limited set of experiments, the standard deviation of three replications performed in the same laboratory, by the same operator, and on the same sample, was estimated in the range:

$$8\ \% < \text{standard deviation} < 25\ \%$$

for final gas concentrations over 25 µl/l of hydrogen, methane or ethane.

NOTE The repeatability estimated for this test method includes the repeatability of the DGA measurement performed after incubation. For information about the repeatability of DGA, see IEC 60567.

A.7.3 Reproducibility

The standard deviation of the round robin test, where the same sample was tested by different laboratories (n = 18), was estimated in the range:

$$30\ \% < \text{standard deviation} < 80\ \%$$

. for final gas concentrations over 25 µl/l of hydrogen, methane or ethane

NOTE The reproducibility estimated for this test method includes the reproducibility of the DGA measurement performed after incubation. For information about the reproducibility of DGA, see IEC 60567.

Despite the high spread of individual results (for different gases, under different incubation conditions), the results of the RRT in terms of estimation of an oil as "stray gassing" or "non stray gassing" were the following:

– Occurrence of false negatives (laboratories casting an oil as "non stray gassing" when the majority of the results cast the oil as "stray gassing"): 1 or 2 cases out of 18 laboratories.
– Occurrence of false positives (laboratories casting an oil as "stray gassing" when the majority of the results cast the oil as "non stray gassing"): none.

A.8 Results of the RRT

A.8.1 General

The round robin test allowed identifying some "stray gassing patterns". The results are reported here for information. The reported patterns of stray gassing are, of course, limited to the experiments done within CIGRE WG D1.70, and do not represent a full picture of all the possible stray gassing patterns. Nevertheless, they may be useful for better understanding the performance of different types of mineral insulating oils.

Stray gassing patterns are described in graphs, where the development of hydrogen, methane, ethane and carbon monoxide is reported for each tested condition.

A.8.2 Stray gassing pattern 1

The pattern shown in Figure A.2 has been shown by an uninhibited unused mineral oil.

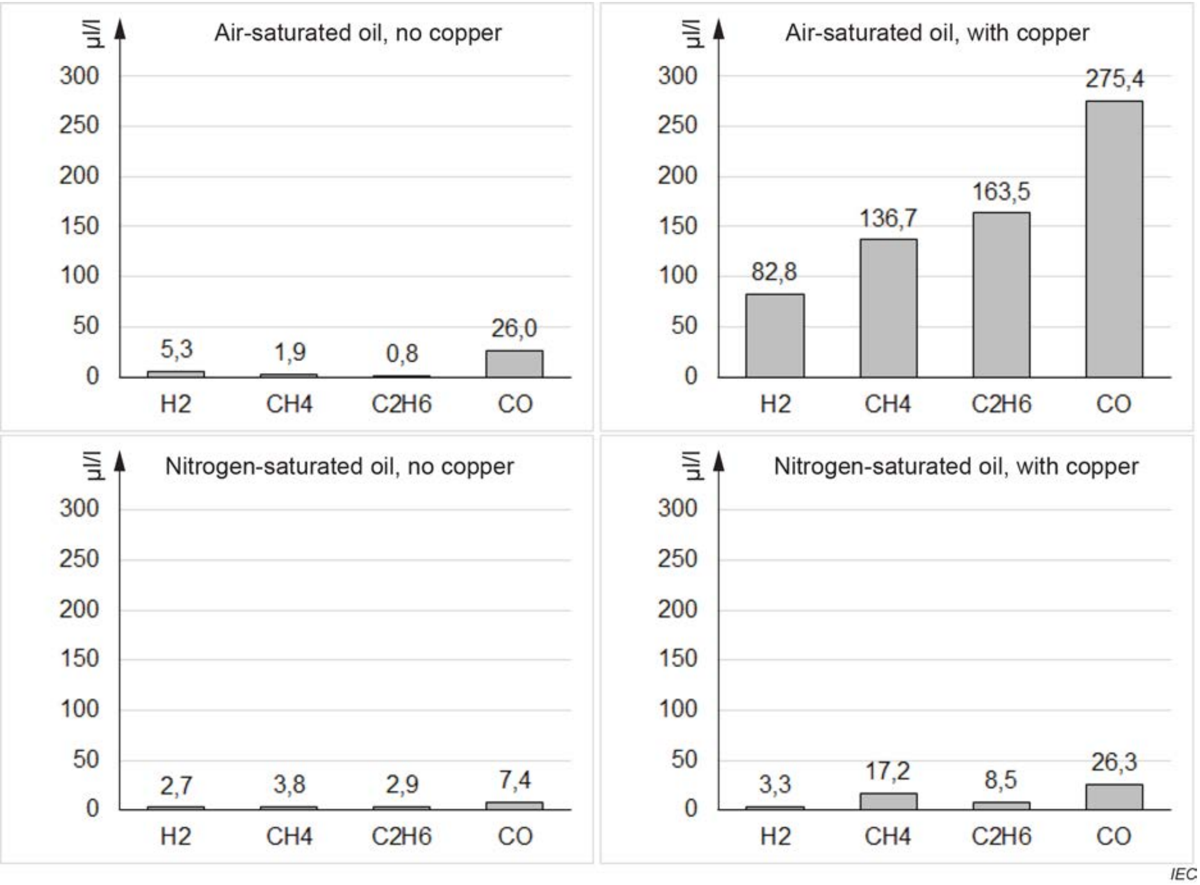

Figure A.2 – Stray gassing pattern 1

A.8.3 Stray gassing pattern 2

The pattern shown in Figure A.3 has been shown by an inhibited unused mineral oil, containing a triazolic passivator additive.

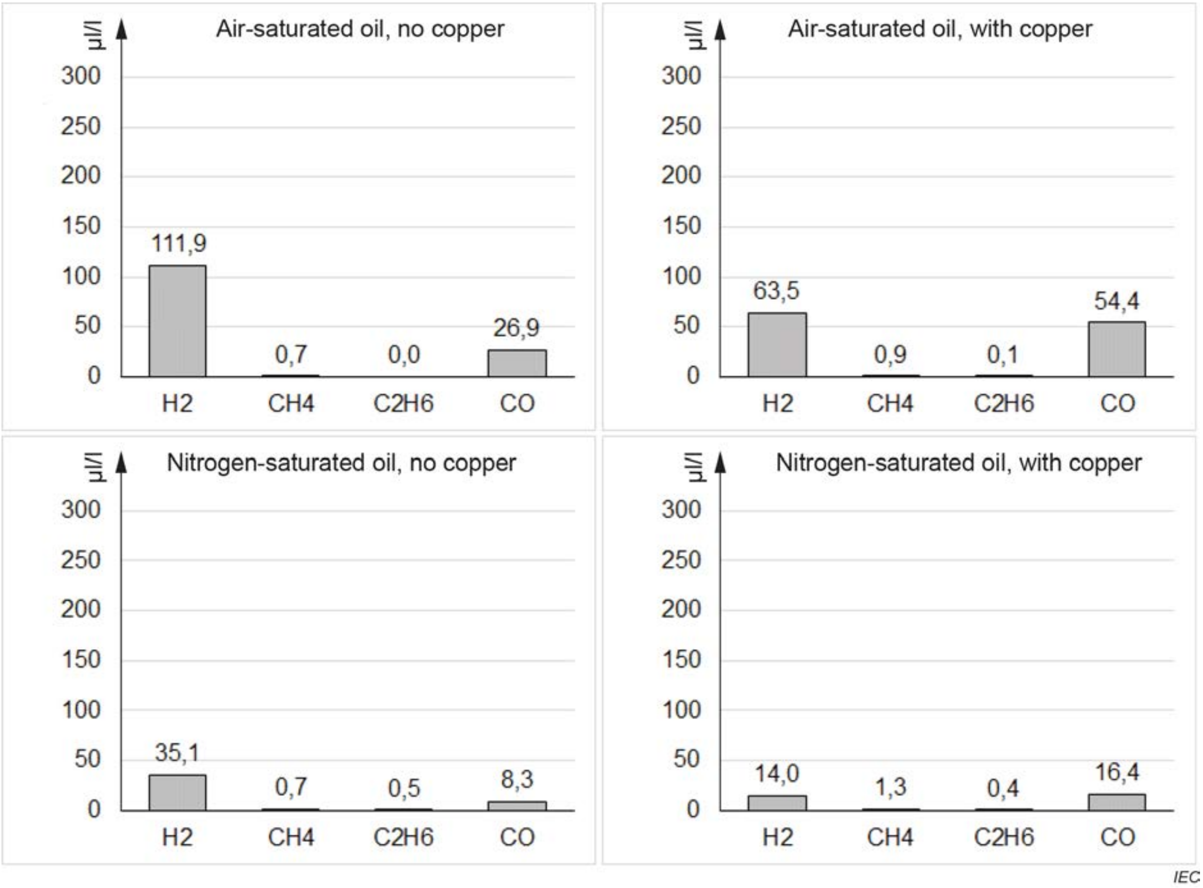

Figure A.3 – Stray gassing pattern 2

A.8.4 Stray gassing pattern 3

The pattern shown in Figure A.4 has been shown by inhibited mineral oils, both unused and recycled. The graphs show typical values, considering the very low deviation between the tested oils.

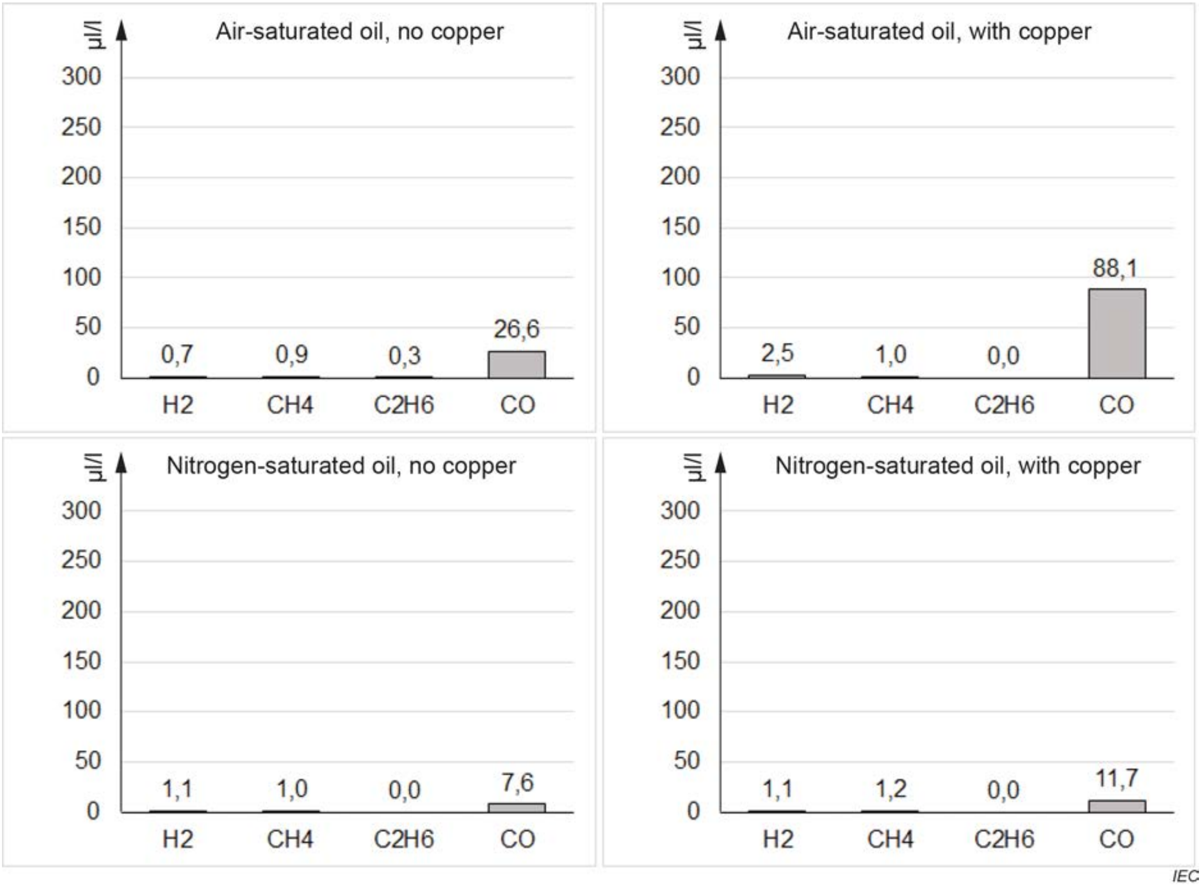

Figure A.4 – Stray gassing pattern 3

A.8.5 Stray gassing pattern 4

The pattern shown in Figure A.5 has been shown by an uninhibited recycled mineral oil.

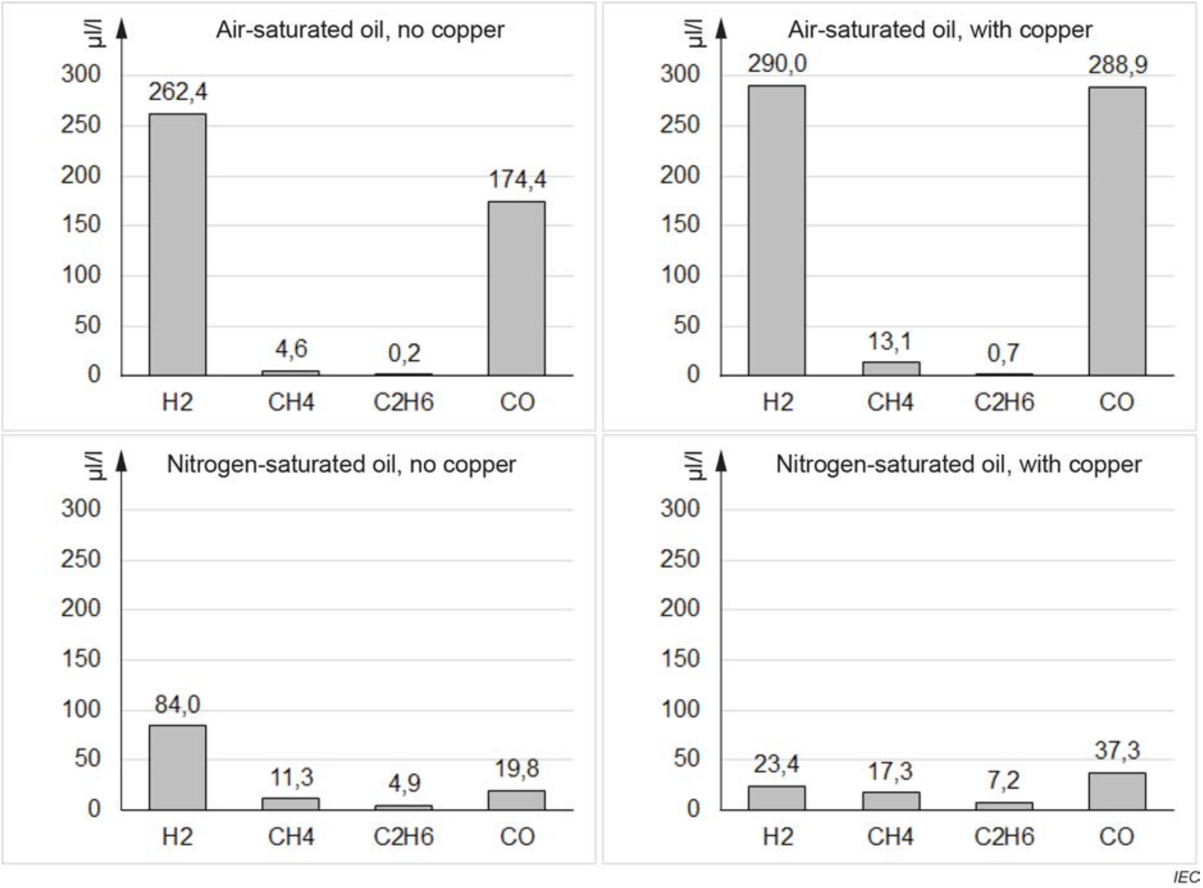

Figure A.5 – Stray gassing pattern 4

Annex B
(informative)

Potentially corrosive sulphur

B.1 Mechanism of copper sulphide deposition

The mechanism of copper sulphide (Cu_2S) deposition has still not been fully elucidated.

The strong influence of temperature and oxygen on this process indicates that some oxidized sulphur species may be more active than those originally present in oil, or that other oxidation products are important as co-complexing agents (see CIGRE Technical Brochure 378). Cu_2S deposition occurs preferentially in equipment where corrosive sulphur compounds are present in oil, unvarnished or unprotected copper is used, operating temperatures are high and the amount of oxygen in oil is limited. The optimal oxygen content for copper transport seems to be relatively low, probably in the region of a few thousand ul/l, but deposition may occur over a wide range of oxygen contents.

B.2 Corrosive sulphur compounds in oil

Although many sulphur compounds are known to be corrosive for copper, few have been identified as components of insulating oil. The only compound shown so far to be a potent Cu_2S forming agent and to be present in significant amounts in mineral insulating oil is dibenzyldisulphide (DBDS), known as an additive in the lubricant industry. Most oils found to be forming Cu_2S contain this substance. Refining processes using severe hydrotreatment can easily remove reactive sulphur compounds which are potentially corrosive and found in crude oil like disulphides, thioethers, various oxidized sulphur compounds and elemental sulphur. Such substances may be released or formed from a non-corrosive oil by imprudent oil treatment (e.g. improper reclaiming, see CIGRE Technical Brochure 625). Silver-coated metal surfaces (e.g. tap-changer contacts) are extremely susceptible to reactions with elemental sulphur to form silver sulphide layers which may increase contact resistance or flake off and initiate flashovers.

B.3 Detection of corrosive sulphur compounds in oils containing passivators

B.3.1 General

When oil in a transformer contains a metal passivator additive, a thin protective layer of passivator is formed on copper surfaces, which prevents copper from dissolving in oil, reacting with corrosive sulphur compounds present in oil, and depositing in paper insulation as harmful copper sulphide (Cu_2S).

The same occurs when testing passivator-containing oils according to IEC 62535. This test method therefore cannot detect corrosive sulphur compounds present in passivator-containing oils and may provide "false negative" results for such oils. Passivator-containing oils testing negative as unused oils may then test positive and start depositing harmful Cu_2S after the additive has been consumed by aging in transformer service.

In order to detect corrosive sulphur compounds in oil containing a metal passivator additive (declared or suspected), the passivator additive has to be removed from the oil first. The following two procedures can be used for that purpose. Both are intended for newly available types of oils only, not for normal deliveries of oil.

B.3.2 Procedure 1

In this procedure, metal passivator additives are eliminated by specific adsorption from the oil:

a) if the initial concentration of passivator was > 200 mg/kg, then stir 100 ml of passivator-containing oil with 500 mg of Chromabond®[2] HR-XC adsorbent (a strong, mixed-mode, polymer-based cation exchanger for basic analytes), for 1 h, then filter out the adsorbent;

   or

b) alternately, if the initial concentration of passivator was < 200 mg/kg, you may extract 60 ml of oil under a slight vacuum on a 3 ml column containing 200 mg of the adsorbent.

B.3.3 Procedure 2

This procedure is based on the observation that metal passivator additives in oil are consumed by oxidation aging (in accelerated tests in the laboratory and in transformers in service):

a) Run the passivator-containing oil in the test cell used in IEC 61125 at 120 °C for 164 h with an air flow of 0,15 l/h to ensure that the passivator has been consumed by oxidation.
b) Test the aged oil for corrosive sulphur in the test cell according to IEC 62535 with a new paper wrapped conductor.
c) To avoid false positives with the aged oil (i.e. where oxidation aging compounds of oil are mistakenly interpreted as Cu_2S), confirm Cu_2S deposition using the SEM/EDX technique or other techniques (according to Annex B of IEC 62535:2008). False positives can also be avoided by carrying out a second test according to IEC 62535 without a copper strip and with paper only, and comparing the appearance of papers after both tests with and without copper.

NOTE 1 The protective layer of passivator on copper has been observed to remain on copper after aging tests in the laboratory, but there is little knowledge on whether and how long it will remain on copper in transformers in service.

NOTE 2 As a complement to IEC 62535 and procedures 1 and 2 for passivator-containing oils, the quantification of corrosive sulphur compounds in oil (e.g., dibenzyldisulphide (DBDS) and total disulphide) can be used to ensure that none of these potentially harmful compounds are present in oil.

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[2] Chromabond® HR-XC is the trademark of a product supplied by Macherey Nagel. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Annex C
(informative)

Contamination of oils with silicone

Mineral insulating oils suspected of having been accidentally contaminated with silicone, phthalates or other surface-active chemicals or oils should not be introduced in transformers, since these compounds can produce foaming in oil when trying to degas the transformer, thus making it difficult or impossible to fully degas the mineral insulating oil. The foaming tendency test of ISO 6247 can be used to detect such a contamination.

Annex D
(informative)

Transformer oil test equivalents

Table D.1 presents a short overview on the comparison of the test methods quoted as reference methods in IEC 60296 to some other test methods.

Table D.1 – Some transformer oil test equivalents

Tested Property	**Test method quoted in IEC 60296,**	**Comparable test method(s)**	**Method equivalency** [a]	**Some related method(s)** (not directly comparable or equivalent)
Kinematic viscosity	ISO 3104	ASTM D7042, ASTM D445	Y	
Viscosity at very low temperature	IEC 61868			
Pour point	ISO 3016	ASTM D97	Y	ASTM D5949
Water content	IEC 60814	ASTM D1533	Y	ASTM D6304
Breakdown voltage	IEC 60156	ASTM D1816	N	ASTM D877
Density	ISO 12185	ISO 3675, ASTM D7042	Y	ASTM D1298
Dielectric dissipation factor at 90 °C	IEC 60247	IEC 61620, ASTM D924	N	
Colour	ISO 2049	ASTM D1500	Y	
Appearance	IEC 60296 (see 6.7)	ASTM D1524	Y	
Acidity	IEC 62021-2, IEC 62021-1	ASTM D974, ASTM D664	Y	
Interfacial tension	IEC 62961	ASTM D971	Y only for new oils N for used oils a	
Total sulphur content	ISO 14596	ISO 8754	Y	
Corrosive sulphur (silver strip)	DIN 51353	ASTM D1275 (Method A)	N	
Corrosive sulphur (copper strip)		ASTM D1275 (Method B)		
Potentially corrosive sulphur	IEC 62535			
Inhibitor and antioxidant content	IEC 60666	ASTM D2668	Y	ASTM D4768
Metal passivator content	IEC 60666:2010, Annex B			
Pour point depressant detection	IEC 60666:2010, Annex C			
Other additive content	IEC 60666			

Tested Property	Test method quoted in IEC 60296,	Comparable test method(s)	Method equivalency [a]	Some related method(s) (not directly comparable or equivalent)
Oxidation stability (acidity, sludge and DDF)	IEC 61125	ASTM D2440	N	
Flash point (PMCC)	ISO 2719	ASTM D93	Y	
Flash and fire points (COC)		ISO 2592, ASTM D92	Y	
Polycyclic aromatic content	IP 346			EN 16143 (selected PAHs)
Polychlorinated biphenyl content	IEC 61619	EN 12766-2	N	ASTM D4059
Furanic compound content	IEC 61198	ASTM D5837	Y	
Dibenzyldisulphide content	IEC 62697-1			
Stray gassing under thermo-oxidative stress	IEC 60296 (see Annex A)			ASTM D7150
Electrostatic charging tendency	CIGRE TB 170			
Gassing tendency under electrical stress and ionization	IEC 60628:1985 (Method A)	ASTM D2300	N	
Thermal properties – Thermal conductivity	ASTM D7896			
Thermal properties – Specific heat capacity	ASTM E1269			
Thermal properties – Thermal expansion	ASTM D1903			
Aromatic content	IEC 60590	ASTM D6591	N	
Carbon-Type distribution[b]		ASTM D2140, ASTM D3238		
Refractive index	DIN 51423-1			ASTM D1218, ISO 5661
Lubricity	DIN 51350-3			ISO 20623, ASTM D4172
Particle content	IEC 60970			
DGA (headspace) [c,d]	IEC 60567:2011, 7.5	ASTM D3612 (Method C)	Y	
DGA (partial degassing) [c,d]	IEC 60567:2011, 7.3	ASTM D3612 (Method A)	Y	

[a] Tests deemed equivalent produce results which are equivalent.

[b] Not valid for isoparaffinic oils.

[c] Not part of IEC 60296.

[d] Results are given at 20 °C in IEC test methods and at 0 °C in ASTM test methods.

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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

3, rue de Varembé
PO Box 131
CH-1211 Geneva 20
Switzerland

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