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COMMENTED VERSION

INTERNATIONAL STANDARD

colour inside

Fluids for electrotechnical applications – Unused natural esters for transformers and similar electrical equipment

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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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FLUIDS FOR ELECTROTECHNICAL APPLICATIONS – UNUSED NATURAL ESTERS FOR TRANSFORMERS AND SIMILAR ELECTRICAL EQUIPMENT

FOREWORD

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This commented version (CMV) of the official standard IEC 62770:2024 edition 2.0 allows the user to identify the changes made to the previous IEC 62770:2013 edition 1.0. Furthermore, comments from IEC TC 10 experts are provided to explain the reasons of the most relevant changes, or to clarify any part of the content.

A vertical bar appears in the margin wherever a change has been made. Additions are in green text, deletions are in strikethrough red text. Experts' comments are identified by a blue-background number. Mouse over a number to display a pop-up note with the comment.

This publication contains the CMV and the official standard. The full list of comments is available at the end of the CMV.

IEC 62770 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications. It is an International Standard.

This second edition cancels and replaces the first edition published in 2013. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition: 1

a) Introduction of IEC 63012 which details other liquids not covered by this document. IEC 63012 was published in 2019 after the first edition of IEC 62770 (2013).
b) New Table 1 inserted which clarifies definitions.
c) Appearance and colour requirements now merged.
d) Pour point: Introduction of the importance of LCSET with advice on cold temperature behaviour of natural esters.
e) Additives: new agreed wording inserted on the declaration of additives.
f) Flash and fire points: now only determined by Cleveland Open Cup method, since the Pensky-Martens closed cup method was identified as problematic with natural esters.
g) Toxicity: Aquatic toxicity now emphasized.
h) Annex B removed as it is no longer needed since the publication of IEC 63012.

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

Draft	Report on voting
10/1215/FDIS	10/1243/RVD

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 International Standard 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/publications.

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, or
- revised.

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INTRODUCTION

Because of their higher fire points and ~~better~~ lower environmental ~~compatibility~~ impact relative to hydrocarbon petroleum derived insulating mineral oil, the use of vegetable oils and other natural esters is on the rise as insulating and heat transfer fluids in electrical devices such as transformers.

This document sets performance criteria for unused natural esters earmarked for electrical applications. However, the use of natural esters is recommended only for equipment that is not open to the atmosphere, for example sealed transformers and reactors because these ~~fluids~~ liquids are ~~prone~~ susceptible to ~~rapid~~ oxidation.

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

Unused natural esters which are the subject of this document should be handled with due regard to personal hygiene. Direct contact with eyes should be avoided. In case of eye contact, irrigation with copious amounts of clean running water should be carried out and medical advice sought.

Performance of some of the tests mentioned in this document could lead to a hazardous situation. Attention is drawn to the relevant document test method for guidance.

~~The disposal of natural esters, chemicals and sample containers mentioned in this standard 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 natural esters into the environment.~~ **2**

FLUIDS FOR ELECTROTECHNICAL APPLICATIONS – UNUSED NATURAL ESTERS FOR TRANSFORMERS AND SIMILAR ELECTRICAL EQUIPMENT

1 Scope

This document describes specifications and test methods for unused natural esters in transformers and similar ~~oil-impregnated~~ liquid-immersed electrical equipment in which a liquid is required as an insulating and heat transfer medium. The exposure of natural ester to air leads to deterioration of the insulating liquid. Use of natural esters is ~~not recommended for electrical equipment that is open to the atmosphere~~ therefore restricted to sealed units, or with the conservator tank protected from the contact with atmosphere by a membrane or other suitable system. **3**

In this document the term "natural esters" applies to insulating ~~fluids~~ liquids for transformers and similar electrical equipment with suitable biodegradability and lower environmental ~~compatibility~~ impact. Such natural esters are vegetable oils obtained from seeds, and oils obtained from other suitable biological materials ~~and delivered to an agreed point, at a set time period~~. These oils are comprised of triglycerides.

Natural esters with additives are within the scope of this document. Because of their different chemical composition, natural esters differ from insulating mineral oils and other insulating ~~fluids~~ liquids that have high fire points, such as synthetic esters or silicone fluids.

Natural ester-derived insulating ~~fluids~~ liquids with low viscosity have been introduced but are not covered by this document. ~~Pertinent properties of such fluids are given in Annex B.~~ IEC 63012 covers these liquids. **4**

This document is applicable only to unused natural esters. Reclaimed natural esters and natural esters blended with ~~non-natural esters fluids~~ other insulating liquids are beyond the scope of this document.

NOTE The chemical nomenclature and scientific notations used in the document are in accordance with the IUPAC handbook (Quantities, Units and Symbols in Physical Chemistry).

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. **5**

~~IEC 60076-14, *Power transformers - Part 14: Liquid-immersed power transformers using high-temperature insulation materials*~~

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 d) and d.c. resistivity ~~of insulating fluids~~*

~~IEC 60296, *Fluids for electrotechnical applications – Unused mineral insulating oils for transformers and switchgear*~~

IEC 60475, *Method of sampling ~~liquid dielectrics~~ insulating liquids*

IEC 60666, *Detection and determination of specific additives in mineral insulating oils*

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

~~IEC 61100, *Classification of insulating liquids according to fire-point and net calorific value*[1]~~

IEC 61125~~:1992~~, ~~*Unused hydrocarbon-based insulating fluids*~~ *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 62021-3, *Insulating liquids – Determination of acidity – Part 3: Test methods for non-mineral insulating oils*[2]

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

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)*

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

ISO 2592, *Petroleum and related products – Determination of flash and fire points – Cleveland open cup method*

~~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 fluids – Determination of kinematic viscosity and calculation of dynamic viscosity*

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

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

~~[1] Withdrawn in 2009 and partially replaced by IEC 61039.~~

~~[2] To be published.~~

~~ASTM D 1275, *Standard Test Method for Corrosive Sulfur in Electrical Insulating Oils*~~

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

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

~~OECD 201-203, *Test Guidelines for ecotoxicity*~~

~~OECD 301, *Guideline for testing of chemicals adopted by European Council on July 17th 1992*~~

~~US EPA, *Office of Prevention, Pesticides and Toxic Substances (OPPTS)*~~

~~835.311, *Fate, Transport and Transformation Test Guidelines*~~

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses: **6**

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

3.1.1
additives
~~suitable~~ chemical substances which are deliberately added to natural ester insulating ~~fluids~~ liquids in order to improve certain characteristics~~, e.g. pour point, viscosity, foaming, and oxidation stability~~

~~Note 1 to entry: Examples include antioxidants, pour point depressants, electrostatic charging tendency depressant, metal passivator or deactivators, antifoam agent, refining process improver, etc.~~

~~**3.2**~~
~~**corrosive sulfur**~~
~~free sulfur and corrosive sulfur compounds detected by subjecting metals such as copper to contact with an insulating liquid under standardized conditions~~

~~[SOURCE: IEC 60050-212:2010, definition 212-18-20, modified – inclusion of "metals such as"]~~

3.1.2
natural esters
vegetable oils obtained from seeds and oils obtained from other suitable biological materials and comprised of triglycerides

~~**3.4**~~
~~**potentially corrosive sulfur**~~
~~organo-sulfur compounds present in transformer oils that may cause copper sulfide formation~~

~~[SOURCE: IEC 62535:2008, definition 3.1 – modified, the NOTE to entry has been omitted]~~

3.1.3
unused natural esters
~~unused~~ natural esters as delivered by the supplier

Note 1 to entry: Such a liquid has not been used in, nor been in contact with, electrical equipment or other equipment not required for its manufacture, storage or transport.

~~Note 2 to entry: The manufacturer and supplier of unused natural esters will have taken all reasonable precautions to ensure that the natural esters are not contaminated with polychlorinated biphenyls, polychlorinated terphenyls or polycyclic aromatics (PCB,PCT, PCAs,) or corrosive sulfur compounds; used, reclaimed, or dechlorinated oils, or other contaminants.~~

3.2 Abbreviated terms

The abbreviated terms are given in Table 1.

Table 1 – Abbreviated terms 7

Abbreviated term	Full term
DBDS	Dibenzyl disulphide
DDF	Dielectric dissipation factor
HSE	Health, safety and environment
IBC	Intermediate bulk container
LCSET	Lowest cold start energizing temperature
PCBs	Polychlorinated biphenyls
RRT	Round robin test

4 Properties, their significance and test methods

4.1 General

~~Salient~~ Required characteristics of unused natural esters are listed in Table 2.

NOTE 1 Additional information on natural esters for transformers and similar electrical equipment is available in CIGRE brochure 436 and IEEE C57.147-2018™.

NOTE 2 In natural ester at normal operating temperatures, a significant formation of stray gases such as hydrogen and ethane is sometimes witnessed for a specific period (weeks to months) after the transformer is activated. Such unexpected gas formation at low temperature can lead to confusion in the dissolved gas analyses interpretation. Natural esters show a higher tendency towards stray gassing. 8

4.2 Physical properties

4.2.1 Appearance and colour

A visual inspection of unused natural esters (with light transmitted through a glass transparent beaker of approximately 10 cm thickness ~~of natural esters~~ at ambient temperature) will indicate the presence of visible contaminants, free water and suspended matter.

The colour of an insulating liquid 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.

4.2.2 Viscosity

Viscosity influences heat transfer and therefore affects ~~the increase of~~ temperature distribution in the transformer and other equipment. The lower the viscosity, the easier the ~~oil~~ liquid circulates, generally leading to better heat transfer. Viscosities at lower temperatures are a

critical factor for the cold start of transformers with ~~ON cooling~~ K class liquid insulation with natural (not forced/pumped) flow (the absence of circulation can lead to possible overheating at hot spots) **9**. It can have a negative impact on the speed of moving parts, such as on-load tap changer mechanisms, pumps and regulators. Due consideration should be given to viscosity at the lowest cold start energizing temperature (LCSET). Viscosity at 40 °C and 100 °C shall be measured according to ISO 3104 (referee method) or ASTM D7042.

4.2.3 Pour point

~~Pour point of unused natural esters is the lowest temperature at which the natural esters will just flow. Pour point shall be measured in accordance with ISO 3016.~~

~~Crystallization behaviour of natural esters depends on time and temperature. Crystals should not be present in liquid at application temperature; precautions shall be taken if oil temperature inside the electrical device is lower than 0 °C. Below this temperature thermal and dielectric behavior of the device with natural esters can be adversely affected. A well-defined method to measure crystallization behavior is not available at present.~~

The pour point of liquids is the lowest temperature at which the liquid will flow. The pour point shall be measured in accordance with ISO 3016.

Longer dwell times (weeks or months) below 0 °C can lead to an increase of the pour point, or, alternatively, to an increase of viscosity of the liquid in comparison to the original state (the so-called "cold-and-hold behaviour"). It depends on the individual molecular structure and formulation and the time the liquid is exposed to the low temperature. While natural ester liquids show pour points in the region of -15 °C to -31 °C, it has been noticed that they can show a tendency to precipitate ester crystals if held for extended periods at temperatures slightly above the pour point. These crystals will re-melt back into the bulk ester liquid with no side effects once the mixture has been warmed up, but this can take time. For references see the bibliography.

Precautions shall be taken if the electrical equipment is to be left or stored at continuously low temperatures below 0 °C. The possible partial crystallization can result in an unforeseeable and undetermined increase in viscosity, which can adversely affect the mechanical, thermal, and dielectric behaviour of the device immersed in natural ester liquid.

An acknowledged method to measure the effect of crystallization behaviour is not available at present. As a substitute, additional viscosity measurement at low temperatures according to IEC 61868 is recommended, using standing times of 24 h, 72 h and 168 h. The lowest cold start energizing temperature (LCSET) is the temperature which, after a defined holding time, leads to a viscosity equal to or lower than the maximum admissible viscosity needed for the proper function of the equipment. During the normal operation of the equipment, no crystals shall be present in the liquid.

Tap-changers: It is common practice that, before energizing, the on-load tap-changer (if present) is operated to reach a position where the transformer can be energized without carrying an abnormally large load. It can happen that the spring-operated diverter switch cannot complete its operation if the natural ester liquid is partially crystalized. Such situation shall be avoided in any case. **10**

4.2.4 Water content

Water content of natural esters affects their dielectric properties. Water content shall be measured in accordance with IEC 60814.

NOTE 1 Due to the moderately polar nature of natural esters, water content at which free water will appear and cause deterioration of electric strength is significantly higher in natural esters than that in mineral insulating oils.

NOTE 2 The terms “water” and “moisture” are the same in this document.

4.2.5 Density

Density of natural esters shall be measured in accordance with ~~ISO 3675~~ ISO 12185 (reference method), but ~~ISO 12185~~ ISO 3675 or ASTM D7042 are also acceptable.

4.3 Electrical properties

4.3.1 Breakdown voltage

Breakdown voltage of unused natural esters shall be measured in accordance with IEC 60156.

~~Because of the difference in properties of natural esters, an initial set-up time is required; it may range between 15 min and 30 min, when there are no visible bubbles in the liquid before measurements are made.~~ **11**

4.3.2 Dielectric dissipation factor (DDF)

DDF is a measure for dielectric losses caused by the liquid. High DDF can indicate contamination of the liquid with moisture, particles, soluble polar contaminants, or poor refining quality. DDF shall be measured in accordance with IEC 60247 ~~or IEC 61620~~ at 90 °C. ~~In case of dispute, IEC 60247 at 90 °C should be used.~~

By agreement between parties, DDF ~~may~~ can be measured at temperatures other than 90 °C. In such cases the measurement temperature ~~should~~ shall be stated in the report.

4.3.3 Relative permittivity (dielectric constant)

Relative permittivity is the ratio of the amount of electrical energy stored in the liquid at an applied voltage, relative to that stored in a vacuum. It shall be measured in accordance with IEC 60247 or IEC 61620 at 90 °C. In case of dispute, IEC 60247 (reference method) at 90 °C ~~should~~ shall be used.

NOTE A typical value is between 2,8 and 3,3.

4.4 Chemical properties

4.4.1 Acidity

Unused natural esters should be near neutral; acidity shall be measured in accordance with IEC 62021-3.

NOTE Natural esters ~~may~~ can contain very low concentrations of free fatty acids; presence of free fatty acids can affect the acidity of natural esters. This is reflected in the values given in Table 2.

4.4.2 Corrosive sulfur

~~Free~~ Corrosive sulfur and potentially corrosive compounds ~~are~~ shall be detected by contacting copper with insulating liquid under the standardized conditions specified in IEC 62535 ~~or ASTM D1275B~~. Known corrosive sulfur compounds such as dibenzyl disulphide (DBDS) shall not be present above the detection limit given in IEC 62697-1.

NOTE Corrosive sulfur compounds are not naturally present in vegetable oils or other natural esters. The tests given in IEC 62535 can verify that any additives used are non-corrosive and that cross-contamination with a potentially corrosive ~~oils~~ liquid has not occurred.

4.4.3 Additive content

Additives include antioxidants, metal deactivators, pour point depressants, dyes, etc. An antioxidant additive slows down the oxidation of esters and, in turn, the formation of gels and acidity. ~~One such antioxidant is 2, 6-di-tert-butyl-p-cresol (DBPC), also known as BHT, but others are also used.~~ Detection and measurement of defined anti-oxidant additives shall be in

accordance with IEC 60666 or other suitable methods. Total concentration of additives shall be less than a weight fraction of 5 %.

~~The supplier with mutual consent should declare the generic types of all additives, and their concentrations in the case of antioxidants and passivators. Information on initial type and concentration of additives is useful for supervision and maintenance guidance during the life of natural esters in transformers and similar electrical equipment.~~

The chemical family and function of all additives and maximum concentration of each family shall be declared in product data sheets and certificates of compliance. **12**

NOTE Example of additives are DBPC = 2,6-di-tert-butyl-para-cresol (also known as BHT); DBP = 2,6-di-tert-butyl-phenol, Irganox®109, Irganox® 1010, Irgamet® 39, Irgamet ®30[3], methacrylate.

4.4.4 Furfural content

Furanic compounds, including 2-Furfural, are degradation products of kraft insulating paper; such compounds are not typically present in unused natural esters. ~~2-Furfural and related compounds shall be determined in accordance with IEC 61198.~~ Furfural compounds shall be determined via an adapted procedure for ester after revision of IEC 61198.

~~NOTE Certain furanic compounds may be present at trace levels in unused natural esters.~~

4.5 Performance

4.5.1 General

~~NOTE~~ This concerns the properties that are related to the long-term behaviour of natural ester insulating ~~fluids~~ liquids in service ~~and/~~or their reaction to high electric stress and temperature, or both. Acceptable operating temperatures for transformers filled with natural esters are ~~provided~~ suggested in IEC 60076-14.

4.5.2 Oxidation stability

4.5.2.1 General

Unused natural ester liquids are recommended for application only in equipment that is not open to atmosphere because these ~~fluids~~ liquids are prone to ~~rapid~~ oxidation. ~~Oxidation stability of these fluids can be assessed with modifications to the procedure as described in Method C of IEC 61125:1992.~~ Oxidation stability of natural esters shall be tested in accordance with IEC 61125 with a test duration of 48 h. The acceptable final limits are indicated in Table 2. See Annex A. **13**

~~NOTE The modifications in IEC 61125 for natural esters are given in Annex A.~~

4.5.2.2 Total acidity

~~Acidity of natural esters subjected to oxidation stability test. Acidity should be measured in accordance with 1.9.4 of IEC 61125:1992.~~

Total acidity is the sum of volatile and soluble acidity of natural esters subjected to the oxidation stability test as specified in IEC 61125.

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[3] Irganox®109, Irganox® 1010, Irgamet® 39, Irgamet ®30 are the trade names of products supplied by BASF. 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.

4.5.2.3 Viscosity

Viscosity of natural esters subjected to the oxidation stability test shall be measured at 40 °C in accordance with ISO 3104 (reference method) or ASTM D7042.

4.5.2.4 Dielectric dissipation factor (DDF)

DDF measurements after the oxidation stability test provide a measure for dielectric losses resulting from the water and soluble polar compounds formed in a dielectric liquid as a result of oxidation. DDF shall be measured in accordance with IEC 60247 ~~or IEC 61620~~.

4.6 Health, safety and environmental (HSE) properties

~~NOTE These are the properties that are related to safe handling of natural esters and minimization of their adverse impact. Examples can include flash and fire points, polycyclic aromatics (PCAs), and polychlorinated biphenyls/polychlorinated terphenyls (PCBs/PCTs).~~

4.6.1 Fire point and flash point

The safe operation of electrical equipment requires an adequately high flash and fire points that ~~is~~ shall be measured in accordance with ISO 2592 (reference method) or ASTM D92 **14**. ~~Flash point is measured according to ISO 2719.~~

4.6.2 Polychlorinated biphenyls (PCBs)

Unused natural esters shall be free from PCBs.

~~Concentrations of these chemicals can be measured according to IEC 61619; total concentration shall be less than 2 mg kg^{-1}.~~

The reference test method that shall be used for these materials is as specified in IEC 61619.

The test method has been developed for mineral oil. Using it for esters could require modification. **15**

NOTE 1 PCBs and related compounds can be present in unused natural esters only because of cross-contamination.

NOTE 2 Acceptable limits of total or individual PCBs are specified in national or local regulations. European specifications are described in Directive 96/59/EC and UN guideline for the identification of PCBs and materials containing PCBs.

4.6.3 Biodegradation

~~Natural esters exhibit better environmental compatibility relative to petroleum-derived insulating mineral oils. Specific tests need to be undertaken to demonstrate ready biodegradability of these fluids. Tests include OECD 301B, C or F; or US EPA – OPPTS 835.311.~~

~~NOTE Natural esters can be classified in accordance with IEC 61039, based on biodegradability observed with OECD 301:1992.~~

Natural ester insulating liquids exhibit lower environmental impact relative to most mineral insulating liquids. Specific tests need to be undertaken to demonstrate ready biodegradability of these liquids. Tests include OECD 301B, C or F. These tests are based on natural materials (inoculum) and hence have a certain variability. Biodegradation tests are defined in IEC 61039.

4.6.4 Aquatic toxicity **16**

Unused natural ester liquids are generally considered non-toxic to aquatic life in the case of accidental release. Suppliers shall supply assays that define the product as non-toxic.

National regulations dealing with the impact on the environment of the disposal of natural esters, chemicals and sample containers mentioned in this document can apply. Every precaution should be taken to prevent the release of natural esters into the environment. 17

NOTE ~~Toxicity of natural esters can be assessed with test methods such as a modified Ames test or other suitable internationally recognized assays such as OECD 201-203; US EPA 600/4.82.068:1983.~~ Aquatic toxicity of natural esters can be assessed using test methods such as OECD 201-203.

5 Classification, identification, general delivery requirements, and sampling

5.1 Classification

Natural esters conforming to this document are classified in a single class:

– less flammable natural ester dielectric liquids.

NOTE ~~There are other natural ester derived liquids, which may have a different classification. However, these liquids are not covered by this standard, an example of such liquids is described in Annex B.~~ Other ester insulating liquids not covered by this document or IEC 61099 are covered by IEC 63012.

5.2 Identification and general delivery requirements

a) Natural esters are normally delivered in bulk, rail tank cars, tank containers, or packed in drums or intermediate bulk containers (IBCs). These shall be clean and suitable for this purpose in order to avoid any contamination.

b) Liquid drums and sample containers shall carry ~~at least~~ the following markings:
   – supplier's designation;
   – classification; and
   – liquid net weight.

c) Each natural ester delivery shall be accompanied by a document from the supplier specifying ~~at least~~:
   – supplier's designation;
   – liquid classification; and
   – quality certificate. ~~At the request of the purchaser and by mutual consent, the supplier should declare all generic types of additives and their concentrations in a datasheet in accordance with the international and local regulations.~~

5.3 Sampling

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

Table 2 – General specifications 18

Property	Test method	Limits
Physical		
Appearance		Clear, free from sediment and suspended matter
Colour	ISO 2049 (reference method), ASTM D1500	Max. 1,0
Viscosity at 100 °C	ISO 3104 (reference method) ASTM D7042	Max. 15 $mm^2 \cdot s^{-1}$
Viscosity at 40 °C	ISO 3104 (reference method) ASTM D7042	Max. 50 $mm^2 \cdot s^{-1}$
Pour point	ISO 3016	Max. –10 °C
Water content	IEC 60814	Max. 200 $mg \cdot kg^{-1}$
Density at 20 °C	ISO 12185 (reference method) ISO 3675 or ASTM D7042	Max. 1 000 $kg \cdot m^{-3}$
Electrical		
Breakdown voltage	IEC 60156 ~~(2,5 mm gap)~~	Min. 35 kV [a]
Dissipation factor (tan δ) 90 °C	IEC 61620 or IEC 60247 (reference method)	Max. 0,05
Chemical		
~~Soluble~~ Acidity	IEC 62021-3	Max. 0,06 mg KOH g_{oil}^{-1}
Corrosive sulfur	IEC 62535 ~~or ASTM D1275B~~	Non corrosive
DBDS	IEC 62697-1	Below detection limit
Total additives	IEC 60666 or other suitable methods	Max. weight fraction 5 %
Performance – ~~Salient~~ Required properties after oxidation stability test in accordance with ~~Method C of~~ IEC 61125~~:1992~~ [b]		
Total acidity	~~1.9.4 of~~ IEC 61125~~:1992~~	Max. 0,6 mg KOH g_{oil}^{-1}
Viscosity at 40 °C	ISO 3104 (reference method) ASTM D7042	Max. 30 % increase over the initial value
DDF (tan δ) at 90 °C	IEC 60247	Max. 0,5
Health, safety and environment (HSE)		
Fire point	ISO 2592 (reference method) ASTM D92	Min. 300 °C
Flash point	~~ISO 2719~~ ISO 2592 (reference method) ASTM D92	Min. 250 °C
Biodegradation	US EPA, OECD 301 B, C or F ~~US EPA OPPTS 835.311~~	Readily biodegradable

[a] At delivery. Expect >70 kV after lab treating, for example degassing and drying

[b] See Annex A for details of oxidation stability parameters.

Annex A
(normative)

Summary of the test method for evaluating oxidation stability of unused natural esters

A.1 ~~Introductory remark~~ General

Oxidation stability of natural esters is evaluated under accelerated ageing conditions similar to those described in IEC 61125~~:1992~~.

Aliquots of the natural esters samples are maintained at 120 °C in the presence of a solid copper catalyst, while a constant volume of air is bubbled through the samples for 48 h. The resistance to oxidation is estimated by measuring volatile acidity, soluble acidity, sludge formation, viscosity and DDF. The values obtained for these parameters after accelerated oxidation are compared against the values obtained prior to accelerated oxidation.

A.2 Test conditions

All test conditions, i.e. the amount of natural esters, length and diameter of copper catalyst, oxidation temperature and oxidant (air) flow rate are the same as described in IEC 61125~~:1992~~. The only modification is in the duration for accelerated ageing, which is set at 48 h.

A.3 Precision

Precision values obtained during the round robin test (RRT) on commercially available natural esters after 48 h oxidation are given in Table A.1. Relative reproducibility for each parameter is based on results obtained from 11 participating laboratories. The values reported in Table A.1 are in general agreement with values reported for mineral insulating oils in IEC 61125.

A.4 Relative repeatability (r)

Duplicate determinations carried out by one laboratory at the 95 % confidence level.

A.5 Relative reproducibility (R)

Duplicate determinations carried out by different laboratories at the 95 % confidence level.

Table A.1 – Relative repeatability and relative reproducibility obtained for different parameters during RRT

Parameter	r %	R %
Viscosity at 40 °C	5	7,5
Total acidity	13	38
Sludge	22	57
DDF (tan δ) at 90 °C	–	47

Annex B 19
(informative)

Specifications of low-viscosity insulating fluids derived from natural esters

From a chemical and biochemical point of view, the natural esters (i.e. esters that can be found in biological materials) comprise many other molecules besides the triglycerides to which the definition of natural esters in this standard refers.

The triglycerides are the main constituents of vegetable oils (and animal fats) and these fluids are usually characterized by high fire point and flash point, which categorize them in class K according to IEC 61100. Compared to mineral oils, they are less flammable but they have also a higher viscosity and a higher pour point.

NOTE According to IEC 61100, class K fluids have a fire point > 300 °C (according to ISO 2592 – open cup) and a flash point > 250 °C (according to ISO 2719 – closed cup)

Other insulating fluids, derived from various natural esters, have been developed for use in some electrotechnical applications (e.g. low/medium voltage power transformers with ONAN cooling). Such fluids mainly consist of fatty acid mono-esters that allow lowering the viscosity or mixtures of triglycerides and fatty acid mono-esters.

The properties of the low viscosity insulating fluids derived from natural esters are close to those of mineral oils, as shown in Table B.1.

Table B.1 – Specifications for low-viscosity of monoesters derived from natural esters

Property	Test method	Limits
Physical		
Viscosity at 100 °C	ISO 3104	Max. 6 $mm^2 \cdot s^{-1}$
Viscosity at 40 °C	ISO 3104	Max. 18 $mm^2 \cdot s^{-1}$
Viscosity at 0 °C	ISO 3104	Max. 90 $mm^2 \cdot s^{-1}$
Pour point	ISO 3016	Max. –25 °C [a]
Electrical		
Breakdown voltage	IEC 60156 (2,5 mm gap)	Min. 35 kV [b]
Dissipation factor (tan δ) at 90 °C	IEC 60247	Max. 0,05
Health, safety and environment (HSE)		
Fire point	ISO 2592 (open cup)	Min. 175 °C
Flash point	ISO 2719 (closed cup)	Min. 135 °C
Biodegradation	US EPA OECD 301 B,C,F US EPA OPPTS 835.311	Readily biodegradable

[a] LCSET Lowest cold start energizing temperature – 10 K.

[b] At delivery and ≥70 kV after treatment (see IEC 60296 for a description of the laboratory treatment).

Bibliography 20

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~~IEC 60050-212:2010, *International Electrotechnical Vocabulary – Part 212: Electrical insulating solids, liquids and gases*~~

~~IEC 60050-421, *International Electrotechnical Vocabulary – Chapter 421: Power transformers and reactors*~~

IEC 60076-14, *Power transformers – Part 14: Liquid-immersed power transformers using high-temperature insulation materials*

IEC 60296, *Fluids for electrotechnical applications – Mineral insulating oils for electrical equipment*

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

IEC 61039, *Classification of insulating fluids*

IEC 61099, *Insulating liquids – Specifications for unused synthetic organic esters for electrical purposes*

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

IEC 62975:2021, *Natural esters – Guidelines for maintenance and use in electrical equipment*

IEC 63012, *Insulating liquids – Unused modified or blended esters for electrotechnical applications*

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International Union of Pure and Applied Chemistry (IUPAC) Handbook of terms and definitions

OECD 201-203, *Test Guidelines for ecotoxicity*

OECD 301, *Guideline for testing of chemicals adopted by European Council on July 17th 1992*

UN Guidelines for the Identification of PCBs and Materials Containing PCBs, 1999

US EPA, *Office of Prevention, Pesticides and Toxic Substances 835.311, Fate, Transport and Transformation Test Guidelines*

List of comments

1 The list of changes from the 2013 edition is added to clarify the advances in this standard.

2 Disposal advice for natural esters is moved to Subclause 4.6.4 Aquatic toxicity.

3 The scope has not changed but extra wording is added for increased clarity. “Fluid” is replaced by “liquid” throughout since “fluid” may also describe gases which are not part of this standard.

4 Annex B is removed and now replaced by IEC 63012 (2019) which was not published at the time of the 2013 edition of this standard.

5 The normative references are updated to reflect the current standard status.

6 Information about IEC/ISO terminology is added in this edition of the standard.

7 Table 1 is added to clarify these terms.

8 A new note is added regarding stray gas generation which has been noted in natural esters in more recent years.

9 The relevance of K class added in to viscosity is highlighted since it has an effect on the cooling properties of the liquid. The measurement of viscosity are adapted to align with IEC 60296.

10 A detailed description of the low temperature behaviour of natural ester is added.

11 The new edition of IEC 60156 now includes mention of increased rest time between samples for higher viscosity liquids hence this advice is unnecessary in this standard.

12 New requirement for additive content is added including dyes for transparency.

13 Oxidation stability is transferred to the main text as it was previously only in Annex A.

14 Open cup testing for flash points replaces closed cup since it is more reliable than closed cup.

15 IEC 61619 will be developed to be adapted for PCB in esters.

16 A more precise definition of toxicity involving aquatic environment is added. “Aquatic toxicity” replaces “toxicity”.

17 This chapter referring to national regulation is added for clarity.

18 When more that one measurement method is included in Table 2, the reference method is always indicated.

19 Annex B is deleted as the information has been replaced by IEC 63012.

20 Bibliography is updated to reflect the current literature.

IEC 62770

Edition 2.0 2024-10

INTERNATIONAL STANDARD

NORME INTERNATIONALE

Fluids for electrotechnical applications – Unused natural esters for transformers and similar electrical equipment

Fluides pour applications électrotechniques – Esters naturels neufs pour transformateurs et matériels électriques analogues

IEC 62770:2024-10(en-fr)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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FLUIDS FOR ELECTROTECHNICAL APPLICATIONS – UNUSED NATURAL ESTERS FOR TRANSFORMERS AND SIMILAR ELECTRICAL EQUIPMENT

FOREWORD

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IEC 62770 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications. It is an International Standard.

This second edition cancels and replaces the first edition published in 2013. This edition constitutes a technical revision.

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

a) Introduction of IEC 63012 which details other liquids not covered by this document. IEC 63012 was published in 2019 after the first edition of IEC 62770 (2013).
b) New Table 1 inserted which clarifies definitions.
c) Appearance and colour requirements now merged.

d) Pour point: Introduction of the importance of LCSET with advice on cold temperature behaviour of natural esters.

e) Additives: new agreed wording inserted on the declaration of additives

f) Flash and fire points: now only determined by Cleveland Open Cup method, since the Pensky-Martens closed cup method was identified as problematic with natural esters.

g) Toxicity: Aquatic toxicity now emphasized.

h) Annex B removed as it is no longer needed since the publication of IEC 63012.

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

Draft	Report on voting
10/1215/FDIS	10/1243/RVD

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 International Standard 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/publications.

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, or
- revised.

INTRODUCTION

Because of their higher fire points and lower environmental impact relative to hydrocarbon petroleum derived insulating mineral oil, the use of vegetable oils and other natural esters is on the rise as insulating and heat transfer fluids in electrical devices such as transformers.

This document sets performance criteria for unused natural esters earmarked for electrical applications. However, the use of natural esters is recommended only for equipment that is not open to the atmosphere, for example sealed transformers and reactors because these liquids are susceptible to oxidation.

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

Unused natural esters which are the subject of this document should be handled with due regard to personal hygiene. Direct contact with eyes should be avoided. In case of eye contact, irrigation with copious amounts of clean running water should be carried out and medical advice sought.

Performance of some of the tests mentioned in this document could lead to a hazardous situation. Attention is drawn to the relevant document test method for guidance.

FLUIDS FOR ELECTROTECHNICAL APPLICATIONS – UNUSED NATURAL ESTERS FOR TRANSFORMERS AND SIMILAR ELECTRICAL EQUIPMENT

1 Scope

This document describes specifications and test methods for unused natural esters in transformers and similar liquid-immersed electrical equipment in which a liquid is required as an insulating and heat transfer medium. The exposure of natural ester to air leads to deterioration of the insulating liquid. Use of natural esters is therefore restricted to sealed units, or with the conservator tank protected from the contact with atmosphere by a membrane or other suitable system.

In this document the term "natural esters" applies to insulating liquids for transformers and similar electrical equipment with suitable biodegradability and lower environmental impact. Such natural esters are vegetable oils obtained from seeds, and oils obtained from other suitable biological materials. These oils are comprised of triglycerides.

Natural esters with additives are within the scope of this document. Because of their different chemical composition, natural esters differ from insulating mineral oils and other insulating liquids that have high fire points, such as synthetic esters or silicone fluids.

Natural ester-derived insulating liquids with low viscosity have been introduced but are not covered by this document. IEC 63012 covers these liquids.

This document is applicable only to unused natural esters. Reclaimed natural esters and natural esters blended with other insulating liquids are beyond the scope of this document.

NOTE The chemical nomenclature and scientific notations used in the document are in accordance with the IUPAC handbook (Quantities, Units and Symbols in Physical Chemistry).

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 d) and d.c. resistivity*

IEC 60475, *Method of sampling insulating liquids*

IEC 60666, *Detection and determination of specific additives in mineral insulating oils*

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

IEC 61125, *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 62021-3, *Insulating liquids – Determination of acidity – Part 3: Test methods for non-mineral insulating oils*

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

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)*

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

ISO 2592, *Petroleum and related products – Determination of flash and fire points – Cleveland open cup method*

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

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

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

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

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

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

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

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

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

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

3.1.1
additives
chemical substances which are deliberately added to natural ester insulating liquids in order to improve certain characteristics

3.1.2
natural esters
vegetable oils obtained from seeds and oils obtained from other suitable biological materials and comprised of triglycerides

3.1.3
unused natural esters
natural esters as delivered by the supplier

Note 1 to entry: Such a liquid has not been used in, nor been in contact with, electrical equipment or other equipment not required for its manufacture, storage or transport.

3.2 Abbreviated terms

The abbreviated terms are given in Table 1.

Table 1 – Abbreviated terms

Abbreviated term	Full term
DBDS	Dibenzyl disulphide
DDF	Dielectric dissipation factor
HSE	Health, safety and environment
IBC	Intermediate bulk container
LCSET	Lowest cold start energizing temperature
PCBs	Polychlorinated biphenyls
RRT	Round robin test

4 Properties, their significance and test methods

4.1 General

Required characteristics of unused natural esters are listed in Table 2.

NOTE 1 Additional information on natural esters for transformers and similar electrical equipment is available in CIGRE brochure 436 and IEEE C57.147-2018™.

NOTE 2 In natural ester at normal operating temperatures, a significant formation of stray gases such as hydrogen and ethane is sometimes witnessed for a specific period (weeks to months) after the transformer is activated. Such unexpected gas formation at low temperature can lead to confusion in the dissolved gas analyses interpretation. Natural esters show a higher tendency towards stray gassing.

4.2 Physical properties

4.2.1 Appearance and colour

A visual inspection of unused natural esters (with light transmitted through a glass transparent beaker of approximately 10 cm thickness at ambient temperature) will indicate the presence of visible contaminants, free water and suspended matter.

The colour of an insulating liquid 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.

4.2.2 Viscosity

Viscosity influences heat transfer and therefore affects temperature distribution in the transformer and other equipment. The lower the viscosity, the easier the liquid circulates, generally leading to better heat transfer. Viscosities at lower temperatures are a critical factor for the cold start of transformers with K class liquid insulation with natural (not forced/pumped) flow (the absence of circulation can lead to possible overheating at hot spots). It can have a negative impact on the speed of moving parts, such as on-load tap changer mechanisms, pumps and regulators. Due consideration should be given to viscosity at the lowest cold start energizing temperature (LCSET). Viscosity at 40 °C and 100 °C shall be measured according to ISO 3104 (referee method) or ASTM D7042

4.2.3 Pour point

The pour point of liquids is the lowest temperature at which the liquid will flow. The pour point shall be measured in accordance with ISO 3016.

Longer dwell times (weeks or months) below 0 °C can lead to an increase of the pour point, or, alternatively, to an increase of viscosity of the liquid in comparison to the original state (the so-called "cold-and-hold behaviour"). It depends on the individual molecular structure and formulation and the time the liquid is exposed to the low temperature. While natural ester liquids show pour points in the region of -15 °C to -31 °C, it has been noticed that they can show a tendency to precipitate ester crystals if held for extended periods at temperatures slightly above the pour point. These crystals will re-melt back into the bulk ester liquid with no side effects once the mixture has been warmed up, but this can take time. For references see the bibliography.

Precautions shall be taken if the electrical equipment is to be left or stored at continuously low temperatures below 0 °C. The possible partial crystallization can result in an unforeseeable and undetermined increase in viscosity, which can adversely affect the mechanical, thermal, and dielectric behaviour of the device immersed in natural ester liquid.

An acknowledged method to measure the effect of crystallization behaviour is not available at present. As a substitute, additional viscosity measurement at low temperatures according to IEC 61868 is recommended, using standing times of 24 h, 72 h and 168 h. The lowest cold start energizing temperature (LCSET) is the temperature which, after a defined holding time, leads to a viscosity equal to or lower than the maximum admissible viscosity needed for the proper function of the equipment. During the normal operation of the equipment, no crystals shall be present in the liquid.

Tap-changers: It is common practice that, before energizing, the on-load tap-changer (if present) is operated to reach a position where the transformer can be energized without carrying an abnormally large load. It can happen that the spring-operated diverter switch cannot complete its operation if the natural ester liquid is partially crystalized. Such situation shall be avoided in any case.

4.2.4 Water content

Water content of natural esters affects their dielectric properties. Water content shall be measured in accordance with IEC 60814.

NOTE 1 Due to the moderately polar nature of natural esters, water content at which free water will appear and cause deterioration of electric strength is significantly higher in natural esters than that in mineral insulating oils.

NOTE 2 The terms “water” and “moisture” are the same in this document.

4.2.5 Density

Density of natural esters shall be measured in accordance with ISO 12185 (reference method), but ISO 3675 or ASTM D7042 are also acceptable.

4.3 Electrical properties

4.3.1 Breakdown voltage

Breakdown voltage of unused natural esters shall be measured in accordance with IEC 60156.

4.3.2 Dielectric dissipation factor (DDF)

DDF is a measure for dielectric losses caused by the liquid. High DDF can indicate contamination of the liquid with moisture, particles, soluble polar contaminants, or poor refining quality. DDF shall be measured in accordance with IEC 60247 at 90 °C.

By agreement between parties, DDF can be measured at temperatures other than 90 °C. In such cases the measurement temperature shall be stated in the report.

4.3.3 Relative permittivity (dielectric constant)

Relative permittivity is the ratio of the amount of electrical energy stored in the liquid at an applied voltage, relative to that stored in a vacuum. It shall be measured in accordance with IEC 60247 or IEC 61620 at 90 °C. In case of dispute, IEC 60247 (reference method) at 90 °C shall be used.

NOTE A typical value is between 2,8 and 3,3.

4.4 Chemical properties

4.4.1 Acidity

Unused natural esters should be near neutral; acidity shall be measured in accordance with IEC 62021-3.

NOTE Natural esters can contain very low concentrations of free fatty acids; presence of free fatty acids can affect the acidity of natural esters. This is reflected in the values given in Table 2.

4.4.2 Corrosive sulfur

Corrosive sulfur and potentially corrosive compounds shall be detected by contacting copper with insulating liquid under the standardized conditions specified in IEC 62535. Known corrosive sulfur compounds such as dibenzyl disulphide (DBDS) shall not be present above the detection limit given in IEC 62697-1.

NOTE Corrosive sulfur compounds are not naturally present in vegetable oils or other natural esters. The tests given in IEC 62535 can verify that any additives used are non-corrosive and that cross-contamination with a potentially corrosive liquid has not occurred.

4.4.3 Additive content

Additives include antioxidants, metal deactivators, pour point depressants, dyes, etc. An antioxidant additive slows down the oxidation of esters and, in turn, the formation of gels and acidity. Detection and measurement of defined anti-oxidant additives shall be in accordance with IEC 60666 or other suitable methods. Total concentration of additives shall be less than a weight fraction of 5 %.

The chemical family and function of all additives and maximum concentration of each family shall be declared in product data sheets and certificates of compliance.

NOTE Example of additives are DBPC = 2,6-di-tert-butyl-para-cresol (also known as BHT); DBP = 2,6-di-tert-butyl-phenol, Irganox®109, Irganox® 1010, Irgamet® 39, Irgamet ®30[1], methacrylate.

4.4.4 Furfural content

Furanic compounds, including 2-Furfural, are degradation products of kraft insulating paper; such compounds are not typically present in unused natural esters. Furfural compounds shall be determined via an adapted procedure for ester after revision of IEC 61198.

4.5 Performance

4.5.1 General

This concerns the properties that are related to the long-term behaviour of natural ester insulating liquids in service or their reaction to high electric stress and temperature, or both. Acceptable operating temperatures for transformers filled with natural esters are suggested in IEC 60076-14.

4.5.2 Oxidation stability

4.5.2.1 General

Unused natural ester liquids are recommended for application only in equipment that is not open to atmosphere because these liquids are prone to oxidation. Oxidation stability of natural esters shall be tested in accordance with IEC 61125 with a test duration of 48 h. The acceptable final limits are indicated in Table 2. See Annex A.

4.5.2.2 Total acidity

Total acidity is the sum of volatile and soluble acidity of natural esters subjected to the oxidation stability test as specified in IEC 61125.

4.5.2.3 Viscosity

Viscosity of natural esters subjected to the oxidation stability test shall be measured at 40 °C in accordance with ISO 3104 (reference method) or ASTM D7042.

4.5.2.4 Dielectric dissipation factor (DDF)

DDF measurements after the oxidation stability test provide a measure for dielectric losses resulting from the water and soluble polar compounds formed in a dielectric liquid as a result of oxidation. DDF shall be measured in accordance with IEC 60247.

4.6 Health, safety and environmental (HSE) properties

4.6.1 Fire point and flash point

The safe operation of electrical equipment requires an adequately high flash and fire points that shall be measured in accordance with ISO 2592 (reference method) or ASTM D92.

4.6.2 Polychlorinated biphenyls (PCBs)

Unused natural esters shall be free from PCBs.

The reference test method that shall be used for these materials is as specified in IEC 61619.

[1] Irganox®109, Irganox® 1010, Irgamet® 39, Irgamet ®30 are the trade names of products supplied by BASF. 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.

The test method has been developed for mineral oil. Using it for esters could require modification.

NOTE 1 PCBs and related compounds can be present in unused natural esters only because of cross-contamination.

NOTE 2 Acceptable limits of total or individual PCBs are specified in national or local regulations. European specifications are described in Directive 96/59/EC and UN guideline for the identification of PCBs and materials containing PCBs.

4.6.3 Biodegradation

Natural ester insulating liquids exhibit lower environmental impact relative to most mineral insulating liquids. Specific tests need to be undertaken to demonstrate ready biodegradability of these liquids. Tests include OECD 301B, C or F. These tests are based on natural materials (inoculum) and hence have a certain variability. Biodegradation tests are defined in IEC 61039.

4.6.4 Aquatic toxicity

Unused natural ester liquids are generally considered non-toxic to aquatic life in the case of accidental release. Suppliers shall supply assays that define the product as non-toxic.

National regulations dealing with the impact on the environment of the disposal of natural esters, chemicals and sample containers mentioned in this document can apply. Every precaution should be taken to prevent the release of natural esters into the environment.

NOTE Aquatic toxicity of natural esters can be assessed using test methods such as OECD 201-203.

5 Classification, identification, general delivery requirements, and sampling

5.1 Classification

Natural esters conforming to this document are classified in a single class:

– less flammable natural ester dielectric liquids.

NOTE Other ester insulating liquids not covered by this document or IEC 61099 are covered by IEC 63012.

5.2 Identification and general delivery requirements

a) Natural esters are normally delivered in bulk, rail tank cars, tank containers, or packed in drums or intermediate bulk containers (IBCs). These shall be clean and suitable for this purpose in order to avoid any contamination.

b) Liquid drums and sample containers shall carry the following markings:
   – supplier’s designation;
   – classification; and
   – liquid net weight.

c) Each natural ester delivery shall be accompanied by a document from the supplier specifying:
   – supplier’s designation;
   – liquid classification; and
   – quality certificate.

5.3 Sampling

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

Table 2 – General specifications

Property	Test method	Limits
Physical		
Appearance		Clear, free from sediment and suspended matter
Colour	ISO 2049 (reference method), ASTM D1500	Max. 1,0
Viscosity at 100 °C	ISO 3104 (reference method) ASTM D7042	Max. 15 $mm^2 \cdot s^{-1}$
Viscosity at 40 °C	ISO 3104 (reference method) ASTM D7042	Max. 50 $mm^2 \cdot s^{-1}$
Pour point	ISO 3016	Max. –10 °C
Water content	IEC 60814	Max. 200 $mg \cdot kg^{-1}$
Density at 20 °C	ISO 12185 (reference method) ISO 3675 or ASTM D7042	Max. 1 000 $kg \cdot m^{-3}$
Electrical		
Breakdown voltage	IEC 60156	Min. 35 kV [a]
Dissipation factor (tan δ) 90 °C	IEC 61620 or IEC 60247 (reference method)	Max. 0,05
Chemical		
Acidity	IEC 62021-3	Max. 0,06 mg KOH g_{oil}^{-1}
Corrosive sulfur	IEC 62535	Noncorrosive
DBDS	IEC 62697-1	Below detection limit
Total additives	IEC 60666 or other suitable methods	Max. weight fraction 5 %
Performance – Required properties after oxidation stability test in accordance with IEC 61125 [b]		
Total acidity	IEC 61125	Max. 0,6 mg KOH g_{oil}^{-1}
Viscosity at 40 °C	ISO 3104 (reference method) ASTM D7042	Max. 30 % increase over the initial value
DDF (tan δ) at 90 °C	IEC 60247	Max. 0,5
Health, safety and environment (HSE)		
Fire point	ISO 2592 (reference method) ASTM D92	Min. 300 °C
Flash point	ISO 2592 (reference method) ASTM D92	Min. 250 °C
Biodegradation	US EPA, OECD 301 B, C or F	Readily biodegradable

[a] At delivery. Expect >70 kV after lab treating, for example degassing and drying.

[b] See Annex A for details of oxidation stability parameters.

Annex A
(normative)

Summary of the test method for evaluating oxidation stability of unused natural esters

A.1 General

Oxidation stability of natural esters is evaluated under accelerated ageing conditions similar to those described in IEC 61125.

Aliquots of the natural esters samples are maintained at 120 °C in the presence of a solid copper catalyst, while a constant volume of air is bubbled through the samples for 48 h. The resistance to oxidation is estimated by measuring volatile acidity, soluble acidity, sludge formation, viscosity and DDF. The values obtained for these parameters after accelerated oxidation are compared against the values obtained prior to accelerated oxidation.

A.2 Test conditions

All test conditions, i.e. the amount of natural esters, length and diameter of copper catalyst, oxidation temperature and oxidant (air) flow rate are the same as described in IEC 61125. The only modification is in the duration for accelerated ageing, which is set at 48 h.

A.3 Precision

Precision values obtained during the round robin test (RRT) on commercially available natural esters after 48 h oxidation are given in Table A.1. Relative reproducibility for each parameter is based on results obtained from 11 participating laboratories. The values reported in Table A.1 are in general agreement with values reported for mineral insulating oils in IEC 61125.

A.4 Relative repeatability (r)

Duplicate determinations carried out by one laboratory at the 95 % confidence level.

A.5 Relative reproducibility (R)

Duplicate determinations carried out by different laboratories at the 95 % confidence level.

Table A.1 – Relative repeatability and relative reproducibility obtained for different parameters during RRT

Parameter	r %	R %
Viscosity at 40 °C	5	7,5
Total acidity	13	38
Sludge	22	57
DDF (tan δ) at 90 °C	–	47

Bibliography

IEC 60050, *International Electrotechnical Vocabulary (IEV)* (available at www.electropedia.org)

IEC 60076-14, *Power transformers – Part 14: Liquid-immersed power transformers using high-temperature insulation materials*

IEC 60296, *Fluids for electrotechnical applications – Mineral insulating oils for electrical equipment*

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

IEC 61039, *Classification of insulating fluids*

IEC 61099, *Insulating liquids – Specifications for unused synthetic organic esters for electrical purposes*

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

IEC 62975:2021, *Natural esters – Guidelines for maintenance and use in electrical equipment*

IEC 63012, *Insulating liquids – Unused modified or blended esters for electrotechnical applications*

ASTM D6871, *Standard specification for natural (vegetable oil) ester fluids used in electrical apparatus*

CIGRE-296 2020 CIGRE Canada Conference Toronto, Ontario, October 19-22, 2020“*What it takes to design, manufacture and operate power transformers under arctic temperature conditions”* Thomas Schneider Siemans Energy, Jinesh Malde M&I Materials Inc.

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Council Directive 96/59/EC of 16 September 1996 on the disposal of polychlorinated biphenyls and polychlorinated terphenyls (PCB/PCT)

IEEE C57.147-2018, *IEEE Guide for Acceptance and Maintenance of Natural Ester Insulating Liquid in Transformers*

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OECD 201-203, *Test Guidelines for ecotoxicity*

OECD 301, *Guideline for testing of chemicals adopted by European Council on July 17th 1992*

UN Guidelines for the Identification of PCBs and Materials Containing PCBs, 1999

US EPA, *Office of Prevention, Pesticides and Toxic Substances 835.311, Fate, Transport and Transformation Test Guidelines*

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COMMISSION ÉLECTROTECHNIQUE INTERNATIONALE

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FLUIDES POUR APPLICATIONS ÉLECTROTECHNIQUES – ESTERS NATURELS NEUFS POUR TRANSFORMATEURS ET MATÉRIELS ÉLECTRIQUES ANALOGUES

AVANT-PROPOS

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L'IEC 62770 a été établie par le comité d'études 10 de l'IEC: Fluides pour applications électrotechniques. Il s'agit d'une Norme internationale.

Cette deuxième édition annule et remplace la première édition parue en 2013. Cette édition constitue une révision technique.

Cette édition inclut les modifications techniques majeures suivantes par rapport à l'édition précédente:

a) l'IEC 63012, qui décrit les autres liquides non couverts par le présent document, a été introduite. L'IEC 63012 a été publiée en 2019, après la première édition de l'IEC 62770 (2013);

b) un nouveau Tableau 1, qui clarifie les définitions, a été inséré;

c) les exigences relatives à l'apparence et la couleur sont désormais fusionnées;

d) point d'écoulement: l'importance de la TMDP a été introduite, avec des conseils relatifs au comportement à basse température des esters naturels;

e) additifs: une nouvelle formulation fixée par accord a été insérée dans la déclaration des additifs;

f) points d'éclair et de feu: désormais uniquement déterminés par la méthode Cleveland à vase ouvert, la méthode Pensky-Martens en vase clos ayant été identifiée comme problématique avec les esters naturels;

g) toxicité: la toxicité aquatique est désormais soulignée;

h) l'Annexe B a été supprimée, dans la mesure où elle n'est plus nécessaire depuis la publication de l'IEC 63012.

Le texte de cette Norme internationale est issu des documents suivants:

Projet	Rapport de vote
10/1215/FDIS	10/1243/RVD

Le rapport de vote indiqué dans le tableau ci-dessus donne toute information sur le vote ayant abouti à son approbation.

La langue employée pour l'élaboration de cette Norme internationale est l'anglais.

Ce document a été rédigé selon les Directives ISO/IEC, Partie 2, il a été développé selon les Directives ISO/IEC, Partie 1 et les Directives ISO/IEC, Supplément IEC, disponibles sous www.iec.ch/members_experts/refdocs. Les principaux types de documents développés par l'IEC sont décrits plus en détail sous www.iec.ch/publications.

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INTRODUCTION

Du fait de leurs points de feu plus élevés et de leur impact moindre sur l'environnement par rapport à l'huile minérale isolante obtenue à partir des hydrocarbures pétroliers, l'utilisation des huiles végétales et d'autres esters naturels est en augmentation en tant que liquides isolants et fluides caloporteurs dans les appareils électriques tels que les transformateurs.

Le présent document établit des critères de performance pour les esters naturels neufs destinés aux applications électriques. Cependant, l'utilisation des esters naturels est recommandée uniquement pour les matériels sans contact avec l'air, par exemple les transformateurs et réacteurs scellés, car ces liquides sont susceptibles de s'oxyder.

Le présent document ne prétend pas couvrir tous les problèmes de sécurité liés à son utilisation. Il est de la responsabilité de l'utilisateur du présent document de mettre en place les pratiques d'hygiène et de sécurité adéquates et de déterminer avant utilisation si des contraintes réglementaires s'appliquent.

Il convient de manipuler les esters naturels neufs dont traite le présent document en respectant scrupuleusement l'hygiène personnelle. Il convient d'éviter tout contact direct avec les yeux. En cas de contact oculaire, il convient de laver les yeux abondamment à l'eau courante propre et de demander un avis médical.

Certains des essais mentionnés dans le présent document sont susceptibles d'entraîner une situation dangereuse. L'attention est attirée sur la méthode d'essai du document applicable à titre de recommandation.

FLUIDES POUR APPLICATIONS ÉLECTROTECHNIQUES – ESTERS NATURELS NEUFS POUR TRANSFORMATEURS ET MATÉRIELS ÉLECTRIQUES ANALOGUES

1 Domaine d'application

Le présent document décrit les spécifications et méthodes d'essai applicables aux esters naturels neufs dans les transformateurs et matériels électriques immergés dans du liquide analogues qui nécessitent un liquide en tant que milieu isolant et caloporteur. L'exposition de l'ester naturel à l'air entraîne une détérioration du liquide isolant. L'utilisation des esters naturels est donc limitée à des unités scellées, ou avec le conservateur protégé du contact atmosphérique par une membrane ou tout autre système approprié.

Dans le présent document, le terme "esters naturels" s'applique aux liquides isolants pour transformateurs et matériels électriques analogues qui présentent une biodégradabilité convenable et un impact moindre sur l'environnement. Ces esters naturels sont des huiles végétales obtenues à partir de graines et des huiles obtenues à partir d'autres matériaux biologiques appropriés. Ces huiles sont composées de triglycérides.

Les esters naturels avec additifs entrent dans le domaine d'application du présent document. En raison de leur composition chimique différente, les esters naturels se distinguent des huiles minérales isolantes et d'autres liquides isolants qui possèdent des points de feu élevés, tels que les esters synthétiques ou les fluides silicones.

Les liquides isolants obtenus à partir d'esters naturels qui présentent une faible viscosité ont été introduits, mais ne sont pas couverts par le présent document. L'IEC 63012 couvre ces liquides.

Le présent document ne s'applique qu'aux esters naturels neufs. Les esters naturels régénérés et les esters naturels mélangés à d'autres liquides isolants n'entrent pas dans le domaine d'application du présent document.

NOTE La nomenclature chimique et les notations scientifiques utilisées dans le présent document sont conformes au manuel de l'IUPAC (Grandeurs, unités et symboles de la chimie physique).

2 Références normatives

Les documents suivants sont cités dans le texte de sorte qu'ils constituent, pour tout ou partie de leur contenu, des exigences du présent document. Pour les références datées, seule l'édition citée s'applique. Pour les références non datées, la dernière édition du document de référence s'applique (y compris les éventuels amendements).

IEC 60156, *Isolants liquides – Détermination de la tension de claquage à fréquence industrielle – Méthode d'essai*

IEC 60247, *Liquides isolants – Mesure de la permittivité relative, du facteur de dissipation diélectrique (tan δ) et de la résistivité en courant continu*

IEC 60475, *Méthode d'échantillonnage des liquides isolants*

IEC 60666, *Détection et dosage d'additifs spécifiques présents dans les huiles minérales isolantes*

IEC 60814, *Isolants liquides – Cartons et papiers imprégnés d'huile – Détermination de la teneur en eau par titrage coulométrique de Karl Fischer automatique*

IEC 61125, *Isolants liquides – Méthodes d'essai de la stabilité à l'oxydation – Méthode d'essai pour évaluer la stabilité à l'oxydation des isolants liquides tels que livrés*

IEC 61198, *Huiles minérales isolantes – Méthodes pour la détermination du 2-furfural et ses dérivés*

IEC 61619, *Isolants liquides – Contamination par les polychlorobiphényles (PCB) – Méthode de détermination par chromatographie en phase gazeuse sur colonne capillaire*

IEC 61620, *Isolants liquides – Détermination du facteur de dissipation diélectrique par la mesure de la conductance et de la capacité – Méthode d'essai*

IEC 62021-3, *Liquides isolants – Détermination de l'acidité – Partie 3: Méthodes d'essai pour les huiles non minérales isolantes*

IEC 62535, *Liquides isolants – Méthode d'essai pour la détection du soufre potentiellement corrosif dans les huiles usagées et neuves*

IEC 62697-1, *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)*

ISO 2049, *Produits pétroliers – Détermination de la couleur (échelle ASTM)*

ISO 2592, *Pétrole et produits connexes – Détermination des points d'éclair et de feu – Méthode Cleveland à vase ouvert*

ISO 3016, *Produits pétroliers et connexes d'origine naturelle ou synthétique – Détermination du point d'écoulement*

ISO 3104, *Produits pétroliers – Liquides opaques et transparents – Détermination de la viscosité cinématique et calcul de la viscosité dynamique*

ISO 3675, *Pétrole brut et produits pétroliers liquides – Détermination en laboratoire de la masse volumique – Méthode à l'aréomètre*

ISO 12185, *Pétroles bruts et produits pétroliers – Détermination de la masse volumique – Méthode du tube en U oscillant*

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

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