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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





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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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

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

FDIS	Report on voting
10/909/FDIS	10/933/RVD

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

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

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

- · reconfirmed,
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- · amended.

INTRODUCTION

Because of their higher fire points and better environmental compatibility relative to 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 standard 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, e.g. sealed transformers and reactors because these fluids are prone to rapid oxidation.

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

Unused natural esters which are the subject of this standard 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 standard could lead to a hazardous situation. Attention is drawn to the relevant standard 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.

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

1 Scope

This International Standard describes specifications and test methods for unused natural esters in transformers and similar oil-impregnated electrical equipment in which a liquid is required as an insulating and heat transfer medium.

Use of natural esters is not recommended for electrical equipment that is open to the atmosphere.

In this standard the term "natural esters" applies to insulating fluids for transformers and similar electrical equipment with suitable biodegradability and environmental compatibility. 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 standard. Because of their different chemical composition, natural esters differ from insulating mineral oils and other insulating fluids that have high fire points, such as synthetic esters or silicone fluids.

Natural, ester-derived insulating fluids with low viscosity have been introduced but are not covered by this standard. Pertinent properties of such fluids are given in Annex B.

This standard is applicable only to unused natural esters. Reclaimed natural esters and natural esters blended with non-natural esters fluids are beyond the scope of this standard.

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

2 Normative references

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

IEC 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 and DC 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

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 value1

IEC 61125:1992, Unused hydrocarbon-based insulating fluids - Test methods for evaluating the oxidation stability

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 oils2

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

IEC 62697-1, Test method for quantitative determination of corrosive sulfur compounds in unused and used insulating liquids - Part 1: Test method for quantitative determination of dibenzyl disulfide (DBDS)

ISO 2592, Determination of flash and fire point - Cleveland open cup method

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

ISO 3016, Petroleum products – 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 D 1275, Standard Test Method for Corrosive Sulfur in Electrical Insulating Oils

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

Withdrawn in 2009 and partially replaced by IEC 61039.

To be published.

3 Terms and definitions

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

3.1

additives

suitable chemical substances which are deliberately added to natural ester insulating fluids 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"]

3.3

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]

3.5

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.

4 Properties, their significance and test methods

4.1 General

Salient characteristics of unused natural esters are listed in Table 1.

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

4.2 Physical properties

4.2.1 Appearance

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

4.2.2 Viscosity

Viscosity influences heat transfer and therefore affects the increase of temperature in the transformer and other equipment. The lower the viscosity, the easier the oil circulates leading to better heat transfer. Viscosities at lower temperatures is a critical factor for cold start of transformers with ON cooling (absence of circulation can lead to possible overheating at hot spots). It can have negative impact on the speed of moving parts such as on-load tap changer mechanism, 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.

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 welldefined method to measure crystallization behavior is not available at present.

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 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.

4.2.5 Density

Density of natural esters shall be measured in accordance with ISO 3675 (reference method), but ISO 12185 is 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.

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 or 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 be measured at temperatures other than 90 °C. In such cases the measurement temperature should be stated in the report.

4.3.3 Relative permittivity (dielectric constant)

It 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 at 90 °C should be used.

NOTE Typical value 2,8 - 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 contain very low concentrations of free fatty acids; presence of free fatty acids can affect acidity of natural esters.

4.4.2 Corrosive sulfur

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

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

4.4.3 Additive content

Additives include antioxidants, metal deactivators, pour point depressants, etc. 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.

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.

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

4.5 Performance

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

4.5.1 Oxidation stability

Unused natural esters are recommended for application only in equipment that is not open to atmosphere because these fluids 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.

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

4.5.2 **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.

4.5.3 Viscosity

Viscosity of natural esters subjected to oxidation stability test shall be measured at 40 °C according to ISO 3104.

Dielectric dissipation factor (DDF) 4.5.4

DDF measurements after oxidation stability test provides 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 fire point that is measured in accordance with ISO 2592. 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.

NOTE PCBs and related compounds can be present in unused natural esters only because of crosscontamination.

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.

4.6.4 **Toxicity**

Unused natural esters are considered non-toxic and suppliers shall supply assays that define the product as non-toxic.

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.

Classification, identification, general delivery requirements and sampling

5.1 Classification

For the purpose of this standard, natural esters are classified in a single class.

less flammable natural esters.

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.

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). 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;
 - liquid net weight.
- c) Each natural esters 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 1 – General specifications

Property	Test method	Limits	
Physical			
Appearance		Clear, free from sediment and suspended matter	
Viscosity at 100 °C	ISO 3104	Max. 15 mm ² ·s ⁻¹	
Viscosity at 40 °C	ISO 3104	Max. 50 mm ² ·s ⁻¹	
Pour point	ISO 3016	Max10 °C	
Water content	IEC 60814	Max. 200 mg·kg ⁻¹	
Density at 20 °C	ISO 3675 or ISO 12185	Max. 1 000 kg·m ⁻³	
Electrical			
Breakdown voltage	IEC 60156 (2,5 mm gap)	Min. 35 kV ^a	
Dissipation factor (tan δ) 90 °C	IEC 60247	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 properties	after oxidation stability test in accord	dance 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	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	Min. 300 °C	
Flash point	ISO 2719	Min. 250 °C	
	US EPA OECD 301 B, C or F		
Biodegradation	US EPA OPPTS 835.311	Readily biodegradable	

^a At delivery.

^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

Oxidation stability of natural esters is evaluated under accelerated aging conditions similar to those described in Method C of 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 Method C of IEC 61125:1992. The only modification is in the duration for accelerated aging, which is set at 48 h.

A.3 Precision

Precision values obtained during the Round Robin test 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 (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

Test method	Limits	
ISO 3104	Max. 6 mm ² ·s ⁻¹	
ISO 3104	Max. 18 mm ² ·s ⁻¹	
ISO 3104	Max. 90 mm ² ·s ⁻¹	
ISO 3016 Max. –25 °C ^a		
IEC 60156 (2,5 mm gap)	Min. 35 kV ^b	
IEC 60247	Max. 0,05	
ent (HSE)		
ISO 2592 (open cup)	Min. 175 °C	
ISO 2719 (closed cup)	Min. 135 °C	
US EPA OECD 301 B,C,F US EPA OPPTS 835.311	Readily biodegradable	
	ISO 3104 ISO 3104 ISO 3104 ISO 3104 ISO 3016 IEC 60156 (2,5 mm gap) IEC 60247 ISO 2592 (open cup) ISO 2719 (closed cup) US EPA OECD 301 B,C,F	

Lowest cold start energizing temperature –10 K.

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

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