

BS 148:2009



# BSI British Standards

## Reclaimed mineral insulating oil for transformers and switchgear – Specification

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ISBN 978 0 580 59225 6

ICS 29.040.10

The following BSI references relate to the work on this standard:

Committee reference GEL/10

Draft for comment 08/30167496 DC

**Publication history**

First published April 1923

First revision May 1927

Second revision April 1933

Third revision January 1951

Fourth revision April 1959

Fifth revision December 1972

Sixth revision September 1984

Seventh revision October 1997

Eighth revision July 1998

Ninth (latest) revision February 2009

**Amendments issued since publication**

Date	Text affected
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### Summary of pages

This document comprises a front cover, an inside front cover, pages i to ii, pages 1 to 16, an inside back cover and a back cover.

## Foreword

### Publishing information

This British Standard is published by BSI and came into effect on 28 February 2009. It was prepared by Technical Committee GEL/10, *Fluids for electrotechnical applications*. A list of organizations represented on this committee can be obtained on request to its secretary.

### Supersession

This British Standard supersedes BS 148:1998, which is withdrawn.

BS 148:1998 was the specification for unused and reclaimed mineral insulating oil for transformers and switchgear. The specification for unused mineral insulating oil is now covered in BS EN 60296. Due to the continued market requirement for reclaimed oil to an agreed specification, BS 148 is now dedicated to reclaimed mineral insulating oil only.

### Information about this document

The limits given in this standard are based on BS EN 60296. For ease of cross-reference, the clause numbering of this standard follows that of BS EN 60296 as far as possible. This standard differs from BS EN 60296 in specifying limits for total furans. The increasing use of total furfural/furans measurements as an in-service diagnostic tool has brought about the necessity to impose limits on reclaimed oil, which is continued in this edition.

This standard acknowledges UK practice whereby the limiting values for reclaimed mineral insulating oils are based on those for unused mineral insulating oils for transformers and switchgear while having due regard for the variability of used oil feedstock from which reclaimed oil will be produced.

### Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its requirements are expressed in sentences in which the principal auxiliary verb is "shall".

*Commentary, explanation and general informative material is presented in smaller italic type, and does not constitute a normative element.*

### Contractual and legal considerations

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

**Compliance with a British Standard cannot confer immunity from legal obligations.**

## 1 Scope

This British Standard specifies requirements for reclaimed oxidation inhibited and uninhibited mineral insulating oils, as delivered, for use in transformers, switchgear and similar electrical equipment in which oil is required as an insulant or for heat transfer.

Oils with and without additives are within the scope of this standard. This standard does not differentiate between the methods used to reclaim used mineral insulating oil; thus it is also applicable to oils having undergone a re-refining process.

This standard does not apply to mineral insulating oils used as impregnates in cables or capacitors, or to hydrocarbon insulating liquids obtained by synthesis.

*NOTE 1 Oils conforming to the requirements of this standard, and containing no additives, are considered to be compatible with one another and can be mixed in any proportion. This does not necessarily apply to oils containing additives, and where the user wishes to mix such oils a check should be made to ensure that the mixture conforms to the requirements of this standard.*

*NOTE 2 Reclaimed mineral insulating oil is produced by the processing, to a greater or lesser extent, of service degraded used mineral insulating oil. Such oil will have been originally supplied in compliance with either BS 148:1998 (or earlier editions) or BS EN 60296. Due to the variability of service conditions and the changes in original specification since the first edition of BS 148, reclaimers may reserve the right to reject used oil if it is considered unsuitable as a feedstock to produce a reclaimed oil in accordance with this specification.*

## 2 Normative references

The following referenced documents are indispensable for the application 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.

BS 2000-15, *Methods of test for petroleum and its products – Part 15: Petroleum products – Determination of pour point*

BS 5263, *Method for sampling liquid dielectrics*

BS 5984, *Method for detection and determination of specified anti-oxidant additives in insulating oils*

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

BS EN 60247, *Insulating liquids – Measurement of relative permittivity, dielectric dissipation factor ( $\tan \delta$ ) and d.c. resistivity*

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

BS EN 61125, *Unused hydrocarbon-based insulating liquids – Test methods for evaluating the oxidation stability*

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

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

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

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

BS EN 62535, *Method for detection of potentially corrosive sulfur in used and unused mineral insulating oils<sup>1)</sup>*

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

BS EN ISO 3104, *Methods of test for petroleum and its products – Part 71: Section 1: Petroleum products – Transparent and opaque liquids – Determination of kinematic viscosity and calculation of dynamic viscosity*

BS EN ISO 3675, *Methods of test for petroleum and its products – BS 2000-160: Crude petroleum and liquid petroleum products – Laboratory determination of density – Hydrometer method*

IEC 60475, *Method for sampling liquid dielectrics*

### 3 Terms and definitions

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

#### 3.1 additive

substance deliberately added to an insulating liquid in small proportion in order to improve certain characteristics

#### 3.2 anti-oxidant

additive incorporated in an insulating liquid to reduce or delay its degradation by oxidation

#### 3.3 inhibited oil

mineral insulating oil which contains an anti-oxidant

#### 3.4 passivator

additive incorporated in an insulating liquid to reduce the chemical reactivity of metallic surfaces within oil-filled equipment

#### 3.5 passivated oil

mineral insulating oil which contains a passivator

#### 3.6 pour point depressant

additive that enables the pour point of a mineral insulating oil to be lowered

#### 3.7 reclaimed oil

mineral insulating oil used in electrical equipment which has been subjected to chemical and/or physical processing to eliminate soluble and insoluble contaminants

#### 3.8 reconditioned oil

mineral insulating oil used in electrical equipment which has been subjected to physical processing to remove contaminants

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<sup>1)</sup> In preparation.

**3.9 re-refined oil**

mineral insulating oil used in electrical equipment which has been subjected to extensive chemical and/or physical processing, similar to that used for the initial production of such oils, to eliminate soluble and insoluble contaminants

**3.10 uninhibited oil**

mineral insulating oil containing no added anti-oxidant but which may contain other additives

**3.11 unused oil**

mineral insulating oil which has not been used in, or been in contact with, electrical equipment or other equipment not required for manufacture, storage or transport

## 4 Identification and delivery

**4.1** Road or rail tank wagons and drums used for the delivery of oil shall be specially cleaned for this purpose.

**4.2** Oil drums and containers of oil samples shipped by the oil supplier shall carry at least the following markings:

- a) supplier's designation;
- b) number and date of this British Standard, i.e. BS 148:2009 <sup>2)</sup>.

**4.3** Each oil delivery shall be covered by a document from the supplier stating at least the following:

- a) supplier's designation;
- b) function and generic nature of any additive present;
- c) that the oil conforms to BS 148:2009.

**4.4** Health and safety information, in line with current legislation, shall be available to the purchaser.

## 5 Sampling

Sampling shall be carried out in accordance with BS 5263.

*NOTE As a general rule, oil samples should be taken where the oil is presumed to be more contaminated, e.g. at the lowest point of the container; however, contaminants at the surface or in the body of the oil should not be overlooked.*

## 6 Properties of the oils

Uninhibited and inhibited oils shall be in accordance with the limiting values specified in Table 1 and Table 2, respectively, when tested in accordance with Clause 7.

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<sup>2)</sup> Marking BS 148:2009 on or in relation to a product represents a manufacturer's declaration of conformity, i.e. a claim by or on behalf of the manufacturer that the product meets the requirements of the standard. The accuracy of the claim is solely the claimant's responsibility. Such a declaration is not to be confused with third party certification of conformity, which might also be desirable.

The limits for water content, breakdown voltage and dielectric dissipation factor for oil packed in drums shall apply only to oils delivered in drums of 200 L nominal capacity and to tests carried out within two weeks following delivery.

*NOTE 1 There is a tendency for water absorption to occur and for the electrical characteristics to deteriorate in oil stored in drums.*

*NOTE 2 Oil in drums cannot be expected to retain the breakdown voltage shown at the time of filling and attention is drawn to the risk of deterioration of electrical properties, which is greater in smaller drums (less than 200 L). It is recommended that the breakdown voltage is checked on oil that has been drummed for more than three months and/or if the drum has been damaged.*

Reclaimed uninhibited oil shall meet the limiting values in Table 1 and reclaimed inhibited oil shall meet the limiting values in Table 2.

*NOTE 3 Precision data given in the specified methods are to be used solely as a guide to the expected agreement between duplicate measurements of the property and are not to be regarded as tolerances applicable to the limits specified in Table 1 and Table 2.*

## 7 Test methods

### 7.1 Appearance

To test appearance, a representative sample of the oil shall be examined in transmitted light under an oil depth of approximately 100 mm and at ambient temperature.

### 7.2 Density

Density shall be measured using any recognized standard test method or, in the event of a dispute, the method specified in BS EN ISO 3675.

Density shall be measured at 20 °C.

*NOTE Density values can be converted to 20 °C using Table 59 D of Petroleum measurement paper No.3: Computer implementation procedures for correcting densities and volumes to 20 °C [1].*

### 7.3 Kinematic viscosity

Kinematic viscosity shall be measured by the method described in BS EN ISO 3104.

### 7.4 Flash point

Flash point shall be determined by the method described in BS EN ISO 2719.

### 7.5 Pour point

Pour point shall be determined by the method described in BS 2000-15.

### 7.6 Neutralization value

Neutralization number of the oil shall be determined in accordance with BS EN 62021-1 or BS EN 62021-2.

**7.7 Corrosive sulfur**

Corrosive sulfur shall be determined by the method described in BS EN 62535.

**7.8 Passivator content**

Passivator content shall be determined by the method described in Annex A.

**7.9 Water content**

Water content shall be determined by the method described in BS EN 60814.

**7.10 Anti-oxidant additives**

Specified anti-oxidant additives shall be detected and determined quantitatively by the methods described in BS 5984.

**7.11 Oxidation stability**

Oxidation stability shall be determined by Method C of BS EN 61125.

**7.12 Breakdown voltage**

Breakdown voltage shall be determined by the method described in BS EN 60156.

**7.13 Dielectric dissipation factor**

Dielectric dissipation factor shall be measured at 90 °C by the method described in BS EN 60247.

**7.14 Polychlorinated biphenyls (PCBs)**

PCBs shall be determined by BS EN 61619.

**7.15 Total furans**

The 2-furaldehyde and furan derivatives shall be determined by the methods described in BS EN 61198.

Table 1 Uninhibited mineral insulating oils

Property	Test method	Limiting values for measured characteristics
Appearance	7.1	Clear, free from sediment and suspended matter
Density, kg/dm <sup>3</sup> at 20 °C	7.2	≤ 0.895 <sup>A)</sup>
Kinematic viscosity	7.3	
mm <sup>2</sup> /s 40 °C <sup>B)</sup>		≤ 13
–15 °C		≤ 800
Flash point, °C	7.4	≥ 135
Pour point, °C	7.5	≤ –30
Neutralization value, mg KOH per g of oil	7.6	≤ 0.03
Corrosive sulfur	7.7	Non-corrosive
Passivator content	7.8	Not detectable <sup>C)</sup>
Water content, mg/kg	7.9	
bulk delivery		≤ 20
drum delivery		≤ 30
Anti-oxidant additives	7.10	Not detectable <sup>D)</sup>
Oxidation stability, 164 h	7.11	
total acidity, mg KOH per g of oil		≤ 1.2
sludge, % by mass		≤ 0.8
Breakdown voltage, as delivered, kV	7.12	≥ 30
Dielectric dissipation factor, at 90 °C and 40 Hz to 62 Hz	7.13	≤ 0.005
Total PCB content mg/kg	7.14	To be agreed <sup>E)</sup>
Total furans mg/kg	7.15	< 0.2

<sup>A)</sup> The corresponding viscosity value at 20 °C is approximately 40 mm<sup>2</sup>/s.

<sup>B)</sup> A maximum limit for density is specified in an attempt to minimize the risk of ice crystals floating in the oil where oil-insulated equipment is exposed to very low temperatures.

<sup>C)</sup> Subject to agreement between purchaser and supplier, a metal passivator may be incorporated in the oil to a maximum concentration of 100 mg/kg as measured by the method given Annex A.

<sup>D)</sup> No anti-oxidant shall be included in uninhibited oils.

<sup>E)</sup> To be agreed between supplier and purchaser but not more than 10 mg/kg.

**NOTE 1** The purchaser might wish to agree with the supplier limits for sulfur content and resistivity at 90 °C.

**NOTE 2** A pour point depressant should only be included in the oil formulation if this is agreed between supplier and purchaser.

**NOTE 3** In the present context, "bulk" refers to shipments made in breather-protected vehicles or containers and does not apply to oil carried in ships or barges or by pipeline.

Table 2 Inhibited mineral insulating oils

Property	Test method	Limiting values for measured characteristics
Appearance	7.1	Clear, free from sediment and suspended matter
Density, kg/dm <sup>3</sup> at 20 °C	7.2	≤ 0.895 <sup>A)</sup>
Kinematic viscosity	7.3	
mm <sup>2</sup> /s 40 °C <sup>B)</sup>		≤ 13
–15 °C		≤ 800
Flash point, °C	7.4	≥ 135
Pour point, °C	7.5	≤ –30
Neutralization value, mg KOH per g of oil	7.6	≤ 0.03
Corrosive sulfur	7.7	Non-corrosive
Passivator content	7.8	Not detectable <sup>C)</sup>
Water content, mg/kg	7.9	
bulk delivery		≤ 20
drum delivery		≤ 30
Anti-oxidant additives <sup>D)</sup> , %	7.10	> 0.08 and < 0.40
Oxidation stability, 500 h	7.11	
total acidity, mg KOH per g of oil		≤ 1.2
sludge, % by mass		≤ 0.8
Breakdown voltage, as delivered, kV	7.12	≥ 30
Dielectric dissipation factor, at 90 °C and 40 Hz to 62 Hz	7.13	≤ 0.005
Total PCB content mg/kg	7.14	To be agreed <sup>E)</sup>
Total furans mg/kg	7.15	< 0.2

<sup>A)</sup> The corresponding viscosity value at 20 °C is approximately 40 mm<sup>2</sup>/s.

<sup>B)</sup> A maximum limit for density is specified in an attempt to minimize the risk of ice crystals floating in the oil where oil-insulated equipment is exposed to very low temperatures.

<sup>C)</sup> Subject to agreement between purchaser and supplier, a metal passivator may be incorporated in the oil to a maximum concentration of 100 mg/kg as measured by the method given in Annex A.

<sup>D)</sup> Methods for the detection and quantitative determination of some anti-oxidant additives are described in BS 5984.

<sup>E)</sup> To be agreed between supplier and purchaser but not more than 10 mg/kg.

**NOTE 1** The purchaser might wish to agree with the supplier limits for sulfur content and resistivity at 90 °C.

**NOTE 2** A pour point depressant should only be included in the oil formulation if this is agreed between supplier and purchaser.

**NOTE 3** In the present context, "bulk" refers to shipments made in breather-protected vehicles or containers and does not apply to oil carried in ships or barges or by pipeline.

Annex A (normative) **Analysis method for determination of passivators in mineral oils by high performance liquid chromatography (HPLC)**

### A.1 General

This test method covers the determination of passivators N-bis(2-ethylhexyl)-aminomethyl-tolutriazol (TTAA), Benzotriazole (BTA) and 5-methyl-1H-benzotriazole (TTA) in mineral oils by high performance liquid chromatography (HPLC) in used and unused insulating mineral oils.

*NOTE 1 This method is based on an existing method for determination of BTA, which can be detected in the same chromatographic run of TTAA, as described in this Annex.*

*NOTE 2 This test method uses the commercial product of TTAA for calibration. Its inherent uncertainty is related to the degree of purity supplied.*

*NOTE 3 TTAA is commercially available as Ciba® IRGAMET® 39. To the best of the committee's knowledge, this is the only commercial product available at time of publication. This information is given for the convenience of users of this document and does not constitute an endorsement by BSI or the committee of this product.*

This test method covers the determination of TTAA for routine analysis.

TTAA is an amine derivate of tolutriazole, liquid at room temperature, added in mineral insulating oils mainly as a metal passivator, for its capability to inhibit the corrosive reactions involving surfaces of copper (and other metals) and of metal-reactive compounds present in the oil. TTAA is usually added to mineral oils in concentrations of 0.005% to 0.02%.

Other triazole derivates are used in insulating mineral oils, such as BTA and TTA, which have a lower solubility in oil. BTA is more widely used than TTA, mainly to modify the electrical behaviour of copper surfaces.

TTAA is a mixture of 2 isomers:

N,N-bis(2-ethylsil)-4-methyl-1H-benzotriazol-1-methylamine and N,N-bis(2-ethylsil)-5-methyl-1H-benzotriazol-1-methylamine. The two isomers are not usually separated in the conditions described in this method, but they might give two partially overlapping peaks if a high efficiency column is used (C18, 250 mm); in this case the total area of two peaks shall be considered.

Heavily oxidized oils might partially affect the analysis, giving relevant interferences from UV-absorbing polar compounds. In the case of doubt, the standard addition method can be used for more accurate determinations.

This method can be used for monitoring TTAA content in passivated used and unused insulating mineral oils.

## A.2 Principles

### A.2.1 Summary

A weighed portion of the sample oil is diluted with pentane and passed under a vacuum through a silica gel solid phase extraction (SPE) cartridge, previously rinsed with methanol and pentane. The residue of non-polar oil constituents retained by the solid phase is eluted with a further volume of pentane and discarded. The cartridge is then dried by flushing it with air under a vacuum.

The analytes are eluted with a known volume of methanol and filtered through a 0.45 µm PTFE filter.

The solution is injected into an HPLC system equipped with a reverse-phase column, and the TTAA is detected with a UV detector at a wavelength of 260 nm to 270 nm.

*NOTE In order to obtain the optimal separation and detection condition with individual chromatographic systems, this method allows a large flexibility in choice of stationary phase and mobile phase separation.*

### A.2.2 Interferences

#### A.2.2.1 Co-eluting compounds

TTA has been found to co-elute with TTAA in the conditions described in this method. TTAA decomposes to TTA during some stage of the chromatographic run, the UV spectra of the two compounds (recorded from the chromatogram) being identical.

*NOTE It is recommended that the effective co-elution of TTAA and TTA under the selected separation conditions is verified.*

#### A.2.2.2 UV-adsorbing interfering compounds

Heavily oxidized oils might contain UV-adsorbing compounds showing retention times close to TTAA. For the same reason, background noise might be encountered. In these cases, when the integration of the peak is difficult, or an overlapping peak appears, use the standard addition method for quantification.

## A.3 Equipment

### A.3.1 Apparatus

**A.3.1.1** Balance, top loading, with automatic tare, capable of weighing to 0.001 g, capacity of 100 g minimum.

**A.3.1.2** Vacuum manifold for SPE, for vacuum elution of silica cartridges.

**A.3.1.3** Silica SPE cartridges sorbent substrate silica; sorbent weight: 500 to 1 000 mg; pH range: 2 to 8; particle size: 20 µm to 200 µm.

*NOTE The choice of the sorbent weight should be carefully correlated with the weight of the sample analysed and to the load capacity of the cartridge. While optimizing the method, a check for analyte recovery is recommended.*

**A.3.1.4** PTFE filters, 0.45 µm, Luer plug fitting.

**A.3.1.5 HPLC system**, equipped with:

- a pumping device suitable for at least two solvents;
- an injection device suitable for injection of 10  $\mu\text{L}$  to 100  $\mu\text{L}$  (automatic injection is preferable);
- RP column, C8 or C18, end-capped, suitable for mobile phase with pH 2 to 8;

*NOTE* The choice of the length of the column and particle diameter is the responsibility of the laboratory applying this method. Good analytical results have been obtained with 150 mm to 250 mm columns, particles  $\varnothing$  3.5  $\mu\text{m}$  to 5  $\mu\text{m}$ , column diameter 4.6 mm.

- RP pre-column, with the same stationary phase;
- UV detector (a diode array detector is preferable, to record UV spectra);
- data acquisition device.

**A.3.1.6 Glass volumetric flasks**, 5 mL, one per sample or control to be tested.

**A.3.2 Reagents and materials****A.3.2.1 Purity of reagents**

Reagent grade chemicals shall be used in all tests.

All solvents used for chromatographic elution shall be HPLC grade.

**A.3.2.2 Required reagents**

- Methanol for HPLC;
- HPLC grade water;
- Pentane;
- Toluene.

**A.3.2.3 Standard materials**

- TTAA of analytical grade (not obtained by dilution of TTAA in mineral oil or other suitable solvents, even if the TTAA content is known);
- TTA of analytical grade;
- Blank oil:  
A mineral insulating oil, free from TTAA. For the reasons reported in **A.2.2.1**, the blank oil for dilution shall be also TTA and BTA free;
- Control sample (see **A.5.2.1**, Note 2).

**A.3.2.4 Solutions****A.3.2.4.1 Stock solution**

A concentrated solution of TTAA in toluene.

*NOTE 1* It is recommended that a fresh stock solution is prepared every 3 months, and stored in dark bottles at room temperature.

*NOTE 2* Stock solutions of 1 000 mg/kg have been found to be stable for at least 3 months. If a longer lasting stock is desired, the stability should be checked by comparison with a fresh solution.

#### **A.3.2.4.2 Standard solutions**

From the stock solution (A.3.2.4.1), prepare at least 5 diluted solutions for calibration (A.5.2.1).

Prepare the solutions freshly for each calibration stage, by diluting the stock solution with blank oil.

The standard solution shall cover the range of 5 mg/kg to 500 mg/kg.

### **A.4 Sampling**

Take laboratory samples in accordance with IEC 60475.

### **A.5 Analytical procedure**

#### **A.5.1 Preparation and operation of apparatus**

Consult the manufacturer's instructions for operating the instrument according to the selected separation and detection conditions.

*NOTE* Design differences between instruments, columns and detectors make it impractical to detail the operating conditions.

##### **A.5.1.1 Separation conditions**

Both C8 and C18 end-capped RP columns are suitable for separation of TTAA. Good separation can be carried out either with isocratic or gradient elutions, with mobile phase water and methanol. The solvent ratio shall be 50% water and 50% methanol (with C8 columns) or 20% water and 80% methanol (with C18 columns).

A flow rate of 0.5 mL/min to 1 mL/min is suitable.

*NOTE 1* Table A.1 reports some experimental conditions as a guide, but each laboratory should optimize its own separation parameters.

A good separation is obtained if a sharp, shoulderless peak is obtained, without overlapping with a BTA peak.

*NOTE 2* See A.2.2 for information on interferences.

*NOTE 3* In some cases, to have a better separation and to avoid peak tailing, it is preferable to use a buffer instead of pure water in the mobile phase. Acetic buffers have been found to be satisfactory at pH 3 (concentration between 50 and 80 mM), increasing the quality of the separation. In the case of use of buffers, check for absorbance spectrum of TTAA, since it might vary with pH.

##### **A.5.1.2 UV detection**

Detect the TTAA with a UV detector.

*NOTE* It is recommended that a wavelength of 264 nm is used for UV detection of TTAA, corresponding to the maximum absorbance (see Figure A.1). However, a lower wavelength may be used if necessary.

Table A.1 Examples of separation condition

Column mm	Separation conditions mL/min	Timetable			TTAA Retention Time min	BTA Retention Time min	Notes
		Time (min:sec)	H <sub>2</sub> O %	Methanol %			
C18, 250	Isocratic, 1	0:00	30	70	3.5 to 4.5	2.5 to 3	
		20:00	30	70			
C18, 250	Gradient, 1	0:00	30	70	3.5 to 4.5	2.5 to 3	The step with 100% methanol provides column clean up
		4:00	30	70			
		6:00	0	100			
		10:00	0	100			
		14:00	30	70			
C18, 150	Gradient, 0.5	0:00	50	50	8 to 9	5 to 6	
		15:00	50	50			
		20:00	0	100			
		45:00	0	100			
		50:00	0	100			
C18, 150	Gradient, 1	0:00	20	80	6 to 7	5 to 6	
		7:30	0	100			
		14:00	50	50			
		18:00	20	80			
C8, 150	Isocratic, 0.5	0:00	50	50	3.5 to 4.5	2.5 to 3	
		20:00	50	50			

## A.5.2 Calibration

Establish the linear range for the particular instrument being used and the selected separation procedure.

*NOTE* The method should show a linear response in a concentration range of 5 mg/kg to 500 mg/kg.

### A.5.2.1 Calibration procedure

Prepare at least 5 standard solutions by diluting the stock solution (A.3.2.4.1) with blank mineral oil.

Extract the blank oil (A.3.2.3) and each standard solution following the procedure in A.6.1. Run in triplicate at least the two external points (the minimum and the maximum).

Plot the peak area against concentration and calculate the regression model ( $y = bx + m$ ) as a calibration curve. A correlation factor higher than 0.99 shall be considered acceptable. The intercept  $m$  shall be very close to the origin; and  $(m/b) < 1$ .

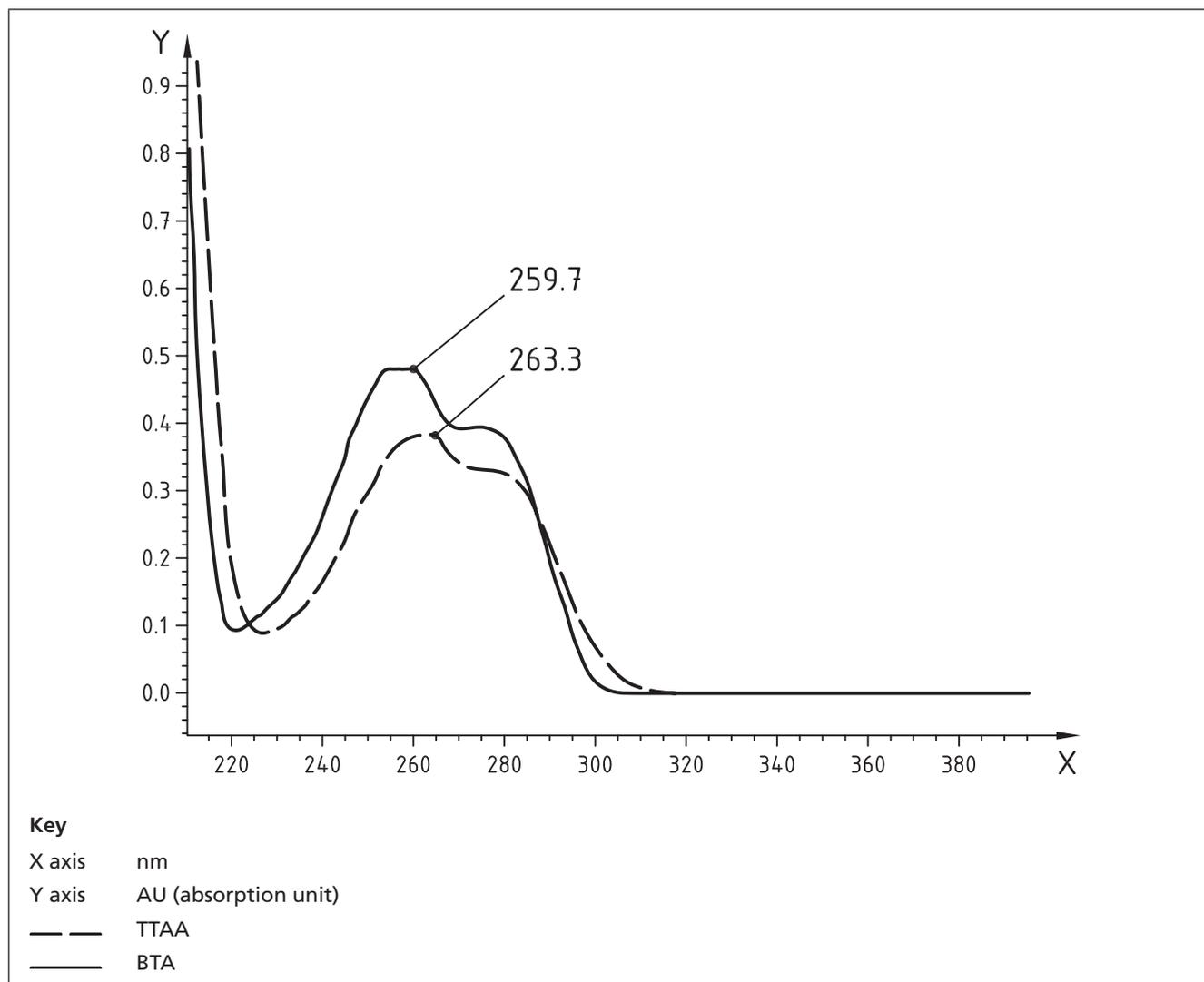
If the intercept is not very close to the origin, there are impurities. Repeat the calibration procedure.

The standard solution shall be prepared fresh each calibration.

*NOTE 1* Recalibration every 6 months is recommended.

*NOTE 2* A control sample (A.3.2.3) of known concentration should be tested periodically to verify the method's stability.

Figure A.1 UV spectra of TTAA and BTA



## A.6 Analysis

### A.6.1 Sample pre-treatment by SPE

Using a vacuum manifold, slowly rinse an SPE silica cartridge (A.3.1.3) with approximately 5 mL of methanol, then condition the cartridge by passing through it approximately 10 mL of pentane.

Weigh a sample portion of 0.5 g to 2 g, to the nearest 0.01 g.

The weight of the sample shall be optimized in connection with the sorbent material mass in the cartridge; see A.3.1.3, Note.

*NOTE 1 An excessive weight of sample can overload the sorbent and affect the linearity of the method, underestimating the highest concentrations.*

Dilute the sample portion with 10 mL pentane and pass the solution through the pre-conditioned cartridge at a maximum rate of 3 mL/min. Discard the eluate.

Rinse the cartridge with 20 mL fresh pentane at a maximum rate of 3 mL/min, to remove the non-polar oil constituents adsorbed by the silica. Discard the eluate.

Dry the sorbent material by flushing it under a vacuum for 5 to 10 minutes.

Slowly elute the cartridge (in the same vacuum manifold or manually, with a syringe) with methanol, collecting the first 5 mL into a volumetric flask (A.3.1.6).

*NOTE 2* The elution can be done with a different solvent, e.g. with the chromatographic mobile phase. Check for the complete solubility of TTAA if an alternative solvent is used.

*NOTE 3* A different volume of solvent can be used to satisfy the requirements of analytic recovery (A.8).

### A.6.2 HPLC analysis

With an injection device (A.3.1.5) inject into the HPLC a portion of the last eluate collected into the 5 mL volumetric flask (A.3.1.6).

*NOTE* The injection volume depends on the sensitivity of the instrument and on the weight of oil analysed: usually 10 µL to 100 µL loops are suitable.

Run the chromatogram and record the area of the peak corresponding to TTAA retention time.

### A.6.3 Calculations

The model obtained during the calibration being  $y = bx + m$ , calculate the result as:

$$\text{mg/kg (TTAA)} = \frac{\text{peak area} - m}{b}$$

### A.7 Report

Report the concentration of TTAA in mg/kg to the nearest whole figure.

### A.8 Analytic recovery yield

#### A.8.1 Adsorption yield

Verify the adsorption yield of the silica SPE cartridges as following.

- Put 2 cartridges in series in the vacuum manifold.
- Pass a standard sample (200 mg/kg) through both cartridges as described in A.6.1, then separate the two cartridges and elute them separately with 5 mL of methanol in each one.
- Analyse the two samples, and record the results as  $X_1$  (concentration found in the upper cartridge) and  $X_2$  (concentration found in the lower cartridge).
- Check that:

$$\frac{X_1}{X_1 + X_2} \geq 0.98$$

#### A.8.2 Elution yield

Verify the elution yield from the silica SPE cartridges as following.

- Pass a standard sample (200 mg/kg) through a cartridge as described in A.6.1.
- Elute the cartridge twice with 5 mL of methanol each time.

- Analyse the two samples separately, and record the results as  $X_1$  (concentration found in the first elution) and  $X_2$  (concentration found in the second elution).
- Check that:

$$\frac{X_1}{X_1 + X_2} \geq 0.98$$

## A.9 Precision data

### A.9.1 Detection limit

In the condition prescribed in this method, a detection limit of < 5 mg/kg is expected.

### A.9.2 Repeatability

Duplicate determinations carried out by one laboratory shall be considered suspect at the 95% confidence level if they differ by more than the value reported in Table A.2 (expressed in percentage of the average value).

Table A.2 Repeatability

Concentration mg/kg	r (repeatability) %
10	10
> 50	5

### A.9.3 Reproducibility

Duplicate determinations carried out by different laboratories shall be considered suspect at the 95% confidence level if they differ by more than the value reported in Table A.3 (expressed in percentage of the average value).

Table A.3 Reproducibility

Concentration mg/kg	R (reproducibility) %
10	15
> 50	8

## Bibliography

### Standards publications

For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

BS EN 60296, *Fluids for electrotechnical applications – Unused mineral insulating oils for transformers and switchgear*

### Other publications

- [1] THE INSTITUTE OF PETROLEUM. Petroleum measurement paper No. 3: Computer implementation procedures for correcting densities and volumes to 20 °C. October 1988. <sup>3)</sup>

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<sup>3)</sup> Available from Energy Institute, 61 New Cavendish St, London W1G 7AR.



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389 Chiswick High Road London W4 4AL UK

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