SLOVENSKI STANDARD
SIST HD 415 S1:1999

01-januar-1999

Detection and determination of specified anti-oxidant additives in insulating oils (IEC 60666:1979)

Detection and determination of specified anti-oxidant additives in insulating oils

Nachweis und Bestimmung spezieller Antioxidantien in Isolierölen

Détection et dosage d'additifs antioxydants spécifiques présents dans les huiles isolantes

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Detection and determination of specified anti-oxidant additives in insulating oils

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

DETECTION AND DETERMINATION OF SPECIFIED ANTI-OXIDANT ADDITIVES IN INSULATING OILS

FOREWORD

1) The formal decisions or agreements of the IEC on technical matters, prepared by Technical Committees on which all the National Committees having a special interest therein are represented, express, as nearly as possible, an international consensus of opinion on the subjects dealt with.

2) They have the form of recommendations for international use and they are accepted by the National Committees in that sense.

3) In order to promote international unification, the IEC expresses the wish that all National Committees should adopt the text of the IEC recommendation for their national rules in so far as national conditions will permit. Any divergence between the IEC recommendation and the corresponding national rules should, as far as possible, be clearly indicated in the latter.

PREFACE

This standard has been prepared by Sub-Committee 10A: Hydrocarbon Insulating Oils, of IEC Technical Committee No. 10: Liquid and Gaseous Dielectrics.

Drafts were discussed at the meetings held in Baden-Baden in 1975 and in Moscow in 1977. As a result of this latter meeting, a draft, Document 10A(Central Office)39, was submitted to the National Committees for approval under the Six Months' Rule in August 1978.

The National Committees of the following countries voted explicitly in favour of publication:

Australia	Netherlands
Austria	Poland
Belgium	Romania
Bulgaria	South Africa (Republic of)
Czechoslovakia	Spain
Denmark	Sweden
Egypt	Switzerland
France	Turkey
Germany	Union of Soviet Socialist Republics
Hungary	United Kingdom
Israel	United States of America
Italy	

Other IEC publications quoted in this standard:

Publications Nos. 474: Test Method for Oxidation Stability of Inhibited Mineral Insulating Oils.
590: Determination of the Aromatic Hydrocarbon Content of New Mineral Insulating Oils.

DETECTION AND DETERMINATION OF SPECIFIED ANTI-OXIDANT ADDITIVES IN INSULATING OILS

1. Scope

The methods described are to be used for the detection and determination of specified anti-oxidant additives in new hydrocarbon insulating oils. The detection methods are to be applied to assess whether or not a hydrocarbon insulating oil contains an anti-oxidant additive as specified by the supplier.

The determination methods are used for the quantitative determination of anti-oxidant additives previously detected by the appropriate detection method.

Note. – In certain cases, the methods described may also be used for oils in service.

SECTION I – METHODS FOR THE DETECTION OF ANTI-OXIDANT ADDITIVES

2. Detection of 2.6-di-tert-butyl-paracresol (DBPC) by thin layer chromatography

2.1 *Summary of the method*

The anti-oxidant additive is extracted from the oil with a suitable solvent. The solvent from the extract is evaporated and the residue analyzed by thin layer chromatography with the aid of a specific reagent.

2.2 *Reagents*

- Methanol, analytical grade.
- *n*-heptane, analytical grade.
- Phosphomolybdic acid: solution of 5 g phosphomolybdic acid in 100 ml isopropanol.
- Ammonia solution, analytical grade (density at 20 °C: 0.91 g/cm³).
- Di-isopropyl ether, analytical grade.

2.3 *Equipment*

- Usual thin layer chromatography (TLC) equipment.
- Silica-gel coated plates.
- Microsyringe.

2.4 *Procedure*

- Extract 50 ml of the insulating oil three times with 20 ml portions of methanol.
- Combine the extracts and evaporate the methanol to a final volume of about 5 ml taking care to avoid overheating. It is best to carry out the evaporation under a nitrogen stream.

- Apply 20 µl of the concentrated extract on a TLC plate and develop the plate in a lined tank with a mixture of 99.5% *n*-heptane plus 0.5% di-isopropyl ether until the solvent front has travelled 10 cm up.
- Remove the plate from the tank and dry.
- After air-drying, spray the plate with the phosphomolybdic reagent.
- Dry at ambient temperature (the colour will appear more quickly if the plate is heated to approximately 90 °C for a few minutes) and expose the chromatographic plate to ammonia vapours.
- The DBPC appears as a blue spot on a white background.
- Limit of detection: about 0.005% by mass on the oil as such. To aid in identifying the developed colour, repeat the above using a standard solution, 0.2% by mass of DBPC in an insulating base oil.

3. Detection of *n*-phenyl-α-naphtylamine (PAN) and *n*-phenyl-β-naphtylamine (PBN) by thin layer chromatography

3.1 *Summary of the method*

The oil sample is dissolved in a suitable solvent and analyzed by thin layer chromatography with the aid of a suitable chromogenic agent.

3.2 *Reagents*

- Dichloromethane, analytical grade.
- Mixture of 97% of iso-octane (2.2.4 trimethyl pentane) and 3% of ethyl acetate, analytical grade.
- Diazotized *p*-nitroaniline. The reagent is prepared immediately before use by adding solution No. 1 to an equal volume of cold solution No. 2.
  Solution No. 1: dissolve 1.44 g sodium nitrite in 250 ml water (stock solution).
  Solution No. 2: dissolve 2.8 g *p*-nitroaniline in 32 ml warm concentrated hydrochloric acid and make up with water to 250 ml (stock solution). Store in brown glass bottle.
- 1% dichlorobenzoquinone-4-chlorimine (DCLQ) ethanolic solution.

3.3 *Equipment*

- Usual TLC equipment.
- Silica-gel coated plates.
- Hair dryer.
- Microsyringe.

3.4 *Procedure*

- Dilute 5 ml of insulating oil with 5 ml of dichloromethane and apply 20 µl of this solution to the TLC plate.
- The plate is developed in a lined tank with the mixture of iso-octane and ethyl acetate (97/3 vol.). When the solvent front has travelled 10 cm up, the plate is removed and air dried.