

CONFIRMED
MAY 1991

Method for

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

[IEC title: Detection and determination of specified anti-oxidant
additives in insulating oils]

UDC 621.315.615.2:665.767.035.8:665.767.038.5:66.094.38:543.83.061/062

NO COPYING WITHOUT BSI PERMISSION EXCEPT AS PERMITTED BY COPYRIGHT LAW

BSI
Documents

AVA

Document Sharing Hub

Cooperating organizations

The General Electrotechnical Engineering Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

- Associated Offices Technical Committee
- British Approvals Service for Electric Cables Ltd.
- British Electrical and Allied Manufacturers' Association (BEAMA)
- British Radio Equipment Manufacturers' Association
- British Steel Corporation
- Cbmpe
- Department of Energy (Electricity)
- Electric Cable Makers' Confederation*
- Electrical Contractors' Association
- Electrical Contractors' Association of Scotland
- Electricity Supply Industry in England and Wales*
- Electronic Components Industry Federation
- Electronic Engineering Association
- Engineering Equipment Users' Association
- Era Technology Limited*
- Health and Safety Executive
- Home Office
- Institution of Electrical Engineers
- Ministry of Defence
- National Coal Board
- Oil Companies Materials Association
- Post Office

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

- British Railways Board
- British Lubricants Federation Limited
- Electric Cable Makers Confederation — Supertension Cables Group
- Institute of Petroleum
- Transmission and Distribution Association (BEAMA)

This British Standard, having been prepared under the direction of the General Electrotechnical Engineering Standards Committee, was published under the authority of the Executive Board and comes into effect on 29 August 1980

© BSI 12-1999

The following BSI references relate to the work on this standard:
Committee reference GEL/115
Draft for comment 78/29342 DC

ISBN 0 580 11583 6

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

	Page
Cooperating organizations	Inside front cover
National foreword	ii
1 Scope	1
Section I. Methods for the detection of anti-oxidant additives	
2 Detection of 2,6-di-tert-butyl-paracresol (DBPC) by thin layer chromatography	1
2.1 Summary of the method	1
2.2 Reagents	1
2.3 Equipment	1
2.4 Procedure	1
3 Detection of <i>n</i> -phenyl- α -naphtylamine (PAN) and <i>n</i> -phenyl- β -naphtylamine (PBN) by thin layer chromatography	1
3.1 Summary of the method	1
3.2 Reagents	1
3.3 Equipment	1
3.4 Procedure	2
4 Detection of anthranilic acid in new insulating oils by thin layer chromatography	2
4.1 Summary of the method	2
4.2 Reagents	2
4.3 Equipment	2
4.4 Procedure	2
Section II. Methods for the determination of anti-oxidant additives	
5 Determination of 2,6-di-tert-butyl-paracresol	2
5.1 Quantitative determination by infra-red spectrophotometry	2
5.2 Semi-quantitative determination by thin layer chromatography	4
6 Determination of <i>n</i> -phenyl- β -naphtylamine by spectrophotometry	5
6.1 Summary of the method	5
6.2 Reagents	5
6.3 Equipment	5
6.4 Procedure	5
6.5 Calculation	6
6.6 Lower limit of detection	6
Figure 1 — Typical infra-red spectrum to determine DBPC content	7
Publications referred to	Inside back cover

National foreword

This British Standard has been prepared under the direction of the General Electrotechnical Engineering Standards Committee. It is identical with IEC 666:1979 *“Detection and determination of specified anti-oxidant additives in insulating oils”*, published by the International Electrotechnical Commission (IEC).

Terminology and conventions. The text of the International Standard has been approved as suitable for publication, without deviation, as a British Standard. Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

Cross-references

At present there are no corresponding British Standards for IEC 474:1974 and IEC 590:1977.

The technical committee has reviewed the provisions of these IEC Publications, and has decided that they are acceptable for use in conjunction with this standard.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 -naphthylamine (PAN) and *n*-phenyl- β -naphthylamine (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.