

ICS 27.100
CCS F 20

DL

**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

DL/T 1977-2019

**Determination of oxidation stability of mineral insulating oil by
differential scanning calorimetry (DSC)**

矿物绝缘油氧化安定性的测定 差示扫描量热法

(English Translation)

Issued on: June 4, 2019

Implemented on: October 1, 2019

Issued by: National Energy Administration

Table of Contents

Foreword
1 Scope
2 Normative references
3 Terms and definitions
4 Method summary
5 Equipment
6 Reagents and materials
7 Calibration
8 Preparation
9 Test Procedure
10 Precision
11 Report
Appendix A
Appendix B

Foreword

This standard was drafted in accordance with GB/T 1.1-2009.

Please note that certain contents of this document may involve patents. The issuing agency of this document is not responsible for identifying these patents.

The revision of this standard adopts IEC/TR 62036:2007 "Test Method for Oxidation Stability of Mineral Insulating Oil Differential Scanning Calorimetry (DSC)".

This standard is redrafted according to IEC/TR 62036:2007. For the convenience of comparison, this standard and IEC/TR 62036:2007 are given in Appendix A A list of structural differences. A list of technical differences between this standard and IEC/TR 62036:2007 is given in Appendix B.

This standard was proposed by the China Electricity Council.

This standard is under the jurisdiction of the National Electrochemical Standardization Technical Committee (TC322).

Drafting organization of this standard. State Grid Hunan Electric Power Research Institute;

Participants in this standard. State Grid Zhejiang Electric Power Co., Ltd. Electric Power

Research Institute, PetroChina Lanzhou Lubricant Research and Development Center,
State Grid Fujian Electric Power Co., Ltd. Electric Power Research Institute.

1 Scope

This standard specifies the use of Differential Scanning Calorimetry (DSC) to characterize the mineral insulation through oxygen pressurization and programmed heating to determine the oxidation induction period.

The method of oil oxidation stability.

This standard is applicable to the determination of oxidation stability of unused and used mineral insulating oils.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this document.

For undated references, the latest version (including all amendments) applies to this document.

GB/T 7597 Power oil (transformer oil, steam turbine oil) sampling method SH/T 0790-2007 Lubricating grease oxidation induction period measurement method (pressure differential scanning calorimetry)

3 Terms and definitions

The following terms and definitions apply to this document.

Thermal curve

Graph of sample heat flow versus time.

[SH/T 0790-2007, Terms and Definitions 3.4] 3.2 Extrapolated onset time the time measured on the thermal curve is the time at the intersection of the baseline extrapolation line and the tangent line at the maximum rate of the oxidation exothermic peak.

[SH/T 0790-2007, Terms and Definitions 3.1] 3.3 Oxidation induction time (OIT) the oxidation rate increases from zero to the maximum rate for a period of time, and its value is consistent with the extrapolated inflection point time.

[SH/T 0790-2007, Terms and Definitions 3.2]

4 Method summary

Weigh a certain quality of mineral insulating oil sample, put it into the differential scanning calorimeter test cell, pass the program under the specified oxygen pressure the

temperature rise control causes the sample to undergo an oxidative exothermic reaction, and the extrapolated inflection point time of the oxidation exothermic peak on the thermal curve is measured and recorded as the oxidation induction period.

Characterize the oxidation stability of mineral insulating oil.

5 Equipment

5.1

Differential scanning calorimeter. It can maintain the pressure above 2.0 MPa, control the heating rate from 0 °C/min to 20 °C/min and keep it constant Temperature, and can automatically record the heat flow difference between the sample and the blank according to the required sensitivity and deviation.

5.2

Sample pan and reference pan. flat-bottomed aluminum pan for differential scanning calorimeter.

5.3

Sample transfer device. a clean and inert device that can transfer the sample to the sample pan. It is advisable to use a glass rod with a diameter of 3 mm to 4 mm.

Platinum wire with a diameter of 0.25 mm, disposable pipette or disposable syringe needle, etc.

5.4

Oven. can be heated and kept constant at $120\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

5.5

Analytical balance. Sensitivity is 0.1 mg.

5.6

Desiccator. a desiccator that can dry and preserve the sample pan and reference pan.

6 Reagents and materials

6.1

Petroleum ether. analytically pure, boiling range $60\text{ }^{\circ}\text{C} \sim 90\text{ }^{\circ}\text{C}$.

6.2

Acetone. analytically pure.

6.3

Oxygen. the purity is not less than 99.5% (volume fraction).

6.4

Indium. the purity is not less than 99.9% (mass fraction), used as a calibration standard.

7.1 Temperature calibration

7.1.1

Calibrate with indium under normal pressure and air atmosphere. The calibration should be performed once a month. If the equipment is not used frequently, it should calibration.

7.1.2

Weigh about 10 mg of indium from the sample pan into the sample cell, put the reference pan into the reference cell, close the test cell, and turn on the differential display the scanning calorimeter is heated from room temperature to 180 °C at a heating rate of 10 °C/min. After the operation is completed, the measured indium melting point temperature should be 156.6 °C±0.3 °C.

7.1.3

If the melting point temperature deviates from this value, it should be calibrated in accordance with the hardware or software calibration procedures in the manufacturer's instrument manual.

7.2 Temperature controller calibration

7.2.1

The temperature controller calibration should be carried out once a year.

7.2.2

The calibration procedure is to leave the sample pan and reference pan in the test cell, close the test cell, slowly open the oxygen pressure to 2.0 MPa ± 0.1 MPa, Turn on the differential scanning calorimeter to heat up and keep the test cell at the selected test temperature (130 °C, 160 °C, 210 °C). After 10 minutes, If the difference between the displayed temperature and the selected temperature exceeds ±0.5 °C, the temperature controller should be adjusted until it meets the requirements. After adjustment, continue to the next step Wait at least 5 minutes to verify that the temperature is stable.

7.2.2

If the instrument does not have a temperature controller adjustment function, the calibration of the temperature controller should be carried out according to the recommendations of the instrument manufacturer.