

# Thermal – Oxidation Stability of Insulating Fluids

P. Semančík, R. Cimbala, K. Zálíš

*This paper deals with the thermal – oxidation stability of insulating fluids by the ČEZ– ORGREZ test method [1]. It describes the principle test method as a preparation of experiment and the thermal – oxidation stability measuring of insulating oil.*

*Keywords: oxidation stability, oil life, dielectric dissipation factor, ČEZ – ORGREZ test method.*

## 1 Introduction

Insulating oils should have stable high-quality properties, not only in the original state, but also during the up time in operation. The stability of insulating oils has an elementary meaning during operation, because they work under high temperatures usually in the presence of oxygen, so they should be oxidation resistant.

The oxidation of oil increases its acidity and the content of sediments. Low sediment values indicate high oxidation stability, leading to long oil life. Minimizing the creation of sediments, the dielectric dissipation factor, corrosion of metals, electric failures maximizes the insulating ability of oil. Oxidation stability is measured by IEC 61125, method C [2, 3], or by the ČEZ – ORGREZ method [1].

Oxidation stability is an indicator that allows us to set stricter limits for oils in special applications. In some countries, stricter limits or other requirements and tests are imposed.

## 2 ČEZ – ORGREZ methodology

During the test, the sample of new or reclaimed oil is exposed to conditions simulating a load application, similar to the load during operation. Individual factors are simplified. High-quality parameters are periodically monitored until sediments form and the oil is no longer usable.

### 2.1 Laboratory instruments, devices and chemicals [1]

- glass sulphonation flask, 6000 ml
- separation trap 250 ml
- tube immune temperatures and oils (teflon, silicon)
- air compressor
- laboratory drying chamber with temperature regulation (100 °C)
- plastic component syringe for taking samples (150 ml)
- clean copper wire without surface treatment (unvarnished)
- analytical scales, precision 0.001 g
- measuring cylinder, 2000 ml.

### 2.2 Preparation of the experiment, and testing process [1]

Before the test begins, the initial high-quality parameters are determined and 500ml is taken away from the oil sample.

A measuring cylinder is used to measure out 5000 ml of oil, marked out for testing, into the sulphonation flask.

The required quantity of copper wire will be added to the oil – 10g (quantity cca. 0.1 cm<sup>2</sup>/g of oil) to each litre of oil.

The flask with oil will be put into the laboratory drying chamber, at a temperature of 100 °C.

Using tubes perhaps made of glass air will be conducted into the samples to ensure delivery of condensed fluid into the separation trap outside the drying chamber.

A control test and verification of the temperature regulated in the drying chamber will be carried out every day.

Some, of the samples, will be removed at weekly intervals to determine the values of selected parameters (acidity, interfacial tension and content of inhibitors – 1× per week, dielectric dissipation factor – 1× per 3 weeks).

The test will be completed when sediments insoluble in n-heptane are present or when there are no more samples for continuing the test or after 840 hours of testing.

### 2.3 Comparison with the ČSN EN 61125 standard, method C

This method describes a test for interpreting the oxidation stability of new hydrocarbon insulating fluids under accelerated conditions, without reference to whether antioxidant additives are present.

*Test conditions:*

The filtered fluid sample oxidizes under the following conditions [3]:

- oil weight: 25 g ± 0.1 g,
- oxidation gas: air,
- gas flow speed: 0.15 l/h ± 0.015 l/h,
- test period:
  - 164 h – for uninhibited oil,
  - 500 h – for inhibited oil,
- accelerator: copper wires in quantities 28.6 cm<sup>2</sup> ± 0.3 cm<sup>2</sup> measured oil.

Table 1: Values measured by the ČEZ – ORGREZ test method [4]

Test period (h)	tgδ ( $\times 10^{-2}$ )			$\varepsilon_r$ (-)			$\rho$ (GΩ m)			ČK	$\sigma$	$Q_i$	Sediments insoluble in the n-heptan
	20 °C	70 °C	90 °C	20 °C	70 °C	90 °C	20 °C	70 °C	90 °C				
0	0.018	0.075	0.138	2.257	2.199	2.175	2203.8	598.1	232.9	0.004	51	0.35	-
168										0.006	50	0.37	-
336	0.040	0.126	0.385	2.255	2.159	2.136	856.8	178.5	107.1	0.006	49	0.38	-
504										0.007	49	0.37	-
672										0.010	47	0.29	-
840	0.076	0.832	1.473	2.263	2.207	2.182	561.4	147.3	122.8	0.012	47	0.29	-
1008										0.011	45	0.27	-
1176										0.016	44	0.27	-
1344	0.122	0.886	1.866	2.262	2.190	2.165	235.8	108.8	48.3	0.016	43	0.25	-
1512										0.016	42	0.24	-
1680										0.021	41	0.23	-
1848	0.200	2.509	3.761	2.268	2.191	2.164	169.3	52.0	24.7	0.026	41	0.23	-
2016										0.030	39	0.23	-
2184										0.037	38	0.22	-
2352	0.168	3.300	5.978	2.271	2.202	2.181	127.4	15.2	9.5	0.048	37	0.16	-
2520										0.065	37	0.13	-
2688										0.079	35	0.08	-
2856	0.483	7.890	17.648	2.300	2.244	2.227	51.2	2.7	1.0	0.132	28	0.00	presence

ČK – mg KOH/g,

$\sigma$  – mN/m

$Q_i$  – %<sub>hmot.</sub>,

F – sample filtered using white tape filter paper (6 – 6.8  $\mu$ m) before the measurement.

### 3 Experimental results

The thermal – oxidation stability test of insulating oil was made using the ČEZ – ORGREZ method [1]. Power transformer insulating oil was used as a sample. Further information about the sample confidential to the manufacturer and to the plant operator. During the experiment, the data, interpreted in (Table 1) was measured [4].

The principle of the test is based on the air oxidation of the measured oil with added accelerator at a given temperature.

The test was carried out under the following conditions [1]:

- temperature 100 °C,
- volume of oil samples 5 l,
- bubbling of oil dried and refined by air in larger amounts than are needed for reaction of oil with the air,
- accelerator: copper wires in quantities cca 0.1 cm<sup>2</sup>/g measured oil.

The separation trap, placed outside the drying chamber, gathers the condensed fluid released during the test.

The values measured in dependency on the length of test periods are recorded in tables (Table 1), which show the degradation process of the oil until the moment when sediments insoluble in n-heptane form, or until the test is terminated.

The graphic dependencies in Figs. 1–4 were made from the measured values monitoring the individual parameters.

*Monitored parameters [2–5]:*

tgδ – dielectric dissipation factor,

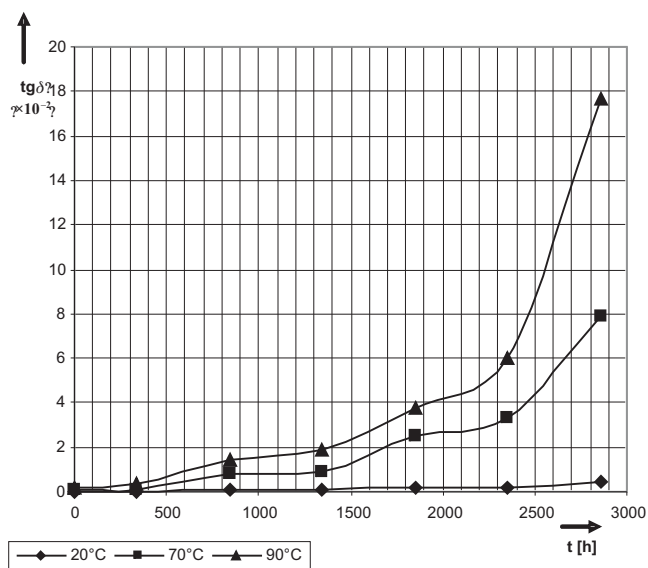
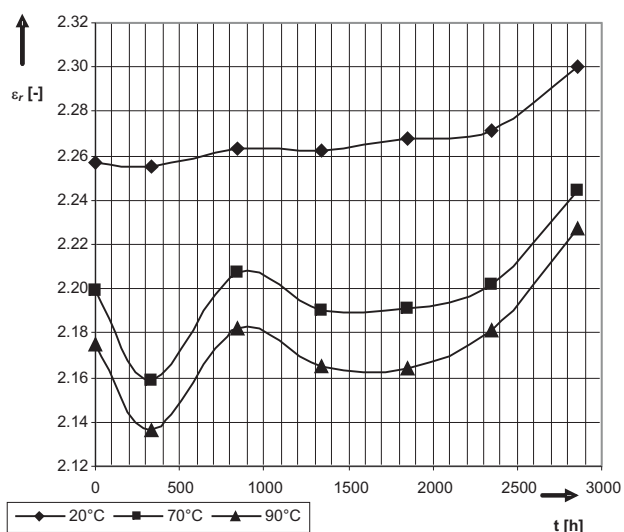
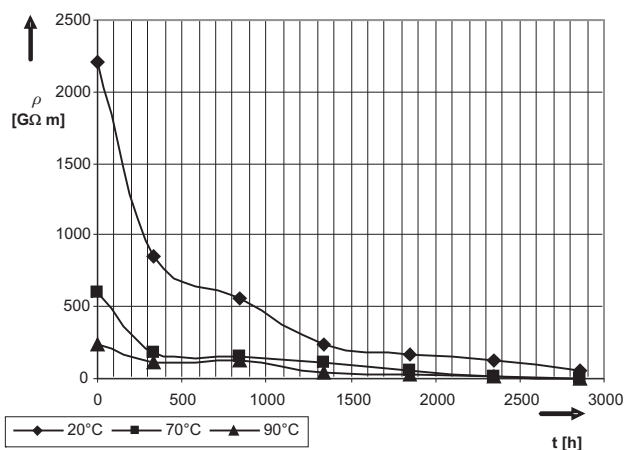
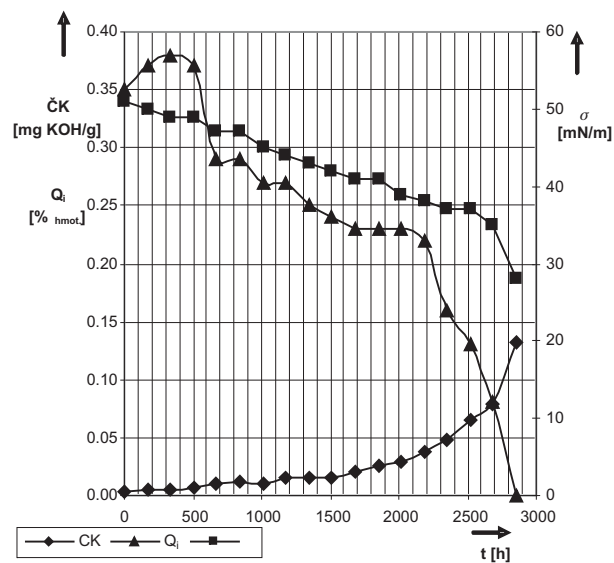
$\varepsilon_r$  – dielectric permittivity,

$\rho$  – volume resistivity,

ČK – determination of acidity,

$\sigma$  – determination of interfacial tension of oil against water,

$Q_i$  – contents of inhibitors.

Fig. 1: Dependence of  $\text{tg}\delta$ Fig. 2: Dependence of  $\epsilon_r$ Fig. 3: Dependence of  $\rho$ Fig. 4: Dependence of  $\check{C}K$ ,  $Q_i$ ,  $\sigma$ 

## 4 Conclusion

The oxidation stability of oil is evaluated by period of time until sediments for that are soluble in the insulating oil (insoluble in the n-heptane), or by the creation of sediments that are insoluble in insulating oil.

In the test of thermal-oxidation stability the submitted sample of insulating oil degraded in 2856 hours. This was documented by the presence of sediments insoluble in the n-heptanes. The thermal – oxidation stability test was carried out using, the ČEZ – ORGREZ method. The graphic dependencies were assessed from the monitored oil parameters.

## Acknowledgments

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Ing. Peter Semančík  
phone: +421 556 023 560  
e-mail: peter.semancik@tuke.sk

Doc. Ing. Roman Cimbala, Ph.D.  
phone: +421 556 023 557  
e-mail: roman.cimbala@tuke.sk

Department of Electric Power Engineering

Technical University in Košice  
Faculty of Electrical Engineering and Informatics  
Mäsiarska 74  
041 20 Košice, Slovak Republic

Doc. Ing. Karel Zális, CSc.  
phone: +420 224 352 369  
e-mail: zalis@fel.cvut.cz

Department of Electrical Power Engineering

Czech Technical University in Prague  
Faculty of Electrical Engineering  
Technická 2  
166 27 Prague 6, Czech Republic