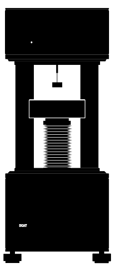
Application Note

Measuring interfacial tension of insulating liquids against water using the Du Noüy ring method according to IEC 62961

Monitoring the degradation of insulating liquids is crucial for detecting early signs of transformer faults and preventing catastrophic failures. Interfacial tension (IFT) of insulating liquids changes over time due to the formation of ageing by-products. This change is more significant in aged insulating liquids compared to new ones. The interfacial tension of insulating liquids against water has long been used as a criterion for evaluating ageing. IEC 62961 [1], 'Test methods for the determination of interfacial tension of insulating liquids – Determination with the ring method', is a widely used standard method for examining the quality of insulating liquds. This is a middle ground between the less accurate but fast ASTM D971 method and the precise but time-consuming EN 14210 procedure. In the following, the application of the IEC 62961 will be presented using a tensiometer of the DCAT series (Fig. 1) from DataPhysics Instruments.

Measurement device
Force tensiometers of the DCAT series



Measurement method
Du Noüy ring method

Measured quantities
Density
Surface tension
Interfacial tension

Environmental conditions
20 °C

Samples
Insulating liquid

Industries
Electrical applications
Transformers

dataphysics

Fig. 1: Force tensiometer DCAT 8T.

Technique and Method

According to the IEC 62961 standard, the Du Noüy ring method can be used to measure the interfacial tension (IFT) of insulating liquid against water. The IFT is evaluated by measuring the maximum force (F_{max}) a liquid lamella exerts on a wire ring while it is pulled out of a liquid. When the ring radius R is large compared to the radius r of the wire the IFT σ can be calculated based on the following equation:

$$\sigma = \sigma_{app} \cdot f = \frac{F_{max}}{2L} \cdot f = \frac{m_{max} \cdot g}{4\pi R} \cdot f$$

where L is the circumference of the ring, m_{max} is the maximum mass of the pulled lamella and g is Earth's gravitational acceleration of approximately 9.81 m/s^2.

The difference between the apparent and "actual" interfacial tension is corrected by the ring correction factor f. The tensiometer software DCATS offers correction factors according to various authors like Huh-Mason, Zuidema-Waters, or Harkins-Jordan.

A tensiometer of the DCAT series from DataPhysics Instruments is a universal measuring system for force-based studies of interfacial parameters and phenomena. With comprehensive software modules and standard test bodies, such as the Wilhelmy plate or the Du Noüy ring, it can determine the static, as well as time- and temperature-dependent IFT, thanks to its precise weighing system. Tensiometers are particularly useful for studying opaque liquids or liquids with similar refractive indices i.e., when the limits of optical contour analysis are reached.

Hence, a DCAT is the ideal device for measuring the interfacial tension of insulating liquid against water according to IEC 62961, as will be shown in this note.

Experiment

In this application note the interfacial tension of insulating liquids against water was determined using a DCAT 8T with a Du Noüy ring RG 11. According to the requirements of the standard IEC 62961, rings should be made of platinum or platinum-iridium alloy with a thickness not greater than 0,4 mm and a mean circumference of 60 mm (for example: inner diameter 18.7 mm, outer diameter 19.5 mm). It shall be suspended horizontally and connected to the tensiometer. A Du Noüy ring RG 11 fits those criteria and is therefore suitable in the test.

The measurement was conducted according to standard IEC 62961 requirement:

- The measurement temperature shall be between 18 °C and 25 °C. The water and insulating liquid shall be at the same temperature.
- The density of the insulating liquid shall be deter-mined at the temperature of measurement or can be calculated from a linear extrapolation of density from measurement at a standard temperature (e.g. 20 °C) to the temperature used for the IFT measurement.
- The correction of Zuidema and Waters is the preferred correction.
- Cylindrical glass vessel with a minimum diameter of 60 mm

In order to calculate the ring correction factor f the density difference of water and insulating liquid has to be known. Thus, the experiment was conducted in two parts: (I) Density determination of insulating liquid; (II) Interfacial tension measurement.

Note that the sample vessels were cleaned with water, followed by a rinse in acetone, then thoroughly rinsed again with tap and distilled water and dried before usage.

I: Density of insulating liquid

The density of the insulating liquid at room temperature was determined using a DCAT 8T tensiometer with the density determination set DIS 11 (Fig. 2). Using the DCATS 34 software module, the density of the insulating liquid can be evaluated automatically and was found to be 0.875 g/cm^3.

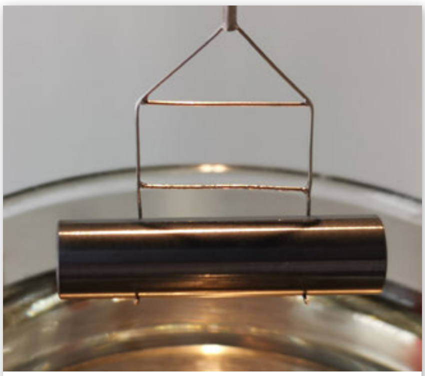


Fig. 2: Density determination of the insulating liquid using DIS 11.

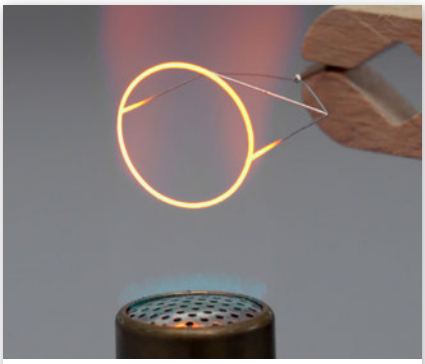


Fig. 3: The ring is annealed inside the flame of a Bunsen burner.

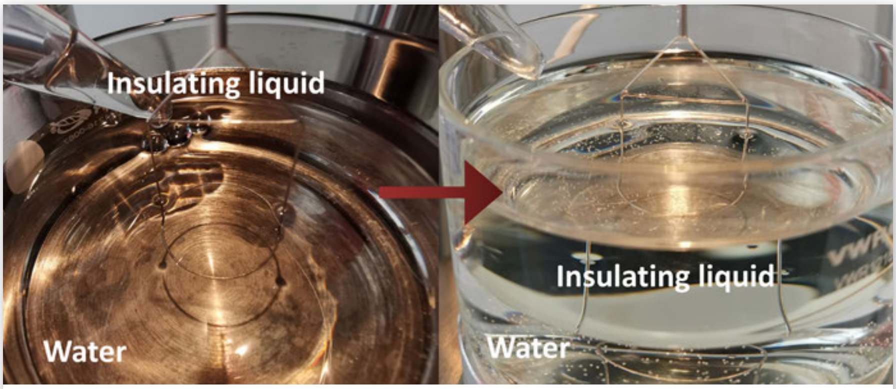


Fig. 5: The insulating liquid is carefully added onto the water surface using a pipette.

II: IFT measurement

a) The ring shall be cleaned with a suitable solvent and then by flame cleaning.

- Rinse three times with n-heptane, and afterwards with deionized water;
- Heat in the oxidizing flame for approximately 5 s in an ethanol or natural gas burner to red heat (Fig. 3).

b) To ensure the purity of the distilled water, the cleanness of the sample vessel and the accuracy of the ring dimensions, a preliminary test was carried out. The distilled water at room temperature was filled into a clean vessel with a filling level of around 25 mm (between 20 and 30 mm). No foam should be visible at the water surface. Foam indicates that the water is contaminated, or the vessel is not clean. The vessel was placed on the sample stage of the DCAT 8T. The ring was placed into the coupling lock of the measuring balance. The method 'Normal Ring – RG 11' in the software module 'Surface Tension' was selected and the surface tension of distilled water was automatically determined to be 71.61 mN/m, which is in good agreement with literature values[2] and lies within the standard range of > 70 mN/m (permission range of 18 °C to 25 °C)

Fig. 4: Method selection in DCATS.

c) After repeating step a) the ring was placed into the coupling lock of the measuring balance. The method 'IEC 62961' in the software module 'Interfacial Tension', was selected for the measurement (Fig. 4). The system automatically raised the sample stage until the ring was immersed in the water with a depth of 3.5 mm (> 3 mm). Then, the system waited for the insulating liquid to be added. The insulating liquid was carefully layered on the water surface using a pipette. Meanwhile the ring was still submerged in the water (Fig. 5 left). The insulating liquid was added until a layer thickness of about 16 mm (between 15 and 20 mm), as it should not cover the cross bar of the ring RG 11 (Fig. 5 right). This process shall be done within 30 s. After the last drop of oil was pipetted onto the water, begin the time counting. The oil-water interface was left undisturbed for around 150 s to form the interface.

d) Choosing 'OK' to continue in the software the sample stage was automatically lowered at a speed of 0.2 mm/s and the changes of lamella weight against position were recorded. The maximum weight of the lamella (Fig. 6) was detected by the software and the interfacial tension calculated automatically. It took around 180 s to complete the experiment, from the last drop of the insulating liquid was pipetted onto the water until the beak point was reached (within 180 s ± 30 s).

Results

The interfacial tension of the insulating liquid was determined to be 31.46 ± 0.01 mN/m. Based on the information provided from the supplier, the IFT of fresh insulating liquid is ranging from 40 to 50 mN/m. The lifetime of insulating liquid is considered to have expired when its IFT is lower than 24 mN/m. [3] Therefore, the tested insulating liquid is still of acceptable quality.

Summary

The tensiometers of the DCAT series with their comprehensive software from DataPhysics Instruments GmbH can be used to easily and reliably measure the interfacial tension of insulating liquid against water according to IEC 62961, and hence provides a fast and simple way to determine the quality of insulating liquid.

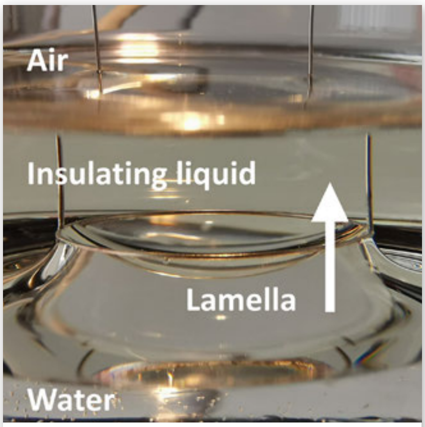


Fig. 6: A lamella forms when the ring is pulled out from the water layer into the insulating liquid layer.

References

[1] Standard IEC 62961, 2018, "Insulating liquids - Test methods for the determination of interfacial tension of insulating liquids - Determination with the ring method", ISBN: 9782832260371, https://webstore.iec.ch/en/publication/27653.

[2] N. B. Vargaftik, B. N. Volkov, and L. D. Voljak , “International Tables of the Surface Tension of Water”, Journal of Physical and Chemical Reference Data 12, 817-820 (1983), DOI: 10.1063/1.555688

[3] T. O. Rouse, “Mineral insulating oil in transformers”, in IEEE Electrical Insulation Magazine, vol. 14, no. 3, pp. 6-16, May-June 1998, DOI: 10.1109/57.675572.

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