Vol. XVI & Issue No. 10 October - 2023
INDUSTRIAL ENGINEERING JOURNAL

INVESTIGATION AND COMPARISON OF IN-SERVICE TRANSFORMER INSULATING OIL PROPERTIES USING VARIOUS ANALYTICAL TEST METHODS

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Abstract

In the existing situation, increased power consumption has put a significant burden on transformers, while power utility companies are cutting back on capital investments. Therefore, fault-free operation of the existing transformers becomes a principal concern for industrial consumers in ensuring their reliability. The main objective of this paper is to investigate and compare critical properties of insulating oil (mineral oil), such as breakdown voltage (BDV), water content, interfacial tension, and acidity, by using IEC, ASTM, and IS standard test methods. Repeatability and intermediate reproducibility tests of currently accepted standard test methods have also been performed to ensure accurate results. This paper will help professionals and analysts evaluate the process accurately based on the reported results to strengthen quality assurance.

Keywords: Mineral oil, transformer, breakdown voltage, precision, repeatability.

1. INTRODUCTION

The power transformer plays a vital role in synchronization between power generating stations, grids, distribution networks (such as substations), and consumer end [1, 2]. Therefore, the reliable operation of the power transformer becomes a major concern among the entire utility sector over the last decade. But, the squeezed budget of utility suppliers on their capital investment has increased a huge load on the existing transformers as it is the costlier equipment of the switchyard. So, a fault during its operation may invite the shutdown of the plant/premises which may create considerable monetary losses for the utility [3, 4]. The temperature inside the transformer rises as the load increases because of heating. Therefore, it is essential to dissipate the heat by adequate cooling, specifically under high-loading conditions [5, 6]. In transformers, several billion gallons of insulating liquid are being used to mitigate the heat's effect [7]. Mineral oil has always been and will continue to be the most commonly used dielectric liquid since the 19th century due to its widespread availability, good electrical insulation, cooling characteristics, and less cost [8-10]. It not only provides the cooling medium but it has better compatibility with cellulose and therefore the oil-impregnated paper becomes the tradition to be used inside the transformer for wrapping up winding. Furthermore, it offers a practical way to regularly assess the state of electrical equipment during its life span. Indeed, insulating liquid plays a significant role in keeping the transformer in good and healthy condition. It is primarily responsible for the functional serviceability of the insulation system, the condition of which can be a deciding factor when evaluating the service life of the transformer [5]. The liquid insulation tests for transformers are similar to the blood tests of humans because they can diagnose our health and can advise us by providing early-stage detections before any catastrophic failure [11]. Consequently, it is necessary to identify the degradation properties of transformer oil. Test methods that comply with regulatory criteria of the American Society for Testing and Materials (ASTM), the International Electro-Technical Commission (IEC), and the Indian Standard (IS) are frequently used to investigate various properties of mineral oil. However, the results obtained from these test methods may get different because of their different equipment, chemicals, reagents, and standard of procedures. As a result, it is crucial to devise reliable and appropriate test procedures for mineral oil testing and analysis.

The paper investigates crucial characteristics of mineral oil, such as physical, chemical, and dielectric properties, as these properties influence mineral oil replacement and rectification in in-service transformers. These properties are typically reviewed as breakdown voltage (BDV), water content, interfacial tension (IFT), and neutralization number (acidic value). It also includes statistics on the accuracy, precision, repeatability, and intermediate precision of the different test methods available for these properties in a concerted effort to find an accurate and precise analysis for mineral oil.

2. VERIFICATION PARAMETERS AND THEIR IMPACT ON ANALYTICAL METHODS

Method verification, often known as the process of supplying verified evidence that the method achieves what it is designed to do, ensures reliability in everyday use. The primary goal of the verification is to confirm the analytical method's accuracy, precision, suitability, and dependability. In this paper, the tests are performed for the existing standard test methods of IS, IEC, and ASTM with regard to system suitability and real sample testing. The typical verification characteristics of the analytical procedures (as illustrated in Table 1) are generally performed after development. The present research work only evaluated some of them, which needed to be compared while establishing their accuracy and precision.

2.1 Accuracy and Precision

The accuracy of an analytical method is the closeness of the test results produced by that method to the true value. It is often referred to as trueness. The accuracy of an analytical method should indeed be ascertained within its limits. Whereas, the precision of an analytical method is the level of consistency among independent test findings when the method is applied repeatedly to multiple samplings of a homogeneous sample. It is measured as numerically in terms of standard deviation (Std. dev.) or relative standard deviation (RSD) of a set of the proximity of test findings derived by that method to measurements.

Table 1. Typical verification parameters.

S. No.	Performance Characteristics	Verification
1	Ruggedness	Usually performed before validation
2	Selectivity	Not Examined
3	Sensitivity	Not Examined
4	Limit of Detection (LOD)	Considered as given in the test method
5	Limit of Quantitation (LOQ)	Considered as given in the test method
6	Analytical Range	Considered as given in the test method
7	Linearity	Not Examined
8	Accuracy	Examined
9	Precision	Examined
10	Repeatability	Examined
11	Reproducibility	Examined
12	Measurement Uncertainty	Not Examined

2.2 Repeatability and Intermediate Precision

Repeatability is the consistency of measured values across repeated observations of the same sample, performed under the same circumstances, such as at the same location, by the same analyst, using the same method or procedure, and on the same equipment, whereas the intermediate precision is the consistency of measured values between measurements of the same sample performed under changed conditions, such as a different place, a different analyst, different equipment, or any of these [12]. In the present study, repeatability is performed by the same analyst (i.e., analyst #1) on the second day with the same sample, and the same setup at the same place in the same environmental conditions. Whereas intermediate precision is performed on the same sample with the same setup and same conditions by analyst #2.

3. EXPERIMENTAL WORK AND DISCUSSION

3.1 Sample Preparation

The poor sample collection techniques may influence the test results because of air and dust contaminations, which can lead to erroneous conclusions concerning quality and, in addition, result in a loss of time, effort, and expense in securing, transporting, and testing the sample. Therefore, it is crucial to exercise the utmost precaution while extracting the oil sample from the transformer. In this work, the procedure for sampling as given in [13] is adopted for the collection of oil samples from a transformer. While collecting the samples, the safety cap is first removed, and the transformer's outlet valve is then wiped with a dry cloth. Then the valve was slowly opened, allowing some of the oil to escape from the valve mouth to assure the clearance of any impurities in the valve's orifice, and then connected the sampling kit to the transformer valve. After collecting some oil in the aluminum bottle, the bottle was rinsed with it 2-3 times. After successive cleaning, the oil sample was collected slowly and gently, allowing the oil to flow at a constant rate against the wall of the bottle to avoid the formation of air bubbles in the oil so that no air can be trapped in the sample. The bottle was filled to approximately 98% of its capacity. After sampling the oil, the sampling kit is removed from the transformer, the valve of the transformer is closed, and then the aluminum bottle is tightly closed with a cap and covered with insulation tape, and a sticker is pasted on it for identification of the sample details. In this study, a total of nine (09) oil samples are collected in dry, clean, and non-permeable aluminum bottles (as prescribed in IEC 60475:2022) [13] from the bottom valve of the main tank of an in-service transformer using a sampling kit (as illustrated in Figure 1). Bottles were tightly sealed and kept shielded from light until ready to be tested.

Figure 1. (a) Arrangement of sampling kit (b) Sample containers.

(a) Sampling kit (b) Sample Containers

3.2 Oil Testing and Analysis

The analysis of oil samples is performed by analyzing their dielectric and chemical properties such as breakdown voltage (BDV), water content, interfacial tension (IFT), and acidity according to the SOPs mentioned in national and international standards. Table 2 presents the various test methods used in the current experimental work. All tests have been carried out repeatedly to obtain more accurate results. Moreover, the standard deviation (σ) and relative standard deviation (%) have also been derived to assess the degree of dispersion of the data points concerning their mean and the precision of the average test results, respectively.

3.3 Breakdown Voltage (BDV)

The dielectric strength of insulation oil is a critical element of its capacity to effectively prevent an electric arc and hence behave as an insulant. As a result, when the voltage applied to the insulating oil rises, the current flowing through it also rises rapidly, leading the oil to lose its natural insulating characteristics and turn into a conductor. This phenomenon is called the breakdown of the insulating oil. It is generally expressed in kilovolts (kV).

Table 2. Performance characteristics with the detail of standard test methods [14-25].

S. No.	Performance Characteristics	Method No.	Test Method	Title of standard
1	Breakdown Voltage (BDV)	Method #1	ASTM D1816-12R19	Dielectric Breakdown Voltage of Insulating Liquids using VDE Electrodes
		Method #2	IEC 60156:2018	Insulating Liquids -Determination of the Breakdown Voltage at Power Frequency
		Method #3	IS 6792:2017	Determination of the Breakdown Voltage at Power Frequency (2nd Rev.)
2	Water Content (WC)	Method #1	ASTM D1533-20	Standard Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration
		Method #2	IEC 60814:1997	Insulating Liquids – Oil Impregnated Paper and Pressboard - Determination of Water by Automatic Coulometric Karl Fischer Titration
		Method #3	IS 13567:2018	Insulating Liquids - Oil-impregnated Paper and Pressboard - Determination of Water by Automatic Coulometric Karl Fischer Titration- (1st Rev.)
3	Interfacial Tension (IFT)	Method #1	ASTM D971-20	Standard Test Method for Interfacial Tension of Insulating Liquids Against Water by the Ring Method
		Method #2	IEC 62961:2018	Insulating Liquids - Test Methods for the Determination of Interfacial Tension of Insulating Liquids - Determination with the Ring Method
		Method #3	IS 6104:1971 (RA2021)	Method of Test for Interfacial Tension of Oil Against Water by the Ring Method
4	Acidity	Method #1	ASTM D974:2014	Standard Test Method for Acid and Base Number by Color-Indicator Titration
		Method #2	IEC 62021-2:2007	Insulating Liquids - Determination of Acidity - Part 2 - Colourimetric Titration
		Method #3	IS 16863-2: 2018	Insulating Liquids- Determination of Acidity – Part 2 - Colourimetric Titration

The transformer insulating oil's breakdown voltage (BDV) reveals its ability to tolerate electric stress without failing. Although it is not an inherent property of insulating oil, it is used to detect contaminants in the oil such as water, fibers, dirt, and solid particles [14, 26, 27]. The result of breakdown voltage depends on the equipment structure, applied voltage, electrode geometry (shape, material, and space), test frequency, etc [28]. Therefore, the current research work measures the BDV of the oil sample at ambient temperature using three different SOPs or Test Methods [14-16]. Table 3 differentiates the ASTM, IEC, and IS test methods based on their experimental setup and procedure. In method # 1, the electrode gap was fixed at 2.0 mm, and the A.C. supply was used with a frequency of 50 Hz. The electrode axis was horizontal. The voltage across the electrodes gradually increases from 0V at a rate of 0.5 kV/s until it reaches a maximum voltage just before the sample breakdown. A total of five consecutive (05) breakdowns were performed and recorded, and the mean ($\bar{x}$) was calculated in kV (kilovolt). In methods # 2 and 3, according to the standard test methods [15, 16],VDE (Verband Deutscher Elektrotechniker) electrodes with a 2.50 ± 0.05 mm electrode gap were used and a test voltage (A.C.) was applied. The volume of the test oil sample was 350 ml. The applied voltage across the electrodes gradually increased from 0V at the rate of 2.0 ± 0.2 kV/s until breakdown occurs. Six (06) breakdowns were executed in a row, with an interval of two minutes between each breakdown, and the mean () was computed. The stirring option was available (which was not available in method # 1), so the oil sample was stirred during the test in both methods (i.e., methods #2 &3).

Table 3. Comparison of Specifications of BDV Standard Test Methods [14-16].

S. No.	Setup Specification	Method #1 (ASTM D1816-12R19)	Method #2 (IEC 60156:2018)	Method #3 (IS 6792:2017)
1.	Origin	USA	Europe	India
2.	Electrodes Shape			-
3.	Electrodes gap (mm)	2.0	2.5	2.5
4.	Type of Electrodes	VDE	VDE	VDE
5.	Magnetic Stirring	NO	Optional	Optional
6.	Sample Temp (°C)	Ambient	15-25	-
7.	Ambient Temp (°C)	20-30	20 °C	-
8.	Applied Voltage	AC	AC	AC
9.	Voltage Rate (kV/s)	0.5	2.0	2.0
10.	Frequency (Hz)	45-65	48-62	48-62
11.	Pause time (Minutes)	1-1.5	2.0	2.0
12.	The time between filling & start of the test (min.)	3-5	5	-
13.	No. of Observations	05	06	06
14.	Test vessel	Requires cover or baffle to prevent air	Must be transparent	-

Table 4 illustrates the results of three distinct breakdown voltage (VBDV) tests. The standard deviation (σ) and the relative standard deviation (%) have also been calculated in addition to the mean value.

Table 4. Test results of breakdown voltage (BDV).

S. No.	Determinations	Breakdown Voltage (kV)		
		Method #1 (ASTM D1816-12R19)	Method #2 (IEC 60156:2018)	Method #3 (IS 6792:2017)
1	Observation #1	75.4	76.2	74.5
2	Observation #2	76.6	72.4	75.8
3	Observation #3	70.6	75.7	73.3
4	Observation #4	73.5	73.2	71.2
5	Observation #5	75.2	74.8	72.7
6	Observation #6	-	75.9	75.2
V_{BDV} (Mean Value) ($\bar{x}$)		74.3	74.7	73.8
Standard Deviation (σ)		2.0	1.6	1.7
Relative St. Dev. (%)		2.80	2.09	2.32

The graphical presentation of BDV test results for all the test methods is presented in Figure 2. The average breakdown voltage (kV) is observed as 74.3, 74.7, and 73.8 in methods #1, #2, and #3, respectively. The accuracy and intermediate precision of method#2 are found to be 1.6 and 2.09, respectively, which is somewhat better than methods #1 and #3. Moreover, the accuracy and precision of the methods are evaluated by performing repeatability and reproducibility on the same sample by different analysts on different days. The results are summarized in Table 5 and presented graphically in Figure 3. This time also, the accuracy of method#2 (i.e., IEC 60156:2018) is observed better. It might be due to the differences in voltage ramp-up speed and electrode gap compared with ASTM D1816. Besides, the IEC electrode shape provides a more uniform electric field.

Figure 2. Trending curves of the observed test results of BDV.

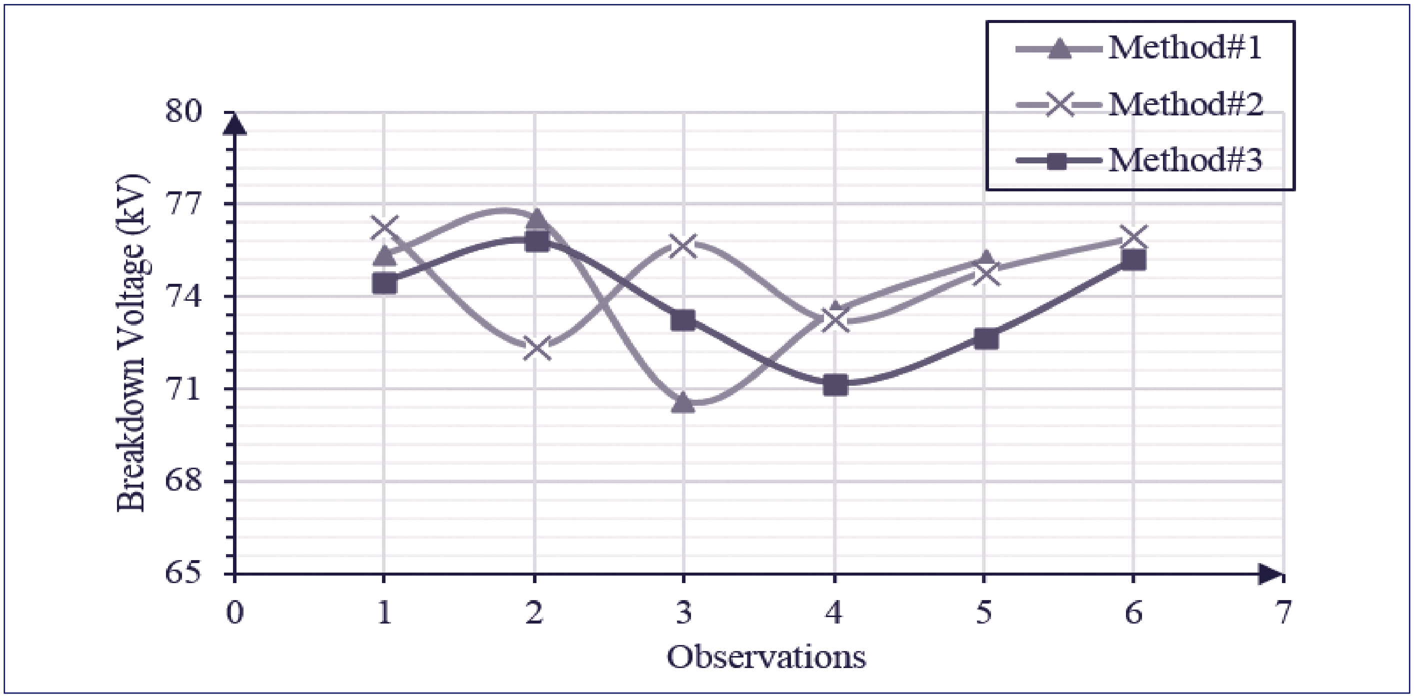
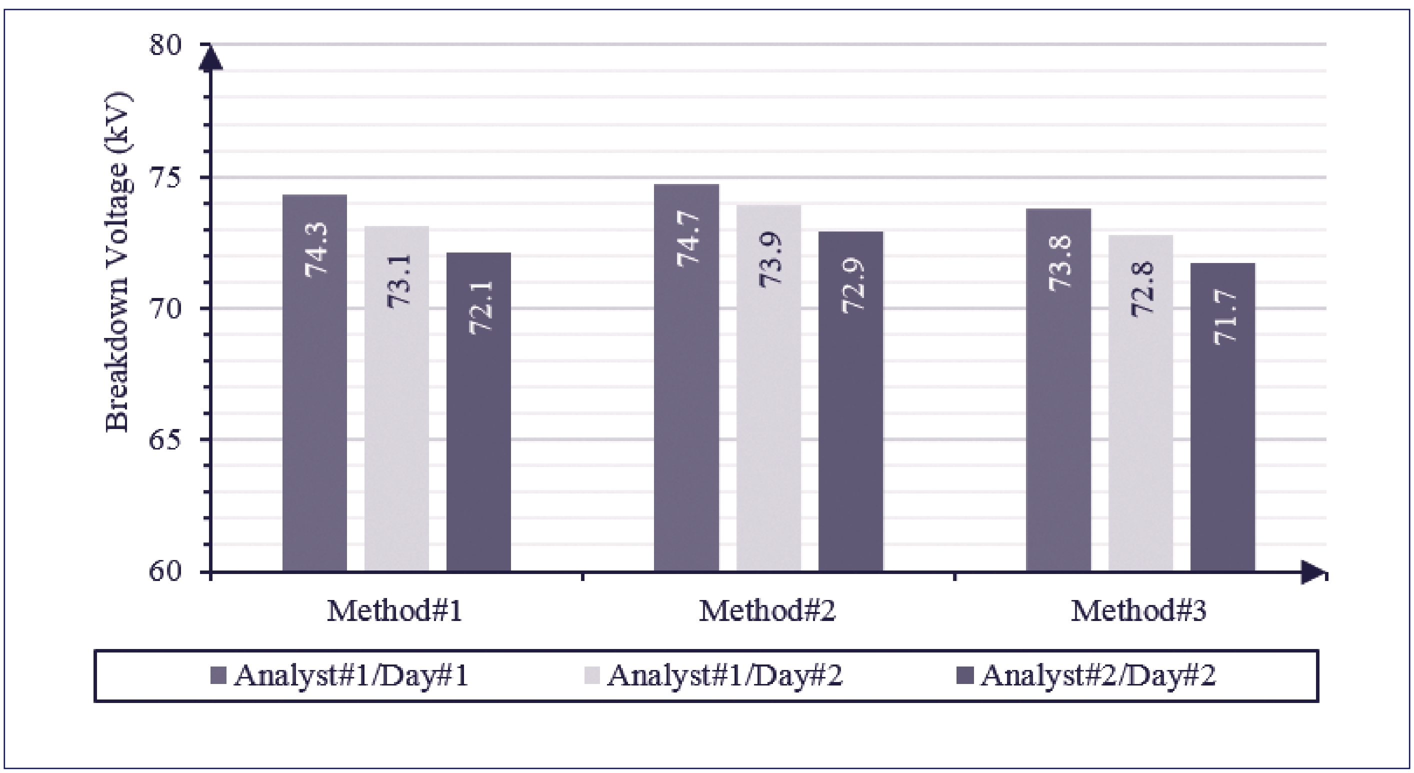


Figure 3. Graphical view of repeatability and reproducibility of BDV test results.



3.4 Water Content

The presence of high moisture content in mineral oil reduces the insulation system's dielectric property and expedites the aging of the paper insulation, so it is essential to ensure low moisture content in new transformers and keep it low in operating transformers [29]; low moisture content is identified as an advantageous characteristic because it can obtain a reasonable breakdown voltage and low dissipation losses. Only high moisture content can indicate an abundance of polar components, which can be detrimental to transformer insulation and mitigate the safety factor [30]. When a transformer is filled with oil, the paper, due to its hygroscopic nature, absorbs moisture from the oil, affecting its insulating property and thus reducing its life [31]. The amount of water in the transformer's insulation affects its longevity; it tends to increase electrical conductivity and dissipation factor while decreasing electric strength. Water content and breakdown voltage are inversely related [32, 33]. The standard test methods [17-19] have been performed to investigate the water content in the collected oil sample. Two readings of each test method have been taken and calculated the mean of them. Method #1 [17] is based on the reduction of iodine-containing reagents according to the traditional Karl Fischer reaction. The oil sample was injected into a titration cell where the iodine consumed by the reaction with water is electrolytically regenerated by anodic oxidation of iodide. The titration cell consists of a sealed vessel containing an anode and cathode separated by a diaphragm. A magnetic stirrer was used during the test.

In Method #2 & #3 [18, 19] (both are merged), the test sample was mixed with a base/alcohol solution of iodide ions and sulfur dioxide. Iodine was generated electrolytically and reacted with water as shown in reactions (1) and (2).

$$H_2O+I_2+ SO_2 + 3C_5H_5N \rightarrow 2C_5H_5N.HI + C_5H_5N.SO_3 \quad (1)$$

$$C_5H_5N.SO_3+CH_3OH\rightarrow C_5H_5NH.SO_4.CH_3 \quad (2)$$

Iodine is generated in proportion to the quantity of electricity according to Faraday's law, as shown in reaction (3)

2I^--2e→I2 (3)

Table 5. Comparative analysis of BDV (kV) test results w.r.t. repeatability and reproducibility.

S. No.	Determinations	Method #1 (ASTMD1816-12R19)			Method #2 (IEC 60156:2018)			Method #3 (IS 6792:2017)		
		Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2	Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2	Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2
1	Observation #1	75.4	75.9	74.5	76.2	75.9	74.2	74.5	75.6	75.5
2	Observation #2	76.6	72.3	71.8	72.4	73.8	72.4	75.8	73.7	71.6
3	Observation #3	70.6	70.4	74.3	75.7	72.4	70.8	73.3	71.1	69.1
4	Observation #4	73.5	71.1	71.6	73.2	73.1	71.3	71.2	72.3	70.3
5	Observation #5	75.2	75.8	68.3	74.8	73.6	75.1	72.7	69.8	72.4
6	Observation #6	-	-	-	75.9	74.8	73.4	75.2	74.2	71.1
V_{BDV} (Mean Value) ($\bar{x}$)		74.3	73.1	72.1	74.7	73.9	72.9	73.8	72.8	71.7
Standard Deviation (σ)		2.0	2.3	2.2	1.6	1.2	1.7	1.7	2.1	2.2
Relative St. Dev. (%)		2.80	3.14	3.05	2.14	1.62	2.33	2.30	2.88	3.06

Table 7. The repeatability and reproducibility test results of water content.

S.No.	Determinations	Method # 1 (ASTM D1533-20)			Method # 2 (IEC 60814:1997)			Method # 3 (IS 13567:2018)		
		Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2	Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2	Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2
1	Observation #1	6.4	6.2	6.3	6.2	6.3	6.3	6.3	6.4	6.5
2	Observation #2	6.2	6.5	6.5	6.4	6.6	6.5	6.5	6.5	6.5
Mean Value ($\bar{x}$)		6.30	6.35	6.40	6.30	6.45	6.40	6.40	6.45	6.50
Standard Deviation (σ)		0.14	0.21	0.14	0.14	0.21	0.14	0.14	0.07	0.00
Relative St. Dev. (%)		2.24	3.34	2.21	2.24	3.29	2.21	2.21	1.10	0.00

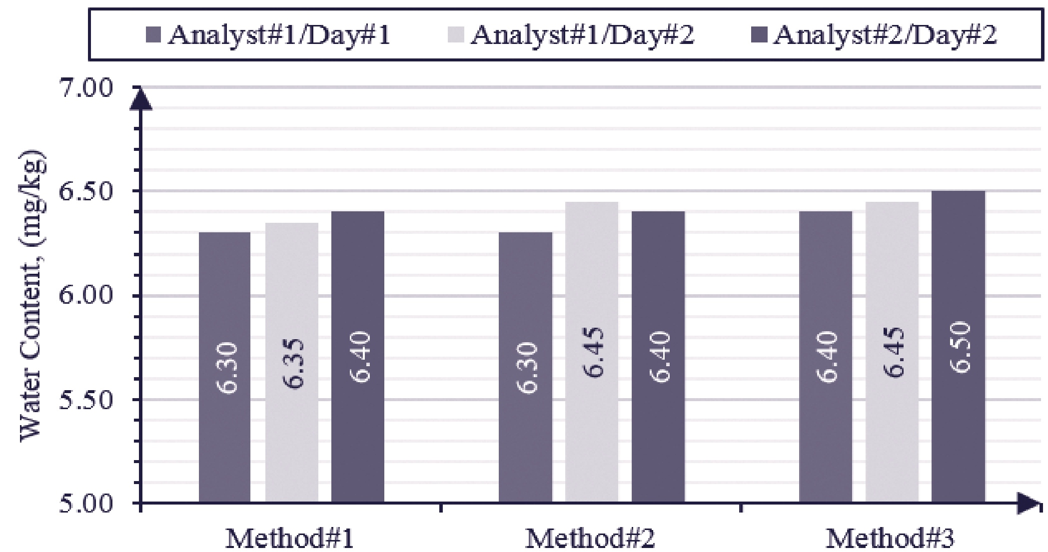
Two observations of each test method have been recorded and indicated in Table 6. The average/mean test results of water content (mg/kg) are observed as 6.30, 6.30, and 6.40 in method#1, method#2, and method#3, respectively. The accuracy of all the test methods is observed same (i.e., 0.14 mg/kg), whereas the precision of method#1, method#2, and method#3 is 2.24, 2.24, and 2.21 mg/kg, respectively.

Table 6. Test results of water content.

S.No.	Determinations	Water Content (mg/kg)		
		Method # 1 (ASTM D1533-20)	Method # 2 (IEC 60814:1997)	Method # 3 (IS 13567:2018)
1	Observation #1	6.4	6.2	6.3
2	Observation #2	6.2	6.4	6.5
Mean Value ($\bar{x}$)		6.30	6.30	6.40
Standard Deviation (σ)		0.14	0.14	0.14
Relative St. Dev. (%)		2.24	2.24	2.21

Repeatability and reproducibility of the same sample are performed by different analysts, and the findings obtained are listed in Table 7, while Figure 4 presents a graphical view of them. According to the results, it is found that there is a negative correlation between breakdown voltage (BDV) and the water content in an insulating liquid (mineral oil). Breakdown voltage (BDV) and water content are inextricably related and have an inverse relationship [26,34].

Figure 4. Graphical view of repeatability and reproducibility of water content test results.



3.5 Interfacial tension (IFT)

Interfacial tension (IFT) is the force of attraction between the particles of two immiscible liquids (for example, oil and water) [35]. It provides a way of detecting soluble polar contaminants and products of degradation [26, 36]. It is commonly considered to be an effective index for evaluating deterioration with increasing age, due to its high sensitivity to contaminants and impurities. The IFT decreases as the concentration of contaminants rises. As a result, the acidity rises and the oil darkens [37, 38]. Fresh insulating oil is likely to have a high IFT (at least 40 mN/m), while insulating oil samples from an in-service transformer with an IFT of less than 25 mN/m are deemed in poor condition. IFT and the acidity of insulating oil are typically inversely related. The IFT value decreases while the acidity rises when the oil oxidizes [7, 39]. The standard test methods [20-22] have been performed to investigate the IFT of the collected oil sample. Table 8 presents the test results of all three test methods. Table 9 gives a comparison view of all three standard test methods.

Table 8. Test results of IFT.

S.No.	Determinations	IFT (mN/m)		
		Method # 1 (ASTM D971-20)	Method # 2 (IEC 62961:2018)	Method # 3 (IS 6104:1971R21)
1	Observation #1	39.6	39.7	39.6
2	Observation #2	39.7	39.8	39.6
3	Observation #3	39.7	39.8	39.7
Mean Value ($\bar{x}$)		39.67	39.77	39.63
Standard Deviation (σ)		0.06	0.06	0.06
Relative St. Dev. (%)		0.15	0.15	0.15

Table 9. Comparison view of standard test methods for IFT [20-22].

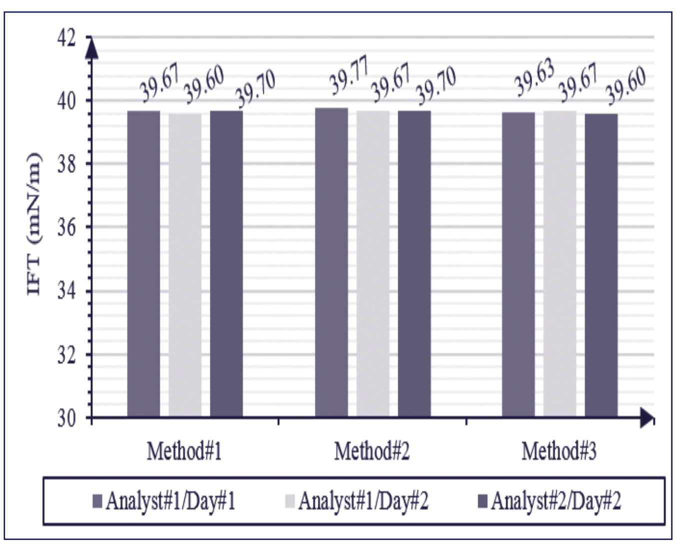
S.No.	Parameters	Method#1 (ASTM D971-20)	Method#2 (IEC 62961:2018)	Method#3 (IS 6104:1971R21)
1	Material of ring	Platinum	Platinum/ Iridium	Platinum
2	Circumference of ring (mm)	40	60	40/60
3	Wire diameter (mm)	0.3	≤ 0.4	0.3
4	Equilibrium condition	Non-equilibrium modus	Close to equilibrium	Non-equilibrium
5	Oil-water interface age (sec.)	60	180 ± 30	30 ± 1
6	Acceptable surface tension range for water	70-74 mN/m	70-73 mN/m	0.071-0.072 N/m
7	Water density check	Every time	Once/day is enough	Every time

The accuracy and precision of all three methods are found to be the same at 0.06 and 0.15, respectively. However, the highest (Mean value) interfacial tension of transformer oil measured as 39.77 using method#2, as compared to 39.67 and 39.63 using method#1 and method#3, respectively. It may be the following reasons; (a) larger circumference (60 mm) of the ring. (b) an equilibrium time of 180 seconds after pouring the oil onto the surface of the water, which provides a realistic expression of the real interfacial tension. Figure 5 illustrates a graphical picture of the repeatability and reproducibility of water content test results and Table 10 displays the repeatability and reproducibility test results for all three test methods.

Table 10. The repeatability and reproducibility test results of IFT.

S.No.	Determinations	Method # 1 (ASTM D971-20)			Method # 2 (IEC 62961:2018)			Method # 3 (IS 6104:1971R21)		
		Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2	Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2	Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2
1	Observation #1	39.6	39.5	39.7	39.7	39.7	39.6	39.6	39.7	39.5
2	Observation #2	39.7	39.6	39.6	39.8	39.7	39.7	39.6	39.7	39.6
3	Observation #3	39.7	39.7	39.8	39.8	39.6	39.8	39.7	39.6	39.7
Mean Value ($\bar{x}$)		39.67	39.60	39.70	39.77	39.67	39.70	39.63	39.67	39.60
Standard Deviation (σ)		0.06	0.10	0.10	0.06	0.06	0.10	0.06	0.06	0.10
Relative St. Dev. (%)		0.15	0.25	0.25	0.15	0.15	0.25	0.15	0.15	0.25

Figure 5. Graphical picture of repeatability and reproducibility of IFT test results.



3.6 Acidity

The amount of potassium hydroxide (KOH), measured in milligrams (mg), needed to neutralize the acid in 1 gram (gm) of transformer insulating oil is the acidity (Acid Number-AN). The more acid there is in the oil, the higher the acid number. There is hardly any acid in new transformer oils [40]. An acidity test can be performed to evaluate the state of the transformer oil. The aging rate of the paper insulation can also be estimated using this method [41]. Due to the reaction between the oxidation products released by the insulating oil and the paper insulation, acids and sludge are produced in transformer oil [42]. The standard test methods [23-25] have been conducted to investigate the acidity number present in the oil test sample. Table 11 illustrates the specification chart of all three test methods for acidity. Whereas, Table 12 shows test results of acidity.

Table 11. Specification chart of standard test methods for acidity [23-25].

S.No.	Parameters	Method#1 (ASTM D974:2014)	Method#2 (IEC 62021-2:2007)	Method#3 (IS 16863-2:2018)
1	Burette least count (ml)	0.02	0.001	0.001
2	Titrant molarity (M)	0.1	0.05	0.05
3	Volume of sample (g)	20	5	5
4	Solvent volume (ml)	100	10	10
5	Solvent type	50.0 % - Toluene 49.5 % -2-propanol 0.50% - DM water	100% - 2-propanol	100% - 2-propanol
6	Indicators	p-Naphtholbenzein	Alkali blue 6B	Alkali blue 6B
7	Titration method	Manual	Manual	Manual

Table 12. Test results of acidity.

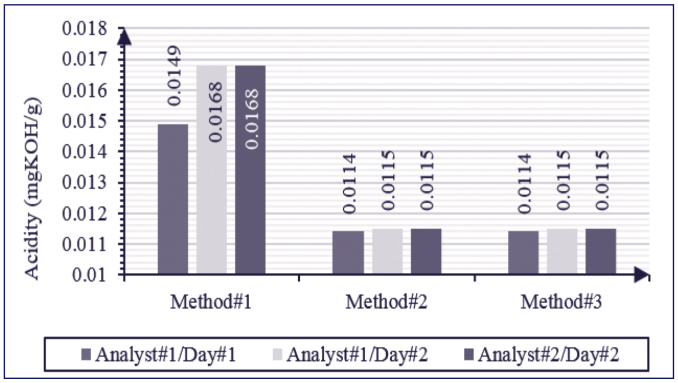
S.No.	Determinations	Acidity (mgKOH/g)		
		Method # 1 (ASTM D974:2014)	Method # 2 (IEC 62021-2:2007)	Method # 3 (IS 16863-2:2018)
1	Observation #1	0.0112	0.0112	0.0117
2	Observation #2	0.0168	0.0117	0.0112
3	Observation #3	0.0168	0.0112	0.0112
Mean Value ($\bar{x}$)		0.0149	0.0114	0.0114
Standard Deviation (σ)		0.0032	0.0003	0.0003
Relative St. Dev. (%)		21.65	2.54	2.54

The average/or mean test results of acidity (mgKOH/g) were observed as 0.0149, 0.0114, and 0.0114 in methods #1, #2, and #3, respectively. The accuracy of the test methods #1, #2, and #3 was observed as 0.0032, 0.0003, and 0.0003, respectively, whereas the precision of methods #1, #2, and #3 was 21.65, 2.54, and 2.54 mgKOH/g, respectively. Table 13 shows the repeatability and reproducibility test results, while Figure 6 illustrates its graphical view.

Table 13. Repeatability and reproducibility test results of acidity.

S.No.	Determinations	Method # 1 (ASTM D974:2014)			Method # 2 (IEC 62021-2:2007)			Method # 3 (IS 16863-2:2018)		
		Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2	Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2	Analyst#1 Day#1	Analyst#1 Day#2	Analyst#2 Day#2
1	Observation #1	0.0112	0.0168	0.0168	0.0112	0.0112	0.0112	0.0117	0.0112	0.0112
2	Observation #2	0.0168	0.0168	0.0168	0.0117	0.0117	0.0117	0.0112	0.0117	0.0117
3	Observation #3	0.0168	0.0168	0.0168	0.0112	0.0117	0.0117	0.0112	0.0117	0.0117
Mean Value (x)		0.0149	0.0168	0.0168	0.0114	0.0115	0.0115	0.0114	0.0115	0.0115
Standard Deviation (σ)		0.0032	0.0000	0.0000	0.0003	0.0003	0.0003	0.0003	0.0003	0.0003
Relative St. Dev. (%)		21.65	0.00	0.00	2.54	2.50	2.50	2.54	2.50	2.50

Figure 6. Graphical view of repeatability and reproducibility test results.



4 CONCLUSIONS

The objective of this paper was to investigate the properties of insulating oil (mineral oil) using various analytical test methods accessible at the national (IS) and international levels (ASTM and IEC). The insulating oil characteristics like breakdown voltage (BDV), water content, interfacial tension (IFT), and acidity have been investigated. Moreover, to find better accuracy and precision, the repeatability and reproducibility of the same sample have also been performed. The results obtained from the experimental work are summarized below:

- Higher accuracy for measurement of breakdown voltage (in kV) has been observed in method#2 (IEC 60156:2018) as compared to method#1 (ASTM) and method#3 (IS). This might be due to the differences in voltage ramp-up speed and electrode gap compared with ASTM D1816. Moreover, the IEC electrode shape provides a more uniform electric field.
- In the case of water content, the ASTM and IEC test methods findings have been found to be identical, whereas the IS test method results differed marginally (see Table 6).
- For the measurement of interfacial tension (IFT), the accuracy and precision of all the test methods (method#1, #2, and #3) were discovered to be the same, measuring 0.06 and 0.15, respectively. However, the highest (mean value) interfacial tension of transformer oil was measured as 39.77 using method#2 as compared to 39.67 and 39.63 using method#1 and method#3. This may be the following reasons; (a) larger circumference (60 mm) of the ring. (b) equilibrium time of 180 sec. after pouring the oil onto the surface of the water which provides a realistic expression of the real interfacial tension.
- In the acidity example, the mean of the test results of methods #1, #2, and #3 are found to be 0.0149, 0.0114, and 0.0114, respectively, and the accuracy was found to be 0.0032, 0.0003, and 0.0003, respectively, which indicates that IEC and IS have the same accuracy as compared to ASTM. It has also been found that the variables, such as the molarity of the titration, type of solvent, burette size, least-count of the burette, and droplet size of the titrant affect the acidity results in a significant manner.

Based on the practical experience of the work presented, it is observed that all the methods have their own specifications and accuracy and continue to advance with time and expert efforts. Extreme caution should be exercised when performing tests, as small gaps can lead to considerable changes. The presented work will be helpful to researchers, academicians, and relevant professionals in this field.

ACKNOWLEDGMENTS

The authors would like to express their gratitude to Spectrum Diagnostic Laboratory & Research Centre (SDLRC), New Delhi, India for providing a productive environment for the completion of testing, technical assistance, and the sharing of laboratory procedures, standards, and test methods and special thanks to Mr. Poshetty Serla, Chief Quality Control Manager, SDLRC. The authors also express their appreciation to Om Stirling Global University, Hisar, Haryana, India for their continuing support in completing the research work.

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