COMPARATIVE STUDY OF MOISTURE REDUCTION TECHNIQUE IN MINERAL OIL

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This report is submitted to the Faculty of Electrical Engineering of UTeM as a partial fulfilment of the requirement for the degree of Bachelor of Electrical Engineering with Honours. The member of the supervisory is as follow:

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(Project Supervisor)

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A report submitted

In fulfilment of the requirement for the Bachelor of Electrical Engineering with honour

Faculty of Electrical Engineering

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I hereby, declared this report entitled “*Comparative Study of Moisture Reduction Technique in Mineral Oil*” is the result of my own research except as cited in references.

Signature: ………………………………..

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Date: ……………………………….

For my beloved parents and family whom without failed to give their full support till the completion of this project. Their love, passion and support help me to do weekly progression and complete the experiment and project report.

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ABSTRACT

The uses of the transformer are important for generation, distribution and transmission of the electrical system network. High dielectric strength and cooling performance of its insulating oil are properties that the transformer depends on in order to maintain normal operation. Several factors affected the dielectric insulation problem is moisture, acidity, pressure and suspended particle. 0.01% presence of moisture in the transformer oil can decrease 20% of the breakdown voltage of the oil. Thus, this project is focusing on reducing the moisture content in the mineral oil which results in improvement of the breakdown voltage. Three techniques of moisture reduction have been used which are Vacuum Oven (VO) technique, Nitrogen Saturated (NS) technique, and Molecular Sieves (MS) technique. The performance of breakdown voltage (BDV) and amount of moisture reduce are measured to determine the most effective technique. Karl Fischer Coulometric (KFC) complies with ASTM D1533 standard is used meanwhile for breakdown voltage (BDV) Megger OTA60PB follows the standard ASTM D1816. The analysis of experimental data shows among VO, NS, and MS techniques, the MS no filer techniques has 82.33% reduction of moisture content but BDV has not been tested due to MS particle will effect the BDV test. But, NS has 80.79% moisture reduction which has increased the BDV of mineral oil with enhancement 150%. The low moisture content value by the NS because theoretically when nitrogen gas saturated in mineral oil, the nitrogen gas replaced the dissolved oxygen in the oil. The result is an enhancement of the performance of the mineral oil. MS with a smaller pore size (0.22μm) has a better result than VO and (MS) bigger pore size (1μm) because the smaller pore size has filtered the moisture from re-dissolving with the good oil. Overall, NS technique has proven the most effective technique throughout the experiment and the best solution to reduce moisture from the mineral oil.

ABSTRAK

Penggunaan pengubah adalah penting untuk penjanaan, pengedaran dan penghantaran rangkaian sistem elektrik. Pengubah bergantung kepada kekuatan dielektrik yang tinggi dan prestasi penyejukan minyak penebat untuk mengekalkan operasi normal. Beberapa faktor yang mempengaruhi masalah penebat dielektrik ialah kelembapan, keasidan, tekanan dan zarah yang digantung. Kehadiran kelembapan 0.01% dalam minyak pengubah dapat mengurangkan 20% daripada volum kerosakan minyak. Oleh itu, projek ini memberi tumpuan kepada pengurangan kandungan kelembapan dalam minyak mineral yang menghasilkan peningkatan voltan kerosakan. Tiga teknik pengurangan kelembapan telah digunakan iaitu teknik Ketuhar Vakum (VO), teknik Nitrogen tepu (NS), dan teknik Molecular Sieves (MS). Prestasi volum kerosakan (BDV) dan jumlah kelembapan yang rendah diukur untuk menentukan teknik yang paling berkesan. Karl Fischer Coulometric (KFC) mematuhi piawaian ASTM D1533 digunakan sementara untuk voltan kerosakan (BDV) Megger OTA60PB mengikut standard ASTM D1816. Analisis data percubaan menunjukkan antara teknik VO, NS, dan MS, teknik MS tidak mempunyai pengurangan 82.33% kandungan kelembapan tetapi BDV belum diuji kerana MS zarah akan mempengaruhi ujian BDV. Tetapi, NS mempunyai pengurangan kelembapan 80.79% yang telah meningkatkan BDV minyak mineral dengan peningkatan 150%. Nilai kandungan lembapan yang rendah oleh NS kerana secara teorinya apabila gas nitrogen tepu dalam minyak mineral, gas nitrogen menggantikan oksigen terlarut dalam minyak. Hasilnya adalah peningkatan prestasi minyak mineral. MS dengan saiz liang yang lebih kecil (0.22µm) mempunyai hasil yang lebih baik daripada saiz lubang VO dan (MS) yang lebih besar (1µm) kerana saiz liang yang lebih kecil telah menyaring kelembapan daripada larut semula dengan minyak yang baik. Secara keseluruhan, teknik NS telah membuktikan teknik yang paling berkesan sepanjang percubaan dan penyelesaian terbaik untuk mengurangkan kelembapan dari minyak mineral

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LIST OF ABBREVIATIONS

ASTM - American Society for Testing and Material
DDF - Dielectric Dissipation Factor
DGA - Dissolved Gas Analysis
DORMS - Dissolved Oxygen and Moisture Removal System
FR3 - Vegetable Oil Envirotemp
KP - Kraft Paper
RBPO - Refined Bleached Palm Oil
PFAE - Palm Fatty Acid Ester
VDE - Verband Deutscher Elektrochiniker
FTIR - Fourier-Transform Infrared Spectroscopy
FO - Fresh Oil
VO - Vacuum Oven
NS - Nitrogen Saturated
MS - Molecular Sieve
ppm - Part per million
CCL_2F_2 - Freon
CO_2 - Carbon Dioxide
OTS60PB Oil Test Set 60 Portable

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CHAPTER 1

INTRODUCTION

1.1 Overview

This chapter describes the research background, motivation, problem statement, objectives and scope research. In the research background of the project will explain the purpose and significance of projects. While a short brief of issues that need to avoid and solved is explained in the problem statement. In objective, the goal of the project is been explained in the scope of research.

1.2 Research Background

Power transformers are valuable assets in electrical power network. The performance of a power transformer mostly depends on its insulation system. Hence, it is vital for the insulation system to have the ability to withstand all the various service conditions. As it say the better the insulation condition, the longer the life span of transformer.

Oil immersed transformer is the most widely used to date. The oil immersed transformer is still based on mineral oil. Due to generalization and standardization, the production of the transformer is a worldwide hit. Now the industry is going toward plant-based insulating oil but technology and knowledge are still lacking. The oil immersed transformer uses liquid insulation material as a heat transfer agent [1].

The lifespan of the insulating oil used in power transformer suffers from thermal, electrical and environmental stresses during operation services. Deteriorating and aging process of the insulation may be accelerated and can be a direct potential threat to the equipment safety. Slow and irreversible changes in material properties will result in a process called aging. Hence, oil become decomposed and oxidized which result in transformer losses and reduce its reliability [2].

The heat that comes from the transformer winding may cause acceleration of the reaction between unstable hydrocarbon molecular oil and oxygen, by product degradation cellulose paper and excess dielectric loss at high temperature [3]–[5]. Therefore, improving the mineral oil dielectric strength properties is important for improving the lifespan of the transformer.

1.3 Motivation

Mineral oil is widely use insulating oil for transformers but there is still a room for improving its performance or properties. This is due to the low cost, oil oxidation stability, cooling performance and good dielectric strength. However, the process of aging produces a by–product contributes to increment of moisture content. The existences of this harmful moisture is a well know threat to all liquid insulation that potentially decrease the breakdown voltage, increases paper degradation rate, decrease inception voltage in partial discharge and increase risk of bubbling with moisture content in paper. The dielectric strength and performance of transformer will decrease when there are existences moisture in transformer[6], [7]. Hence, this study has proposed a lot of research method for reducing moisture in the oil.

1.4 Problem Statement

There are many techniques proposed in the literature to reduce the moisture content in the insulating liquid but most of the techniques are costly and time consuming. But, there is still proper guideline available even there are many practices has been done in the industry without identifying the most effective to reduce moisture

Hence, this research work aims to finds the effectiveness of the proposed methods focusing only on three methods, molecular sieves and filter, bubble and heating. These three methods have different working principle. Therefore, the most convenient method with an effective moisture reduction is proposed in this work based on experimental results. Even so, the presence or existence of moisture can be put into three parts that are chemically bound water, physically dissolved water and free water [7]. These methods are chosen due to the availability of equipment available in the lab. The three methods in theory able reduce moisture content in the insulating oil and which is easy to conduct due to its simple concept.

1.5 Objective

Based on the title, “Comparative Study of Moisture Reduction Technique in Mineral Oil”, the following objectives below are pursued:

- To compare the effectiveness of different techniques in reducing moisture from mineral oil
- To determine effect of molecular sieve on mineral oil
- To analyse the effect of moisture on Breakdown Voltage (BDV) on mineral oil.

1.6 Scope of Research

In this research the mineral-based insulating oil used is Nytro Libra. The dielectric strength and its moisture content are measured. Three methods of moisture reduction are used in example molecular sieves and filter, bubble and heating. Filter paper with a smaller pore size (0.22µm) and bigger pore size (1µm) are used in this experiment. The method used to measure the moisture content in the mineral oil is using Karl-Fisher method. Breakdown voltages are tested at MeggerOTS60PB Portable oil Tester

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter gather theory regarding transformer, including the insulating materials of a transformer and their properties of the insulating material. Methods or techniques reducing moisture for commercial users and for the previous work also provided.

2.2 Transformer

Transformer operates by the principles of electromagnetic induction from one circuit to another, either stepping up or stepping down the voltage. The construction of transformer consists of the iron core, windings, insulating material and bushings.

Transformers in electric power delivery systems around the world are mostly oil-filled. The oil transformers are as an electrical insulation and as a heat transfer fluid. Most of the transformer is petroleum-based which is refined specifically to meet the requirements. In electric power distribution and transmission systems, the transformers are to expected function reliably and efficiently for many years. The quality of the oil plays an important role in a transformer performance[1]

2.3 Insulating Material of Transformer

The insulating material is essential in making the transformer to work properly. One insulator alone is not enough to ensure the safety due fault can occur in any type of form for the transformer. There are three categories that use the transformer that is solid, liquid and gas[9].

2.3.1 Solid Insulation

A good mechanical and bonding strength is the specification of a good solid insulation. Materials used for solid insulation are organic and inorganic material. If a solid insulator is perfectly homogeneous and doesn‟t have any imperfection the breakdown voltage can be high.

The largest insulation inside transformer is the solid insulation. The forms of this solid insulation are electro-technical (Kraft) paper and pressboard. The functions of this solid insulation are mechanical stability, the direction of oil flow, electrical insulation and creation of space. Hence, every material has its limit same goes to this solid insulation where the aging will affect the properties of the mechanical and electrical part. Where aging of the solid insulation depending on the water and temperature [9].

Figure 2.1: Structure of a transformer.

The figure 2.1 above and figure 2.2 below shows the component inside the transformer. The insulating paper is used to separate each winding and cover the conductor. The paper comes in numerous forms like different in structure, density and thickness depending on the type of application of transformer. As so, the pressboard for insulation of high-voltage is soaking with liquid insulation[10].

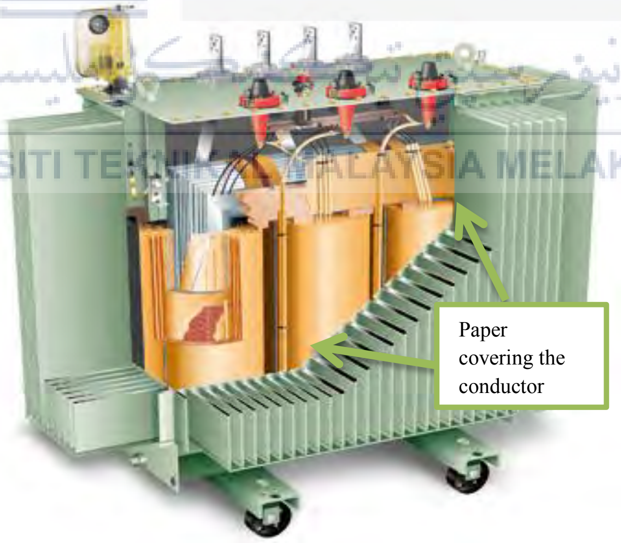

Figure 2.2: More detail location of insulating paper

2.3.2 Liquid Insulation

From figure 2.3 below show that‟s liquid insulation in transformer does not only act as an insulator but also as heat dissipation agent which help to cool the transformer system. Losses in the transformer can be categorized as “no-load losses” and “load losses” which is caused by the existing of the magnetic and electrical field. Types of losses are converted into the copper winding, steel sheet core and other conductor parts. The losses in this transformer will lead to increase of temperature in transformer if the transformer system is not cooled, hence overheating the equipment will lead to fatal damage to the transformer and increased the rate of aging[6], [10].

Thus, it is vital for a transformer to have the transformer oil as it acts as a cooling medium by extracting the heat to the coolant. The low viscosity of oil also allows it to penetrate the solid insulation setting up convection current for conveying heat from core material to the radiators. Hence, to operate for a long period and at high voltage the liquid insulation oxidation has to be stable.

The liquid portion of an insulating system plays an important role and able to withstand the designed and calculated stress. It is due to the transformer can increase in temperature during operation. Furthermore, to prevent and protect the transformer from overheating is by using liquid insulation. The normal factor of BDV for both and liquid insulation are temperature, humidity and water content. The temperature will determine the interactions between two insulations. Hence, all normal and abnormal transformers operating condition the transformer oil should serve as a solvent that can tolerate with the transformer [10].

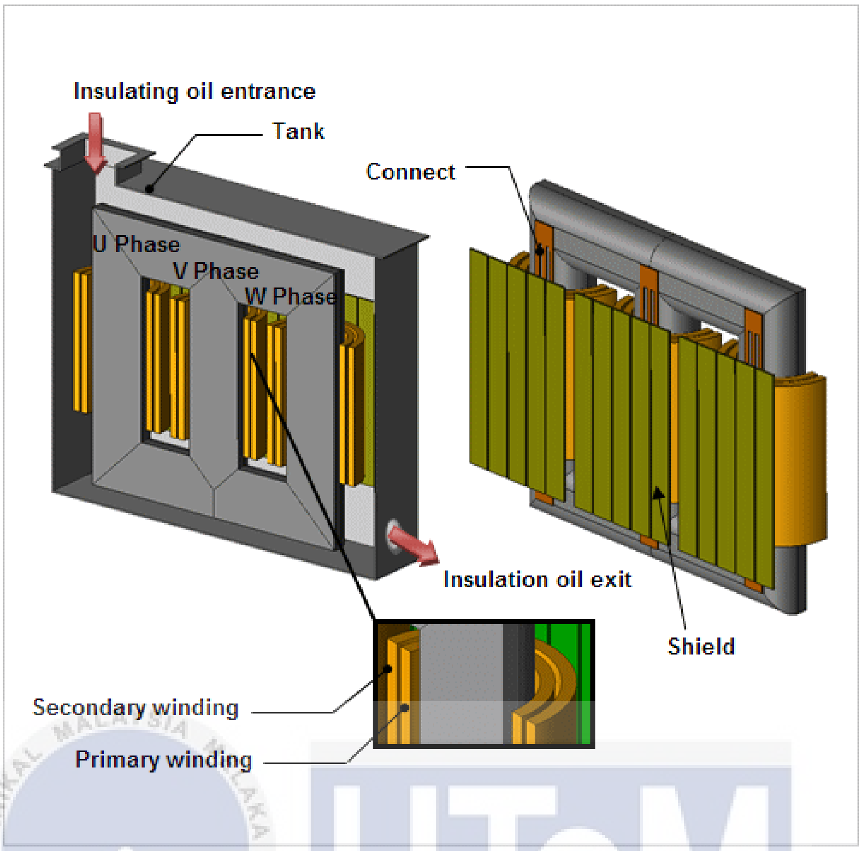

Figure 2.3: Cooling System of a Transformer

2.3.3 Gas Insulation

Air at atmospheric pressure is the most common gasses insulation. The breakdown of gasses occurs when collision ionization between free electron and gas molecule. Chemical inertness and stability are properties that normally chose as an insulating gas. Nitrogen (NS), carbon dioxide (CO_2), Freon (CCL_2F_2) and sulfur hexafluoride (SF_6) are some of the common insulating gasses. An ideal condition for gas insulation is the vacuum, where the absence of gas molecule prevents the collision between molecules. Hence, for the ideal condition will result in a large breakdown voltage.

2.4 Properties of Insulating Material

There are 2 types of oil to be compared, palm fatty acid or vegetable oil and mineral oil. Mineral oil always has been a go-to product for the transformer oil mainly due to its low cost. The other features that make it still in manufacture are it good dielectric strength and as a cooling system for the oil-immersed transformer. The births of this mineral oil are from the refined crude oil. Crude oil consists of many individual hydrocarbon compounds which are complex and the relative amount of these hydrocarbons can be quite different in crude oil from different sources. Acceptable transformer oil varies substantially in composition, like the crude oil from which they are derived. Standard of mineral oil can be seen at [11].

There are not specified limit have been made or stated for the concentration of the various hydrocarbons in the transformer oil. To analyse it is plainly impractical for the several hundred individual molecular species present. The hydrocarbon species somehow reflect the behaviour of broad class the many characteristics of the transformer oils. Of the entire hydrocarbon compound in the transformer oils, virtually are the member of three classes: alkanes, naphthenes, and aromatic hydrocarbons. Alkanes are saturated linear (normal) and branched (iso-) paraffin (n-hexane and 2,3- dimethylbutane are low molecular weight examples of linear and branched alkanes respectively).

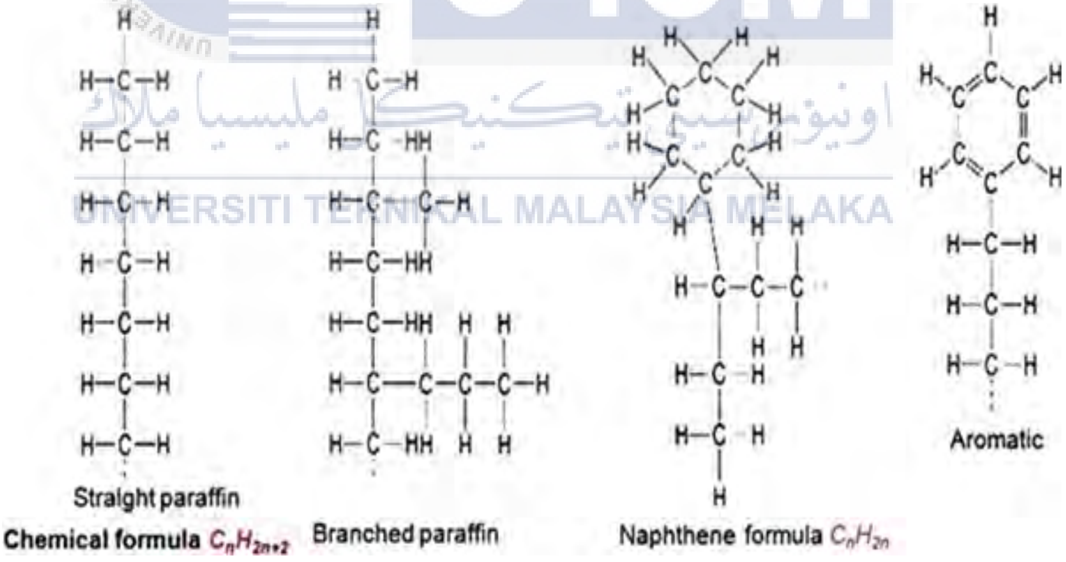

Figure 2.4: A molecular hydrocarbon structure of classes in mineral oil.

Naphthenes are saturated paraffin whose structures include rings of cyclized five, six or seven carbons (cyclohexane is a low molecular weight of napthene). These simple ring structures may be two or more and fuse together in napthene molecule (decahydro - napthene – “decalin”- is the example of a napthene with two fused six-member rings). In naphthene, the structure may have linear or branched alkane side chains.

For aromatic hydrocarbons, they contain unsaturated ring moieties. A discrete benzene ring structure can found only in mono-aromatic hydrocarbon (e.g. methyl benzene-toluene, biphenyl); but for that contain two or more benzene rings that fused together are called poly-aromatic hydrocarbons (e.g. naphthalene anthracene). For mono and poly aromatic hydrocarbon contain discrete or fused benzene rings may have alkane side chains and naphthenic ring substituents. Acceptable transformer oil can be made from “paraffinic” crude oils that are richer with the help of modern technology. The molecular weight of the average hydrocarbon compound in a distillate fraction can be estimated from its boiling range.

The average molecular weights are estimated at 255 in naphthenic oil distillate and 300 in this paraffinic oil distillate. Then, the naphthenic and the paraffinic distillate contain 18 and 21 carbon atoms in the average hydrocarbon. The limit specified for viscosity and flash point of transformer oils determine the boiling temperature range over which distillates can be collected from a particular crude oil in the production of transformer oil. An oil viscosity is colligative properties that reflect the interaction of the entire molecule of different size and structure present in the hydrocarbon ensemble. Higher boiling is included in the ensemble in the paraffin rich distillate, higher molecular weight hydrocarbons that are absent in the naphthenic distillate, the transformer oil made from both meet the specified viscosity limit[1].

For vegetable oil which is a natural ester or the synthetic ester that is categorized as a general classification for the transformer oils. In the late 1880s, vegetable oil or esters have been used as dielectric liquid since the invention of the oil-filled transformer. Due lack of knowledge and modern tool, the earliest natural ester was found to be incompatible with free breathing equipment, due to their natural oxidation characteristics and were gradually replaced by mineral oils[12].

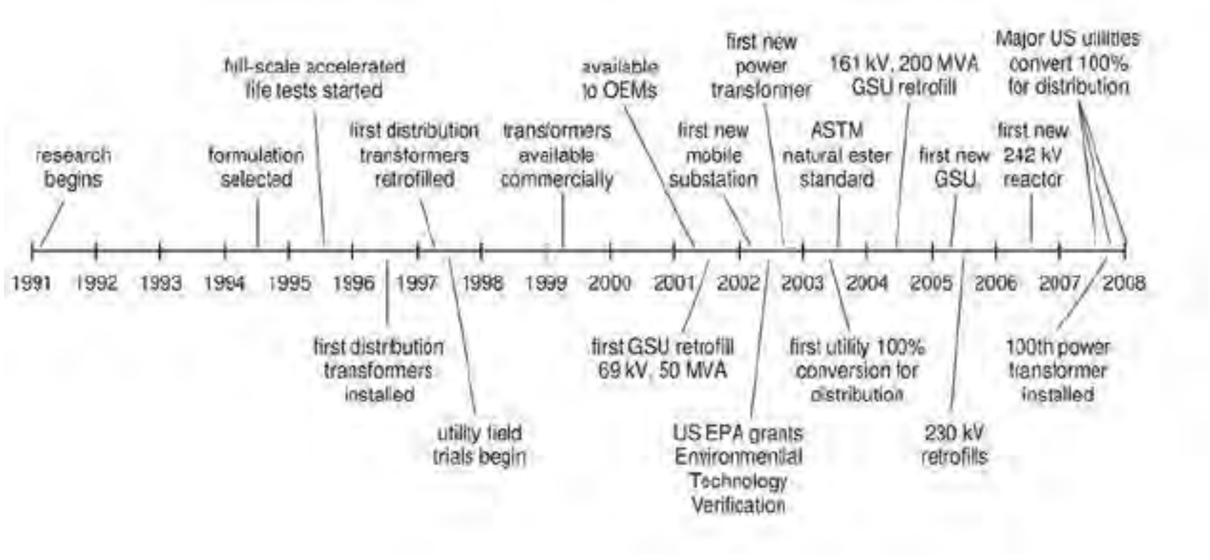

Figure 2.5: Development of natural ester as transformer insulating liquid.

Due to the toxicity and low firing point that the mineral possess which can be dangerous to the environment, because of this the requirement the electrical power transmission and distribution industry to look for a viable alternative to mineral oil. To be acceptable, any environmentally acceptable alternative must be safe, economical, and offer a high standard of electrical performance over a long working life. Nowadays, vegetable oil is a hit due to their green credentials. Natural esters are produced from vegetable oils, which are manufactured from plant crops. The advantages esters oil offer are high fire point as well as good biodegradability, but all type of natural ester suffer from not being oxidation stable as other types of insulating liquids. Even though, there are many varieties of crop oil to produced natural ester liquid, most commonly produced natural ester for electrical applications are from soya, rapeseed and sunflower oil. This is due to factors such as availability, cost and performance characteristic[12].

2.4.1 Moisture

The meaning of moisture is simply the quantity of water contained in a material. Water can be present in 3 forms that are free water, physically dissolved water, chemically bound water. Free water is when absorption limit is exceeded; free water separates from the oil. This can happen when a transformer with moist solid insulation cools down and can maybe affecting the cold start behaviour of a transformer.

For physically dissolved water mostly can be found in aged insulating oil. These substances behave as solutizer for water and change it to finely emulsified form. The drastic reduction of the dielectric properties of the oil and jeopardising the oil operation of the transformer is due to the physically absorbed water in cellulose fibres. Finally, is the chemically bound water where is hard to remove by the conventional way and also can be found only in aged oils. The only way for removal of this chemically bound water is if the binding substances (acids, carbonyl compounds, surfactants) are removed at the same time. The chemically bound is only released at higher temperatures[7].

Table 2.1: Literature Review on past paper relating to moisture measurement content

Reference Number	TITLE	METHOD USE
[2]	S. Province, "Study on the Impact of Initial Moisture Contents on Insulation," pp. 1–4, 2016.	• Four group oil-paper insulation samples were prepared with 1%, 2%, 3%, 4% moisture content under laboratory condition. • After undergo ageing test, all four group were test with Karl Fischer to measure moisture content
[13]	A. A. Suleiman, N. A. Muhamad, N. Bashir, Y. Z. Arief, M. N. Abdul Rahman, and B. T. Phung, "Moisture effect on conductivity of kraft paper immersed in power	• The moisture-in-oil samples were increased by 0.01%(100ppm) ,0.02%(200ppm),0.07%(700ppm), 0.2%(2000ppm), 0.3%(3000ppm).

	transformer vegetable-based insulation oils," IET Gener. Transm. Distrib., vol. 11, no. 9, pp. 2269–2274, 2017.	- Karl Fisher Titration was used to measure the quantity of moisture in materials

The paper for [2], is about study accelerated thermal aging with different moisture content under laboratory condition. The oil used in this paper is naphthenic mineral insulating oil then four group oil-paper insulation samples were prepared with different moisture content under laboratory conditions. The four group samples were prepared with 1%, 2%, 3%, 4% moisture content under laboratory condition. Then the ageing process was setup to see the effect of moisture in the oil-paper insulation. The research result in this paper shows that the effect of moisture on oil-paper insulation is not a superposition but mutual coordination. The more moisture it contains the more the acids content, but with the additional condition that is temperature.

For [13], is about study presents the effect of moisture increase in vegetable oil and its effect on its dc conductivity of Kraft paper (KP) samples. The oil used in this paper was vegetable oil Envirotemp (FR3), palm fatty acid ester (PFAE), and refined bleached deodorised palm oil (RBDPO). The moisture content of the entire oil sample was increased by directly introducing distilled water droplet into the vegetable oil. Moisture in oil samples were increased by 0.01% (100ppm), 0.02% (200ppm), 0.07% (700ppm), 0.2% (2000ppm) and 0.3% (3000ppm). The final result shows that the moisture migrated from the oil insulation and immersed into the dry KP. Hence, the increase of moisture in the oil samples will also increase the conductivity of the dry KP. The different of [2] and [13] is the type of Karl Fisher equipment use and where moisture was introduced in the setup and what standard used in this procedure.

2.5 General Practice in Handling Moisture inside Transformer

Table 2.2 summary general practices below, for the latest transformer report have a low BDV and reduced the high water content[14]. This is the action or option, each with different characteristics and consequences.

Table 2.2: Summary of general practice related to the study.

No	Action	Process Description	Conclusion
1	Do Nothing	The failure of the unit is caused by continuous degradation. The water in paper insulation and oil will lead to breakdown the designed value. Permanent failure and replacement are needed as a result.	At first cost was inexpensive, then next cost will be extreme costly
2	Retro fill	The wet oil is exchange with dry oil but moisture still exists deep within the transformer insulation such as paper insulation. After process retro fill is done, high moisture keep reappearing due to moisture will slowly leach from the paper into new oil.	Moisture remains in the paper insulation but oil temporary is at good condition after retro fill with risk of unchanged oil. The cost is only of outtake cost
3	Conventional dehydration	Removing the moisture from the outer layers of the insulation is a temporary solution by circulates and heats the transformer oil but still moisture stays deep within the paper. The moisture will slowly leach into newly processed oil even after	The process will lead high power consumption and risk of unchanged still occurs. Good result only temporary moisture

		the dehydration unit. After some time the breakdown voltage will be lower.	still remains due hidden behind good oil during process.
4	Field vacuum dehydration	Rated for full vacuum inside the transformer, which the transformer can be disconnected and de-energized from the system. Same step as conventional dehydration by circulates and heats the transformer oil. The unit is drained and placed under vacuum condition after reach certain temperature. The moisture removing from the internal component begins from the combination of heat and vacuum. This process requires a constant and periodic application for a full and proper moisture removal.	The process requires day and week for a full and proper process. The transformer has to be de-energized. Then, the transformer has to be refilled with oil and electrically tested which risk of vacuum tank could be lead to failure of the sealing. Even the effective is good at remove moisture but it comes with high outage costs.
5	Oven Drying	The wet transformer can be dried in an oven same as in factory during assembly of the transformer. As usual, it needs to be disconnected and transported into a drying oven. The drying could take a period of days and week. Reverse process of installing back the transformer for operation.	Cost is very high which involve money and time but it only effective to a small transformer.

2.6 Review on Previous Related Works

Review on the previous study on moisture removal technique, for comparison and improved the technique of moisture removal for transformer insulating oil. The table below shows the summary of the literature review based on the purposed of this study.

Table 2.3: Summary based on previous research related to the study

Ref	Author	Descriptions	Remarks
[15]	J.Sabau et al (2010)	• Study on Dielectric Dissipation Factor (DDF), Dissolved Gas Analysis (DGA) and Water Content of the oil before and after removal process • "Blanketing system" that is referred in the paper where the use of nitrogen gas as cushion to seal the transformer. The upper side of the system conservator is filled with nitrogen where air contain moisture and need to be remove before sealing • Introduces new system of nitrogen blanketing system called "Dissolved Oxygen and Moisture Removal System" (DORMS). • Moisture content was measure using Karl Fisher Titration in unit part per million (ppm).	○ No reason is said for choosing a nitrogen gas ○ Electrical properties such as BDV of oil if not measure before and after moisture is being reduce or (DORMS) ○ No reason is said for heating oil until temperature for moisture removal system. ○ No explanation on how reaction between nitrogen gas and insulating oil during moisture removal process.

[16]	H.F.Jin (2015)	• Study the dielectric strength and thermal conductivity of mineral oil based on Nano fluids with the study moisture content of mineral oil before and after per-processing of oil. • Pre-processing of oil is dry mineral oil in vacuum oven at 60ºC for the moisture removal system.	○ Electrical properties such as BDV of oil if not measure before and after moisture is being reduce. ○ No explanation about the heat oil the temperature for moisture removal system ○ No explanation about reaction inside the vacuum oven during moisture removal system
[17]	H.B.H Sitorus (2014)	• Investigation of physiochemical and electrical properties of Jatropha Curcas Methyl Esther. • Pre-processing is Jatropha Curcas Methyl Esther with the aid of molecular sieve to reduces the moisture content. • Process need 2 hour mixing with the molecular sieve with temperature between 60ºC - 75ºC with 2.2% molecular sieve weightage.	○ No explanation about reaction between molecular sieve and transformer oil during moisture removal system ○ Only Jatropha Curcas Methyl Esther oil was experiment during the process no other transformer oil involves. ○ No explanation about the percentage for molecular sieves for moisture removal system ○ No explanation about the temperature and 2 hour mixing for moisture removal system

2.7 Summary

In this chapter discussed, the theory and principle that related to this work based on previous research. The selection of insulation is the liquid insulation which common and made from petroleum call as mineral oil. When the oil degradation is reduced, this will result in a longer life span of the transformer which it is important for maintenance and cost for some manufacturer. In this research, the selected brand of mineral oil available in the UTeM Oil Lab is Nytro Libra.

Nytro Libra Mineral Oil is a product from Nynas Company. A brand new Nytro Libra colour is colourless. The mineral oil will changes colour to darker colour when the oil started to age. It is due to the oil is being used for a long period without maintenances. The existing of moisture somehow influences the aging process, especially when moisture content in the transformer is high. Standard relating to mineral oil is ASTM D3487. The required property of insulating mineral oil which considers as in good condition when the oil:

- Minimum BDV : 20K (1mm gap of electrode)
- Maximum Moisture Content : 35ppm

Thus, by reviewing previous work discussed in table 2.2, there are techniques are has been identified as a moisture removal system. The three-technique has been confirmed and to compare are Vacuum Oven (VO), Nitrogen Saturated (NS) and Molecular Sieves (MS). The reason nitrogen is selected like in the previous work because the gas is environmentally friendly, inexpensive, available in the UTeM Oil Lab and chemically inert. As for Vacuum Oven technique the temperature 60ºC because it was average operating condition temperature through equilibrium of product and dissipated loss heat in the transformer is reached [10]. Lastly, the molecular sieves are used because to see the reaction molecular sieves and mineral oil to find out whether the effect of moisture removal in the mineral oil.

Finally, for measurement and testing equipment uses are Karl-Fisher method for moisture content in the insulating oil and breakdown voltages are tested at MeggerOTS60PB Portable oil Tester. Standard selection for the Karl- Fisher equipment is according to ASTM D1533. Step on using Karl Fisher is available in the ASTM D1533. For the BDV is ASTM D1816, with electrode type VDE or mushroom and 1mm gap are used.

CHAPTER 3

METHODOLOGY

3.1 Introduction

The methodology contains the techniques and steps used in the project. Some experimental procedures in the project came from the previous work. Moisture reduction procedure such as Nitrogen Saturated (NS), Molecular Sieves (MS), Vacuum Oven (VO), BDV and Karl Fisher coulometer will be discussed briefly.

3.2 Project Methodology

The project methodology is a defined combination of logically related practices; method and process that determine how best to develop and deliver a project throughout the continuous implementation process until successful completion and termination. This also to makes sure the project on track with the time given and keep track of the timeline of the project.

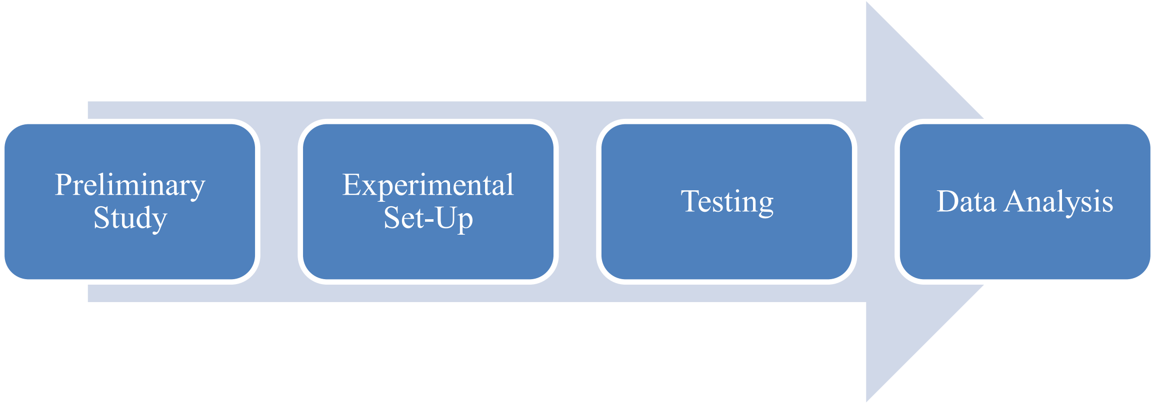

Figure 3.1: Flow chart of the project methodology

From the flow chart in figure 3.1, there are four stages of this project methodology which is preliminary study, experimental set-up, testing and finally data analysis. For preliminary study and experimental setup, consist of literature review, selection of transformer oil, moisture content in transformer oil, breakdown voltage, a different method in moisture reduction and a different method of testing moisture.

Next is testing part, which consists of breakdown voltage test and moisture test where the set-up is found before the testing. The testing is set according to the availability or diversity of equipment in the High Voltage Oil Lab with the reference of the experimental set-up. Lastly, data analyses of the testing result are recorded and analyse to know the characteristic of the oil.

3.2 Flow Chart of overall Procedure

In this part, is about step or process where the oil will undergo. The stages or steps are divided into two part, one is for the waste cooking oil and one is for the commercialize oil. Figure 3.2 below shows testing or flowchart for mineral oil. Which the mineral oil needs to undergo Nitrogen Saturated (NS), Molecular Sieves (MS), Vacuum Oven (VO), before moisture breakdown voltage and breakdown voltage with moisture increment can be done. The baseline for the test is measured before the reduction of moisture treatment.

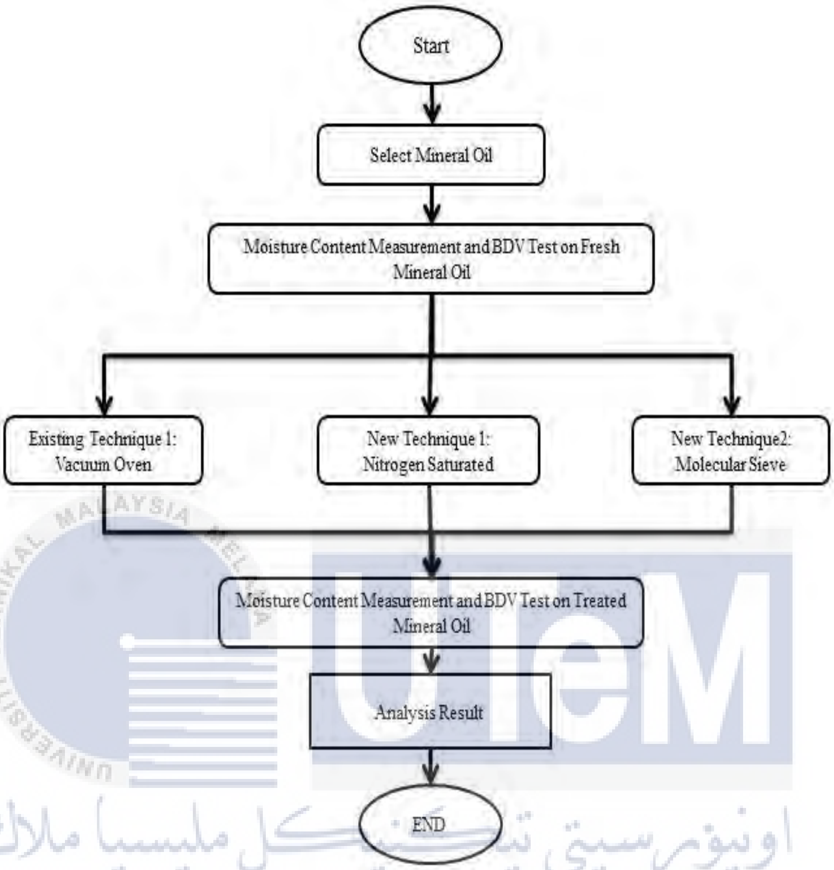

Figure 3.2: Methodology Flow Chart of overall Project

3.3 Mineral Oil Selection

Early in the experiment, transformer oil selection is necessary before proceeding. Selection of transformer oil is according to the availability or diversity of equipment in the High Voltage Oil Lab and agreement with the supervisor. The brand that has been selected is Nytro Libra Mineral Oil. This transformer is being chosen because it properties has a high moisture content and low breakdown voltage. Appendix A shows the properties of Nytro Libra Mineral Oil.

3.4 Breakdown Voltage Test

The breakdown voltage tests are tested using Megger OTS60PB which following the ASTM D1816 standard. This method is for determined the dielectric strength of an insulating liquid by Verband Deutscher Elektrotechniker (VDE) electrodes. According to ASTM D6871, the breakdown voltage for the mineral oil for 1mm gap must be at least 20kV. The sample of mineral oil will be tested for breakdown.

The procedures are followed:

1. 500ml of Nytro Libra Mineral Oil are prepared into oil test vessel.
2. The electrode shape A are set with gap of 1mm following the ASTM D6871.

Figure 3 3: Shape of electrode used.

3. The oil temperature, room temperature and humidity are measured before adding into the Megger OTS60PB.

Figure 3.4: Oil temperature and humidity are measured.

4. Then, on the equipment and select the ASTM D1816.

Figure 3.5: Selecting the standard

5. Lastly, run the breakdown voltage test for about 15-17 minutes. After 5 test the will be shown on the screen.

Figure 3.6: Megger OTS60PB

3.5 Moisture Test

For moisture test, Karl Fischer coulometer is used to determine the water content in the oil which following set-up of apparatus and procedure of ASTM D1533. From the standard, the water content in oil sample must be below the standard value which from standard stated that mineral oil must be below 35ppm.

Figure 3.7: Coulometer Karl Fischer Titration

The procedures are followed:

1. Type of oil is selected and being filled in 6ml syringe tube
2. Air bubble are removed from the syringe by slowly pressing the syringe upwards

Figure 3.8: Syringes fill with oil sample

3. The syringe is then being weighted with an digital analytical balance, mass of oil sample was recorded.

Figure 3.9: A digital analytical balance

4. 1ml of oil sample was filled in the Coulometer Karl Fischer Titration for test one.

5. Press „Start" button to begin the titration located on below the screen of the Coulometer Karl Fischer Titration.
6. Process was repeated for three or four times.

3.6 Techniques of Moisture Removal with BDV and Moisture content measurement.

The study only consist three techniques to remove moisture in Nytro Libra Mineral Oil and all three techniques will be compared with moisture BDV. The three techniques to remove moisture will be discussing more detail below.

3.6.1 Vacuum Oven Technique

The previous work is from "Dielectric Strength and Thermal Conductivity of Mineral Oil based Nanofluids" written by H.F. Jin[16]. The procedure is referred for this study.

1. 500ml Nytro Libra Mineral Oil are filled into a 500ml beaker and placed inside the vacuum oven (VO) shown in figure 3.10 below

Figure 3.10: Placement of Sample in the Vacuum Oven (VO)

2. The Vacuum Oven is set with temperature 60ºC show in figure 3.11 below

Figure 3.11: Vacuum Oven temperature setting

3. After temperature is set, the Vacuum Oven is set with timer for 24 hour equivalent to 1440 minute show in figure 3.12 below

Figure 3.12: Time setting for the Vacuum Oven (VO)

4. After 24 hour in the oven, the oil is then cool down for 24 hour and being wrapped with a transparent wrapper.
5. After 24 hour cooling moisture and BDV test are run on the sample
6. Data is record for a VO technique sample

3.6.2 Nitrogen Saturated Technique

The previous work is from "An Environmentally Friendly Dissolved Oxygen and Moisture Removal System for Freely Breathing Transformer" written by J. Sabau et al [15]. The procedure is referred for this study. Modifications or improvement made for this technique are:

a) Time is cut short from 24 hours to 30 minutes.
b) From a flow rate nitrogen gas on oil to saturated nitrogen gas
c) Heat at 70ºC is being eliminated due to a research said temperature increment will increased amount of moisture in the oil [18].
d) A vacuum is added eliminated moisture circular ling in the beaker.

1. Nitrogen Saturated (NS) as shown in figure 3.13. A 500ml weight of fresh oil is fill into the flask.

Figure 3.13: Apparatus Setup for Nitrogen Saturated (NS)

2. The nitrogen gas is pump from a nitrogen tank shown in figure 3.14 below and the oil will saturate like in the figure above.

Figure 3.14: Nitrogen Gas Tank

3. The gas will go through the oil for 30 minute and exit with help of the vacuum. The vacuum will help to speed the process and make sure moisture is not trapped in the flask. The direction of the gas is shown in figure 3.13 with the indicator of the red arrow.
4. After 30 minute moisture and BDV test are run on the sample
5. Data is record for a NS technique sample

3.6.3 Molecular Sieves Technique

The previous work is from "Physicochemical and electrical properties of Jatropha Curcas methyl ester as a substitute for mineral oil" written by H.B.H Sitorus [17]. The procedure is referred for this study. Modifications or improvement made for this technique are:

a) After sample being mix with molecular sieve the sample is being filter with filter paper as to make sure the BDV of the oil doesn"t not being affected by the particle of the molecular sieve.

b) The filter is divided into two parts, one with large pore 1µm and one with small pore 0.22 µm.

1. Molecular Sieves (MS) as shown in figure 3.15. A weight of 2.2% molecular sieve is being weighted from 1L mineral oil which equivalent to 18.63g before added into the 2000ml beaker for mixing.

Figure 3.15: Molecular sieves use in the previous work [17].

2. After the molecular sieve being weighted, it is added with the mineral oil. Then the sample is being placed on a magnetic stirrer.
3. The sample is being mix for one hour in room temperature with speed of stir 500rpm show in figure 3.16 below.

Figure 3.16: Sample mixing for one hour in room temperature

4. After an hour, the sample is still at the magnetic stirrer but with temperature increased to 70ºC and being stir for 500rpm for another one hour.
5. After two hours of stirring, the sample is being filter. The setup is being shown in figure 3.17 below.

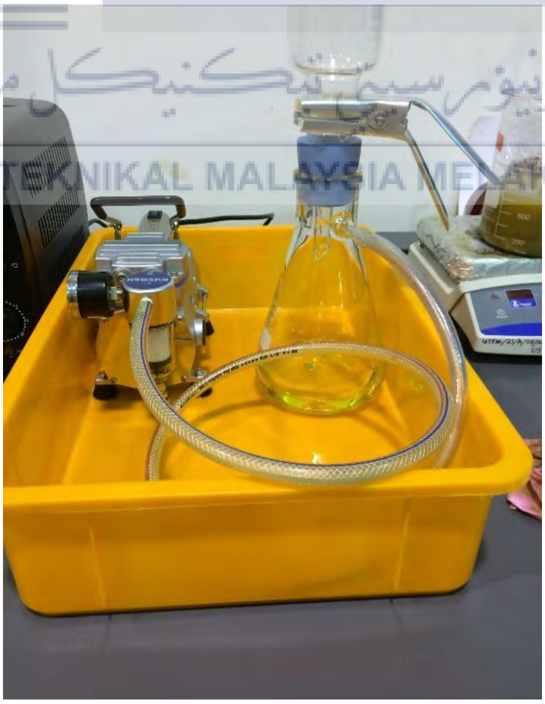

Figure 3.17: Molecular sieves filtration setup

6. The oil sample is being added at the top and vacuum is on to start the filtration.

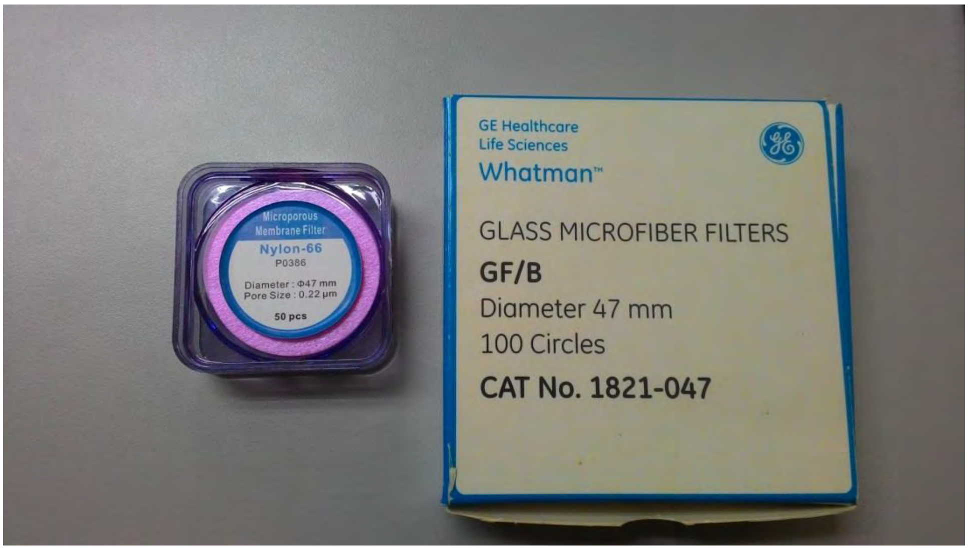

Figure 3.18: Type of filter paper use in the Molecular Sieves Process (MS)

7. After filtration is done, moisture and BDV test are run on the sample
8. Data is record for a MS technique sample
9. Steps are repeated but during filtration filter paper is change.

3.7 Data Analysis

The effect of three techniques on mineral oil in their moisture and breakdown voltage data will be analysed to determine which method have the best effect. The objective of this research should be related to the analysis of the experiment. A conclusion is made after all data have being process.

3.8 Summary

The chapter three is to determine the method used in this project based on research background. The selected techniques, oil, standards for the tools and testing have been discussed in chapter two. The flow chart is made to make other readers to under the flow of this project. The next chapter will discuss detail on the result and data recorded.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

For this chapter, based on the experiment that has been done the recorded data were analysed. To fulfil the objective, the samples for the BDV and moisture test have been carefully prepared and vary with the standard. For easy understanding and comparing all recorded data are presented in graph and table. All the detail and analysis of the data will be discussed as follow.

4.2 Analysis and Project Achievement.

All the recorded data are based on the laboratory experiment. The main things to take note are the achievement of moisture reduction of the mineral oil after undergoes the three techniques state in chapter 3. The first thing to take into account, the amount of moisture content reduction in the mineral oil from before undergoes the three techniques and after. Secondly, study the effect of the molecular sieve on the Nytro Libra Mineral Oil in term of the moisture reduction content. Finally, the BDV increment when processes of moisture reduction have been done. The content below shows the analysis of the experimental result obtains from the laboratory.

4.2.1 Measurement Moisture Content

The baseline of the transformer oil will be measured with the Nytro Libra Mineral Oil fresh obtain from the barrel. All the moisture data will be recorded and compared in Table 4.1 below for the baseline oil and oil after undergoing the treatment. The baseline that obtains directly from the barrel will be measured by the BDV and moisture content without treating it. Meanwhile, the vacuum ovens (VO) will undergo a condensation and heating process for 24 hours. For Nitrogen Saturated (NS), this technique unique due the process only needed to be injected with nitrogen for 30 minutes. For the final method is the Molecular Sieves (MS). This technique is a merging technique of molecular sieves and filtration. Where the mineral oil, need to be treated with the molecular sieves for 2 hours with different temperature for each hour. After that, the oil being filter with two different filter papers where filtration is quite time consuming.

Table 4.1: Data of moisture content for mineral oil.

Techniques	**Moisture Content (ppm)**					
	Test 1	**Test 2**	**Test 3**	**Test 4**	**Test 5**	**Average**
Fresh Oil (FO)	46.3	49.4	31.1	51.1	41.8	43.94
Vacuum Oven (VO)	22.8	19.0	24.7	29.2	31.1	25.36
Nitrogen Saturated (NS)	8.2	7.0	7.0	9.0	11.0	8.44
Molecular Sieves (MS) no filter	4.0	8.5	9.6	10.9	5.8	7.76
Molecular Sieves (MS) Bigger pore size(1µm)	31.8	28.0	23.0	24.6	27.1	26.9
Molecular Sieves (MS) Smaller pore size (0.22µm)	12.0	11.6	10.4	10.3	15.3	11.92

The Table 4.1 above shows the moisture content for each technique that is recorded during the experiment. Five reading or test being conduct is because of reduced uncertainty and get an accurate result. From the table above, each test shown have an uneven result obtain. So to confirm the result is by doing average from five tests for each sample.

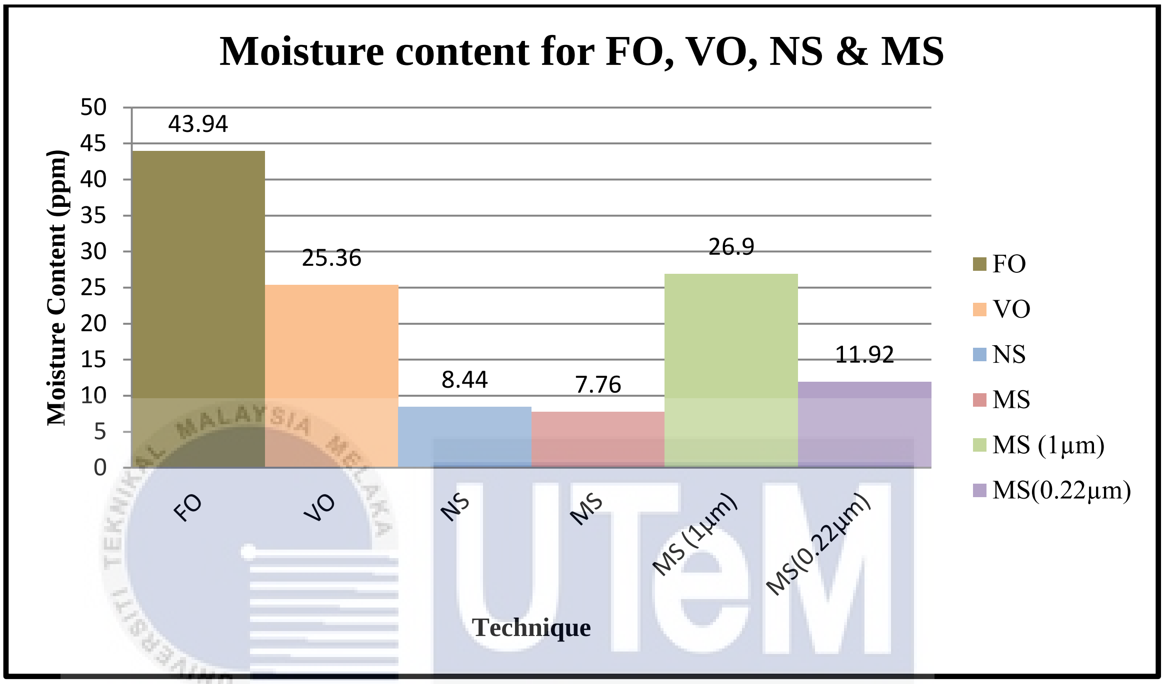

Figure 4.1: Moisture Content for FO, VO, NS and MS techniques

From the Figure 4.1 above, a comparison can be made between the fresh mineral oil and all the sample of moisture removal technique. It is noticeable that the fresh mineral has the highest moisture content in the oil itself compare with other oil after removal technique. The reason behind this high moisture content is maybe due to storage of the oil at a cold cement floor. A good oil storage method of the oil barrel is the floor must be cover with a wood or a rubber material or anything that poor in conduct heat or cold. The cold floor may make the hot air inside the barrel to condense and the oil with mix with the water due to these phenomena. Another factor is, maybe due to the oil has being kept inside the barrel for a long period. Sometimes it can be leakage or small hole at the surface or cap of the tank that leads to the reaction of the mineral to gas from outside

First to being compared is the vacuum oven (VO) and fresh oil. The moisture content difference is about 18.58 equal to 42.28% of moisture reduction shown in Table 2.4. The process of VO is a combination of using heat and vacuum to remove moisture from the mineral oil. The mineral oil was set to be heated to 60ºC during the process of heating. After heating, the mineral oil needed to be cooled down to room temperature (around 20ºC - 30ºC) before a test can run. The cooling process of the mineral oil is the treated mineral oil will be kept in a dark coloured bottle with an inner cap and into a cabinet away from sunlight. The environment inside the cabinet is not vacuumed which mean the chances of the oil to be exposed to the surrounding air is still possible. Hence, there still a possibility of the moisture content of the oil increase during cooling process as reaction can occur between oil and atmosphere such as humidity, temperature and condition of the High Voltage Oil Laboratory lead to the increment of moisture in the oil.

Second is the comparison of fresh oil and nitrogen saturated (NS). As Table 4.2 below shows the moisture content of the mineral oil after being treated with NS techniques. The drop of moisture from 43.94 ppm to 8.44 ppm is quite a lot. The difference of the moisture content reduced is 35.5ppm equal to 80.79%. The second highest in moisture remove among all techniques in Table 4.2 which out of the blue the fastest technique to reduced moisture with the time taken is about 30 minutes. The moisture reduction is also due to after test the oil is directly tested to determine the moisture content. The NS technique does not require a cooling process due no heating is involved during moisture reduction process. Thus, the probability for NS to react with the gas at surrounding is small and the mineral oil temperature is maintained at room temperature. In addition, according to the finding of J. Sabau the dissolving gas analysis (DGA) test that when nitrogen gas flow through the mineral oil, the gas properties of nitrogen dehydrated the oxygen, carbon dioxide and ethylene as the nitrogen gas act as a moisture absorbent like a silica gel. From this recorded data, a theoretical assumption on which the dissolved oxygen in the oil is absorbed by nitrogen is proven[15].

Finally, for the molecular sieve (MS), the reason MS is shown three times is to show the differences of MS with a combination of different filter paper and without the filter. Comparison of three MS technique is MS alone without filter show the most moisture content remove followed by MS Smaller pore size (0.22μm) and then MS Bigger pore size (1µm). As the below show, molecular sieves have reduced the moisture content from 43.94 ppm to 7.76 ppm which is 80.79% reduction which means the molecular sieve have no problem to reduce the moisture content in the mineral oil. This moisture content drop is caused by the structure of the molecular sieve where the structure allows the water to be crystallization to be removed, leaving a porous crystalline structure. Due to the presence sodium, calcium, potassium and by the absolutely enormous internal surface area of close to 1000m^2/g, with the aid of strong ionic forces the molecular sieves will absorb a considerable amount of water[19]–[22]. It is said that the molecular sieve can reduce many liquid impurities to very low level[22]. The reason behind the increase of moisture during filtration with the filter paper is the process took a long time to process and some of the moisture is hidden behind or inside the temporary good oil.

Furthermore, Table 4.2 have shown that NS, MS, MS pore size (0.22µm), MS Bigger pore size (1µm) and VO has achieved ASTM D3487 standard for a good mineral oil which is moisture content must below 35ppm. The calculation is based on (4.1).

$$\text{Percentage Reduced (\%)} = \frac{(Baseline - moisture\ content)}{Baseline} \times 100 \tag{4.1}$$

Table 4.2: Moisture content reduction of mineral oil.

Techniques	Moisture Content (ppm)	Percentage Reduced%
Fresh Oil (FO)	43.94 (Baseline)	0.00
Vacuum Oven (VO)	25.36	42.28
Nitrogen Saturated (NS)	8.44	80.79
Molecular Sieves (MS) no filter	7.76	82.33
Molecular Sieves (MS) Bigger pore size(1µm)	26.9	38.78
Molecular Sieves (MS) Smaller pore size (0.22µm)	11.92	72.87

4.2.2 Breakdown Voltage Test

The breakdown voltage (BDV) for fresh oil and moisture removal technique oil are plotted in Figure 4.2. The Figure 4.2 below shows the total average of 50 reading from the Megger OTS60PB for each technique. Recorded data will be shown in Appendix E.

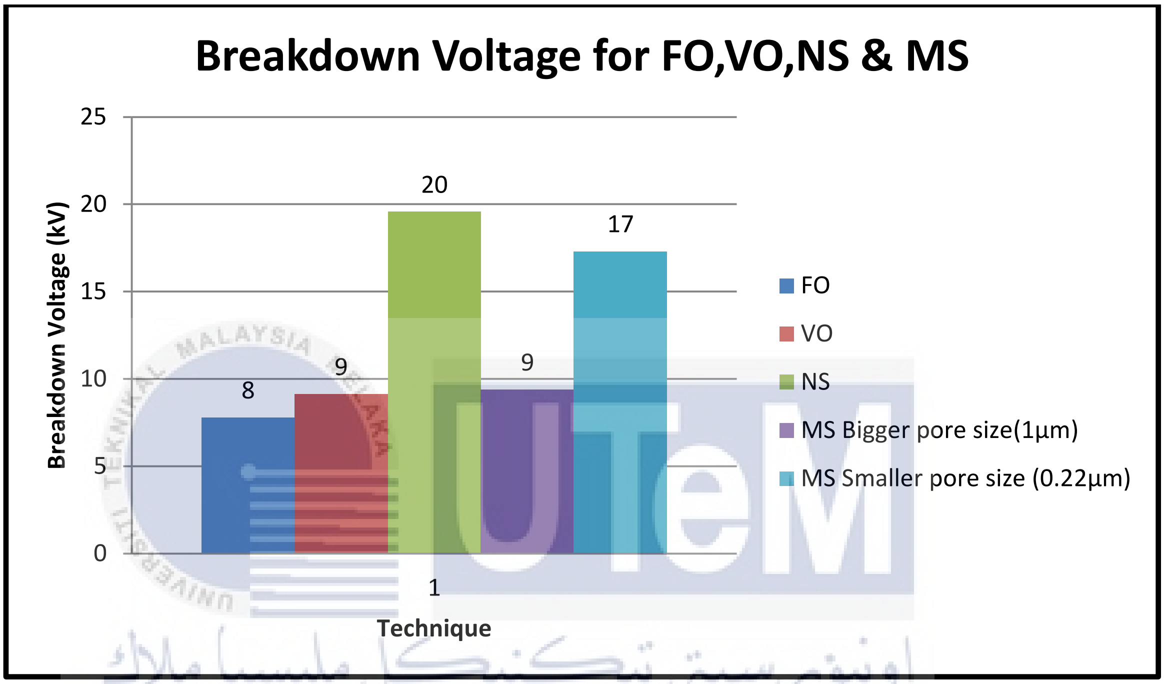

Figure 4.2: Breakdown Voltage of FO, VO, NS & MS technique

Based on Figure 4.2 above, the FO is compared with each other moisture removal techniques. As the figure above shows, there are improvements in each moisture removal technique for the mineral oil and all the result is higher than FO itself. The reason for the increase of BDV is the reduction of moisture content in the oil as stated in Table 4.3.

From the Table 4.3, the breakdown voltage of the Fresh Oil (FO) is 8kV which the lowest and not following the specification of a standard mineral oil in ASTM D3487. But comparing FO and VO technique the BDV different is 1kV and equal to 12.5% enhancement. VO BDV is slightly higher due to low moisture content compare to FO. The also same goes for Molecular Sieves (MS) Bigger pore size (1µm) which have similar BDV of 9kV. The calculation is based on (4.2).

$$\text{Enhancement (\%)} = \frac{(Breakdown\ voltage - Baseline)}{Baseline} \times 100 \qquad (4.2)$$

Table 4.3: Mineral Oil breakdown voltage with enhancement

Samples	BDV (kV)	Enhancement (%)
Fresh Oil (FO)	8 (Baseline)	0.00
Vacuum Oven (VO)	9	12.5
Nitrogen Saturated (NS)	20	150
Molecular Sieves (MS) Bigger pore size(1µm)	9	12.5
Molecular Sieves (MS) Smaller pore size (0.22µm)	17	112.5

Move to the next technique which is Nitrogen Saturated (NS). NS has the highest BDV which satisfied the BDV standard of transformer oil in ASTM D1816 for 1mm gap the BDV is minimum 20kV. The reason is due fact that the moisture content of the mineral oil itself after treatment is 8.44 (ppm). The BDV difference FO and NS is 12kV which is equivalent to 150% enhancement as stated in Table 4.3.

For the Molecular Sieves (MS) Smaller pore size (0.22µm), the BDV is the second highest show in Table 4.3 and Table 4.4. The reason BDV of this technique is higher due to moisture content has being reduced to half compared to Molecular Sieves (MS) Bigger pore size(1µm) shown in Table 4.4. The BDV differences of Molecular Sieves (MS) smaller pore size (0.22µm) with FO is 9kV with enhancement of 112.5% shown in table 4.3.

Table 4.4: Relationship between breakdown voltage and moisture content of the mineral oil

Samples	Moisture Content (ppm)	BDV (kV)
Fresh Oil (FO)	43.94	8
Vacuum Oven (VO)	25.36	9
Nitrogen Saturated (NS)	8.44	20
Molecular Sieves (MS) Bigger	26.9	9

pore size(1µm)		
Molecular Sieves (MS) Smaller pore size (0.22µm)	11.92	17

To conclude this analysis, moisture content and breakdown voltage should be taken into consideration between these two parameter relationships. The reason why no filter Molecular Sieves (MS) is not tested with BDV is due to the oil must not have any other impurities. If there‟s existence of other impurities it will affect the BDV of the mineral oil due low breakdown voltage maybe causes the impurities of contamination[3], [6], [9]. Table 4.4 above shows the summary of data extracted from the experiment. From Figure 4.3 below, clearly see that when the moisture content in the mineral oil is high. From Figure 4.4, the BDV of the mineral oil will also in the lowest position and a vice versa situation can be applied. Hence, the relationship of lowest moisture content will increase BDV of the insulating oil. This experimental has proven a theory about moisture content inside insulating oil will affect the performance of insulating oil.

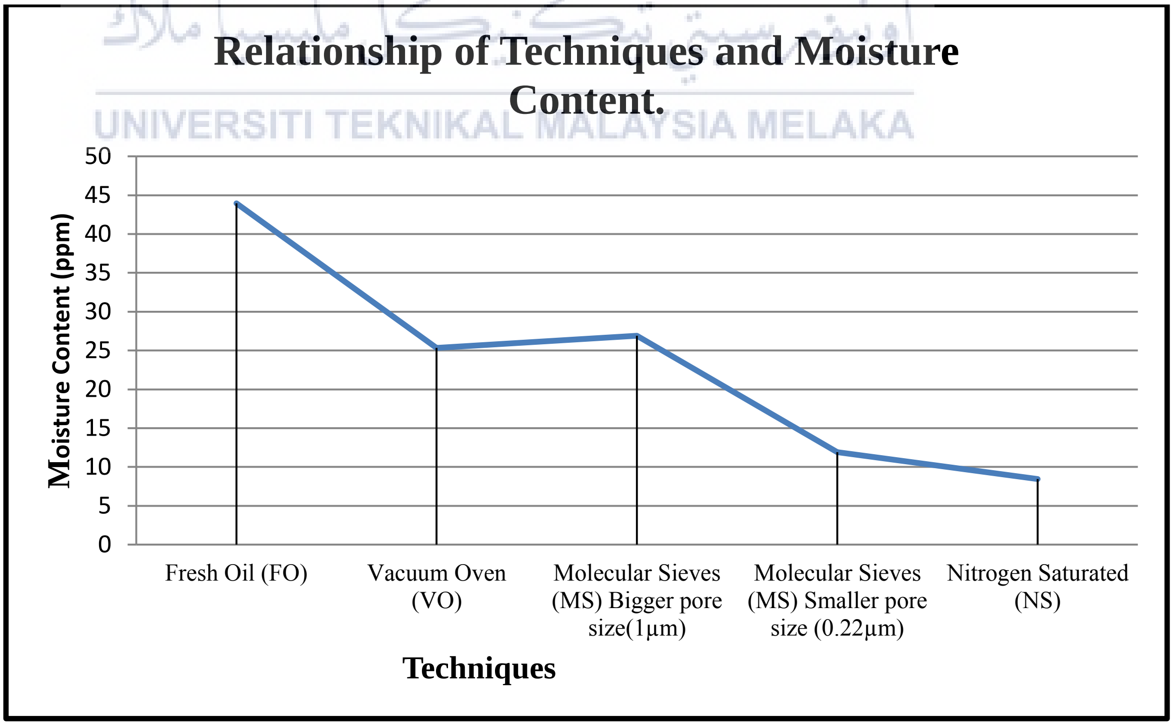

Figure 4.3: Analysis of moisture content in mineral oil.

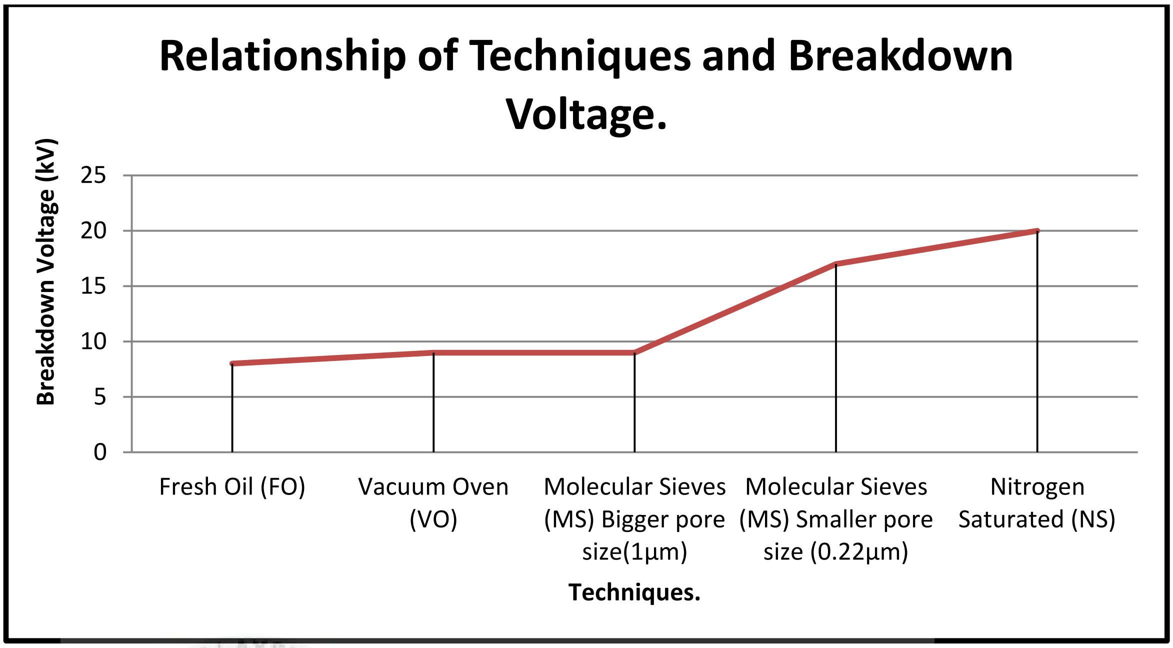

Figure 4. 4: The breakdown voltage of mineral oil after treatment.

4.3 Summary

In this chapter contained all the result and discussion of the experiment. The topic discussed breakdown voltage and moisture content of mineral oil before and after moisture removal technique had been done. Chapter 5 will be discussing the conclusion of the experiment and recommendation of this study.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The analysis of this experiment to establish the moisture removal techniques that is possible to treat mineral- based insulating oil by reducing oil moisture content. During the study, the technique is compared with one and another. The parameter used to compare is the moisture content and the BDV. Then after comparison, each technique is analysed and determine which technique the most effective is for reducing the moisture content of mineral-based insulating oil. There are three technique which is Vacuum oven (VO) Nitrogen Saturated (NS) and Molecular Sieves (MS) but MS is separated into two parts, one of it is Molecular Sieves (MS) Bigger pore size (1µm) and the other part is Molecular Sieves (MS) smaller pore size (0.22µm). The reason behind MS is separated into two parts is because the molecular sieve particles need to be removed before BDV test. If the molecular sieve is not removed it will affect the BDV of the oil even after treatment.

Each technique has step and procedure which will result in a different outcome for each test. All the procedures of test equipment have been conducted according to standard available. Hence, data of the experiment gain was expected and reasonable comply with the standard. Meanwhile, moisture is tested using Karl Fischer Coulometric with standard ASTM D1533[23]. The Megger OTS60PB was used to test the breakdown voltage with the guidance of standard ASTM D1816[24].

In term of moisture reduction, Nitrogen Saturated (NS) and Molecular Sieves (MS) was the most effective technique. But in term of breakdown voltage the Nitrogen Saturated (NS), was the one and only reached 20kV the minimum BDV standard for a 1mm gap. This can be proven by the data obtained and this also indicates an innovative, environmentally friendly and affordable technique can reduce the oxidation decay process of insulation oil when the moisture content is also reduced.

5.2 Recommendations

During the investigation, the data gain and shown that moisture content does play role in affecting the breakdown voltage of the mineral oil. Suggestion on improving and future plans related to this project:

1. Study other methods on removing molecular sieve particle without increasing moisture content.
2. Study the moisture removal technique using molecular sieve and nitrogen with the variance of time.
3. Analyse the properties of mineral-based insulating oil using FTIR for nitrogen and molecular sieve removal technique.

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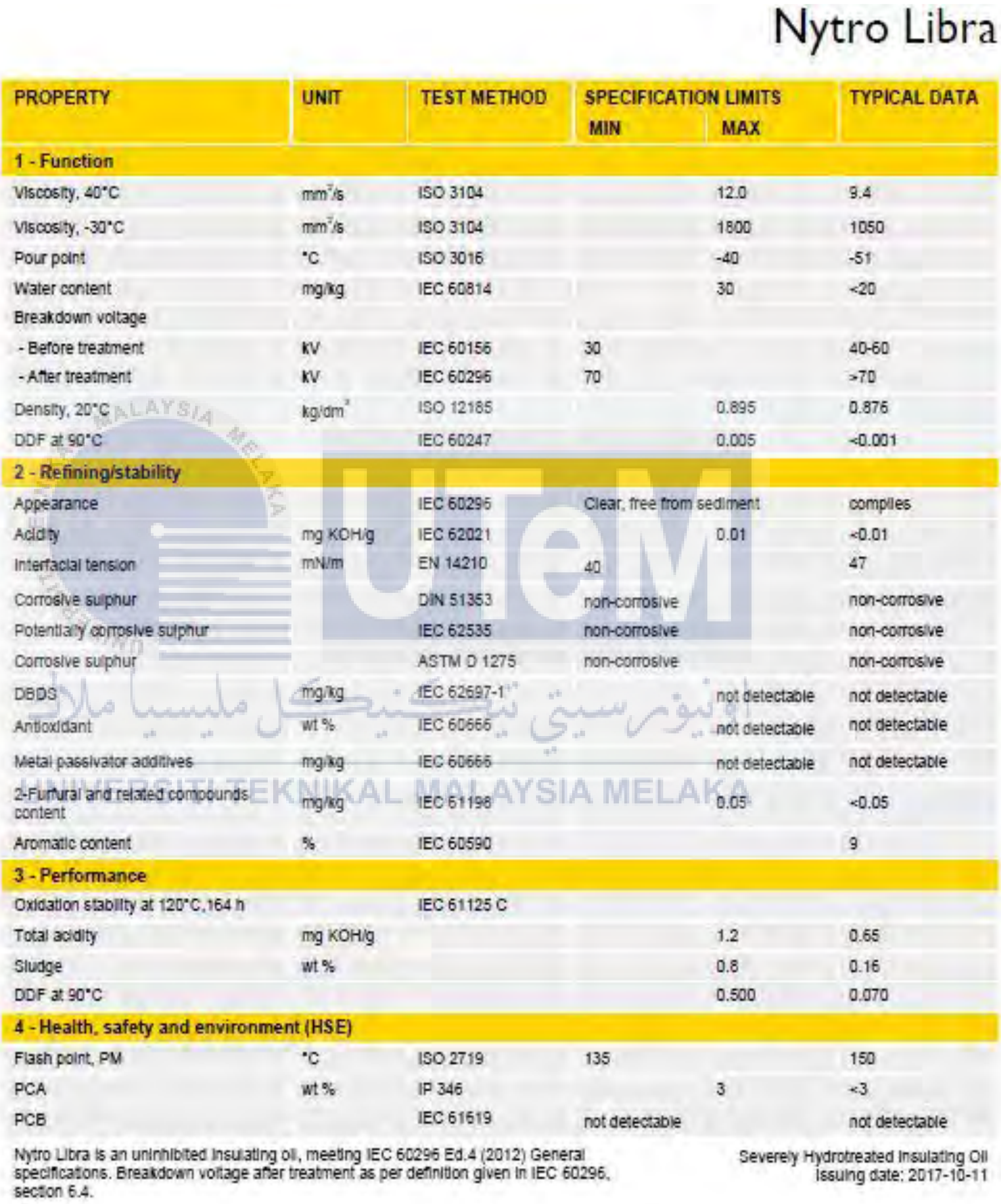
APPENDICES

Appendix A: NYTRO LIBRA MINERAL OIL DATASHEET



Nytro Libra

PROPERTY	UNIT	TEST METHOD	SPECIFICATION LIMITS		TYPICAL DATA
			MIN	MAX	
1 - Function					
Viscosity, 40°C	mm²/s	ISO 3104		12.0	9.4
Viscosity, -30°C	mm²/s	ISO 3104		1800	1050
Pour point	°C	ISO 3016		-40	-51
Water content	mg/kg	IEC 60814		30	<20
Breakdown voltage					
- Before treatment	kV	IEC 60156	30		40-60
- After treatment	kV	IEC 60296	70		>70
Density, 20°C	kg/dm³	ISO 12185		0.895	0.876
DDF at 90°C		IEC 60247		0.005	<0.001
2 - Refining/stability					
Appearance		IEC 60296	Clear, free from sediment		complies
Acidity	mg KOH/g	IEC 62021		0.01	<0.01
Interfacial tension	mN/m	EN 14210	40		47
Corrosive sulphur		DIN 51353	non-corrosive		non-corrosive
Potentially corrosive sulphur		IEC 62535	non-corrosive		non-corrosive
Corrosive sulphur		ASTM D 1275	non-corrosive		non-corrosive
DBDS	mg/kg	IEC 62697-1		not detectable	not detectable
Antioxidant	wt %	IEC 60666		not detectable	not detectable
Metal passivator additives	mg/kg	IEC 60666		not detectable	not detectable
2-Furfural and related compounds content	mg/kg	IEC 61198		0.05	<0.05
Aromatic content	%	IEC 60590			9
3 - Performance					
Oxidation stability at 120°C,164 h		IEC 61125 C			
Total acidity	mg KOH/g			1.2	0.65
Sludge	wt %			0.8	0.16
DDF at 90°C				0.500	0.070
4 - Health, safety and environment (HSE)					
Flash point, PM	°C	ISO 2719	135		150
PCA	wt %	IP 346		3	<3
PCB		IEC 61619	not detectable		not detectable

Nytro Libra is an uninhibited insulating oil, meeting IEC 60296 Ed.4 (2012) General specifications. Breakdown voltage after treatment as per definition given in IEC 60296, section 6.4.

Severely Hydrotreated Insulating Oil
Issuing date: 2017-10-11

Appendix B: ASTM D3487

Designation: D3487 − 16

Standard Specification for Mineral Insulating Oil Used in Electrical Apparatus[1]

This standard is issued under the fixed designation D3487; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This specification covers unused mineral insulating oil of petroleum origin for use as an insulating and cooling medium in new and existing power and distribution electrical apparatus, such as transformers, regulators, reactors, circuit breakers, switchgear, and attendant equipment.

1.2 This specification is intended to define a mineral insulating oil that is functionally interchangeable and miscible with existing oils, is compatible with existing apparatus and with appropriate field maintenance,[2] and will satisfactorily maintain its functional characteristics in its application in electrical equipment. This specification applies only to new insulating oil as received prior to any processing.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
D97 Test Method for Pour Point of Petroleum Products
D117 Guide for Sampling, Test Methods, and Specifications for Electrical Insulating Oils of Petroleum Origin
D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents
D923 Practices for Sampling Electrical Insulating Liquids
D924 Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids
D971 Test Method for Interfacial Tension of Oil Against Water by the Ring Method
D974 Test Method for Acid and Base Number by Color-Indicator Titration
D1275 Test Method for Corrosive Sulfur in Electrical Insulating Liquids
D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
D1524 Test Method for Visual Examination of Used Electrical Insulating Liquids in the Field
D1533 Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration
D1816 Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using VDE Electrodes
D1903 Practice for Determining the Coefficient of Thermal Expansion of Electrical Insulating Liquids of Petroleum Origin, and Askarels
D2112 Test Method for Oxidation Stability of Inhibited Mineral Insulating Oil by Pressure Vessel
D2300 Test Method for Gassing of Electrical Insulating Liquids Under Electrical Stress and Ionization (Modified Pirelli Method)
D2440 Test Method for Oxidation Stability of Mineral Insulating Oil
D2668 Test Method for 2,6-*di-tert*-Butyl-*p*-Cresol and 2,6-*di-tert*-Butyl Phenol in Electrical Insulating Oil by Infrared Absorption
D2717 Test Method for Thermal Conductivity of Liquids
D2766 Test Method for Specific Heat of Liquids and Solids
D2864 Terminology Relating to Electrical Insulating Liquids and Gases
D3300 Test Method for Dielectric Breakdown Voltage of Insulating Oils of Petroleum Origin Under Impulse Conditions
D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

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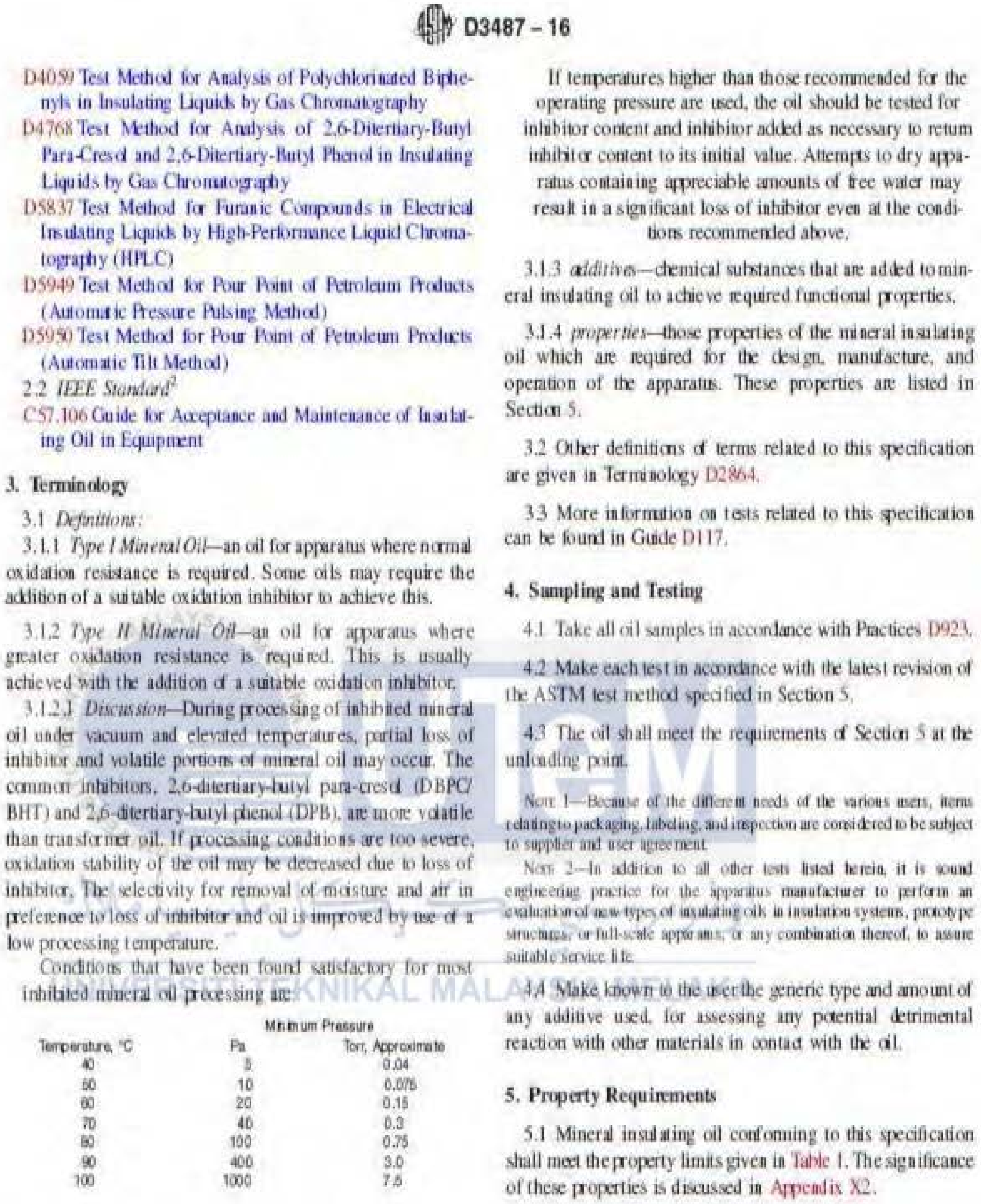
[1] This specification is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.01 on Mineral.

Current edition approved June 15, 2016. Published July 2016. Originally approved in 1976. Last previous edition approved in 2009 as D3487 – 09. DOI:10.1520/D3487-16.

[2] Refer to the Institute of Electrical and Electronic Engineers, Inc. (IEEE) C 57.106, Guide for Acceptance and Maintenance of Insulating Oil in Equipment. Available from IEEE Operations Center, 445 Hoes Lane, Piscataway, NJ 08854-4141, USA.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



D4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography

D4768 Test Method for Analysis of 2,6-Ditertiary-Butyl Para-Cresol and 2,6-Ditertiary-Butyl Phenol in Insulating Liquids by Gas Chromatography

D5837 Test Method for Furanic Compounds in Electrical Insulating Liquids by High-Performance Liquid Chromatography (HPLC)

D5949 Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)

D5950 Test Method for Pour Point of Petroleum Products (Automatic Tilt Method)

2.2 *IEEE Standard*[2]

C57.106 Guide for Acceptance and Maintenance of Insulating Oil in Equipment

3. Terminology

3.1 *Definitions:*

3.1.1 *Type I Mineral Oil*—an oil for apparatus where normal oxidation resistance is required. Some oils may require the addition of a suitable oxidation inhibitor to achieve this.

3.1.2 *Type II Mineral Oil*—an oil for apparatus where greater oxidation resistance is required. This is usually achieved with the addition of a suitable oxidation inhibitor.

3.1.2.1 *Discussion*—During processing of inhibited mineral oil under vacuum and elevated temperatures, partial loss of inhibitor and volatile portions of mineral oil may occur. The common inhibitors, 2,6-ditertiary-butyl para-cresol (DBPC/BHT) and 2,6-ditertiary-butyl phenol (DPB), are more volatile than transformer oil. If processing conditions are too severe, oxidation stability of the oil may be decreased due to loss of inhibitor. The selectivity for removal of moisture and air in preference to loss of inhibitor and oil is improved by use of a low processing temperature.

Conditions that have been found satisfactory for most inhibited mineral oil processing are:

	Minimum Pressure	
Temperature, °C	Pa	Torr, Approximate
40	5	0.04
50	10	0.075
60	20	0.15
70	40	0.3
80	100	0.75
90	400	3.0
100	1000	7.5

If temperatures higher than those recommended for the operating pressure are used, the oil should be tested for inhibitor content and inhibitor added as necessary to return inhibitor content to its initial value. Attempts to dry apparatus containing appreciable amounts of free water may result in a significant loss of inhibitor even at the conditions recommended above.

3.1.3 *additives*—chemical substances that are added to mineral insulating oil to achieve required functional properties.

3.1.4 *properties*—those properties of the mineral insulating oil which are required for the design, manufacture, and operation of the apparatus. These properties are listed in Section 5.

3.2 Other definitions of terms related to this specification are given in Terminology D2864.

3.3 More information on tests related to this specification can be found in Guide D117.

4. Sampling and Testing

4.1 Take all oil samples in accordance with Practices D923.

4.2 Make each test in accordance with the latest revision of the ASTM test method specified in Section 5.

4.3 The oil shall meet the requirements of Section 5 at the unloading point.

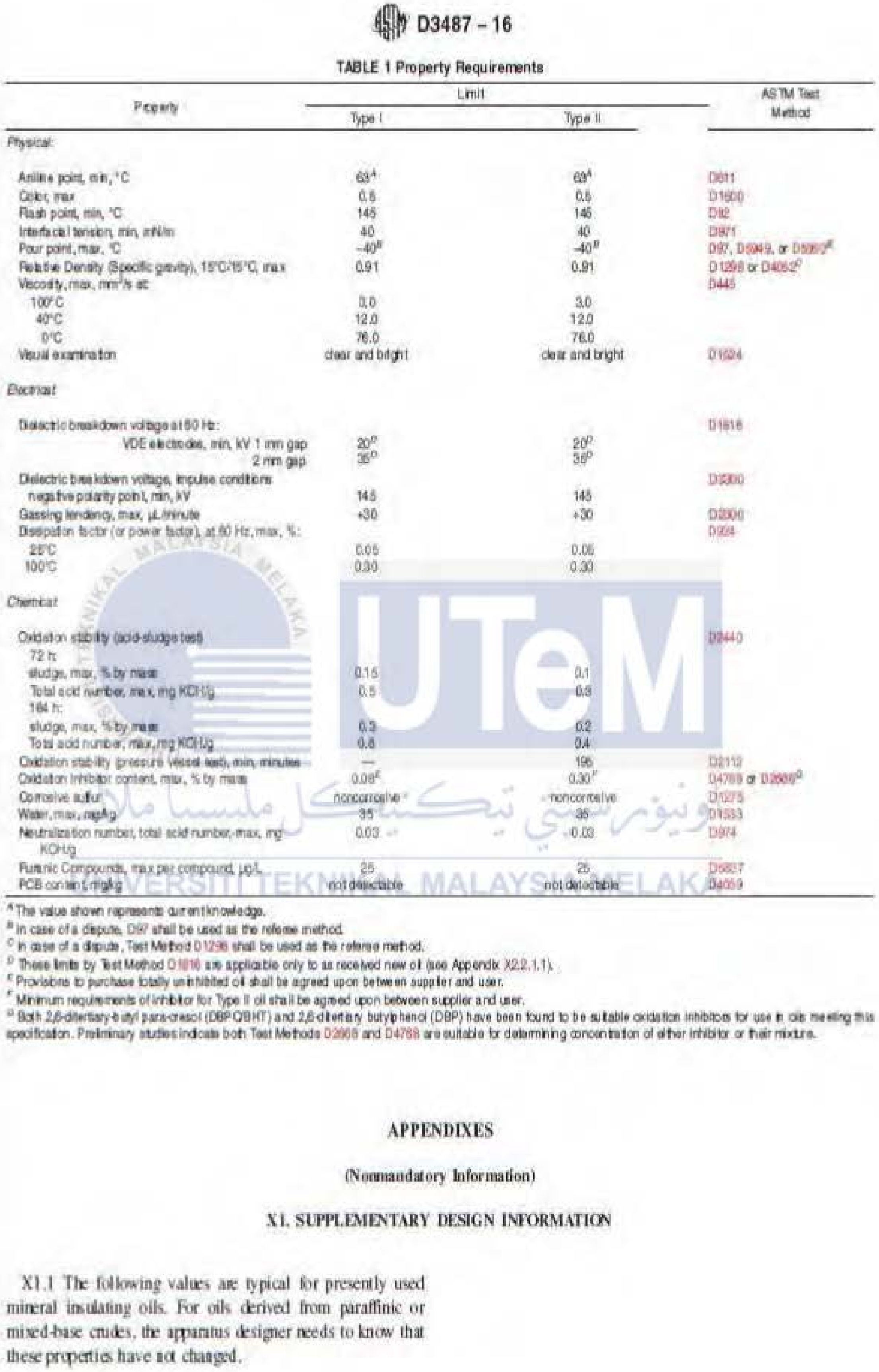
NOTE 1—Because of the different needs of the various users, items relating to packaging, labeling, and inspection are considered to be subject to supplier and user agreement.

NOTE 2—In addition to all other tests listed herein, it is sound engineering practice for the apparatus manufacturer to perform an evaluation of new types of insulating oils in insulation systems, prototype structures, or full-scale apparatus, or any combination thereof, to assure suitable service life.

4.4 Make known to the user the generic type and amount of any additive used, for assessing any potential detrimental reaction with other materials in contact with the oil.

5. Property Requirements

5.1 Mineral insulating oil conforming to this specification shall meet the property limits given in Table 1. The significance of these properties is discussed in Appendix X2.



D3487 – 16

TABLE 1 Property Requirements

Property	Limit		ASTM Test Method
	Type I	Type II	
Physical:			
Aniline point, min, °C	63[A]	63[A]	D611
Color, max	0.5	0.5	D1500
Flash point, min, °C	145	145	D92
Interfacial tension, min, mN/m	40	40	D971
Pour point, max, °C	−40[B]	−40[B]	D97, D5949, or D5950[B]
Relative Density (Specific gravity), 15°C/15°C, max	0.91	0.91	D1298 or D4052[C]
Viscosity, max, mm²/s at:			D445
100°C	3.0	3.0	
40°C	12.0	12.0	
0°C	76.0	76.0	
Visual examination	clear and bright	clear and bright	D1524
Electrical:			
Dielectric breakdown voltage at 60 Hz:			D1816
VDE electrodes, min, kV 1 mm gap	20[D]	20[D]	
2 mm gap	35[D]	35[D]	
Dielectric breakdown voltage, impulse conditions negative polarity point, min, kV	145	145	D3300
Gassing tendency, max, µL/minute	+30	+30	D2300
Dissipation factor (or power factor), at 60 Hz, max, %:			D924
25°C	0.05	0.05	
100°C	0.30	0.30	
Chemical:			
Oxidation stability (acid-sludge test)			D2440
72 h:			
sludge, max, % by mass	0.15	0.1	
Total acid number, max, mg KOH/g	0.5	0.3	
164 h:			
sludge, max, % by mass	0.3	0.2	
Total acid number, max, mg KOH/g	0.6	0.4	
Oxidation stability (pressure vessel test), min, minutes	—	195	D2112
Oxidation inhibitor content, max, % by mass	0.08[E]	0.30[F]	D4768 or D2668[G]
Corrosive sulfur	noncorrosive	noncorrosive	D1275
Water, max, mg/kg	35	35	D1533
Neutralization number, total acid number, max, mg KOH/g	0.03	0.03	D974
Furanic Compounds, max per compound, µg/L	25	25	D5837
PCB content, mg/kg	not detectable	not detectable	D4059

[A] The value shown represents current knowledge.

[B] In case of a dispute, D97 shall be used as the referee method.

[C] In case of a dispute, Test Method D1298 shall be used as the referee method.

[D] These limits by Test Method D1816 are applicable only to as received new oil (see Appendix X2.2.1.1).

[E] Provisions to purchase totally uninhibited oil shall be agreed upon between supplier and user.

[F] Minimum requirements of inhibitor for Type II oil shall be agreed upon between supplier and user.

[G] Both 2,6-ditertiary-butyl para-cresol (DBPC/BHT) and 2,6-ditertiary butylphenol (DBP) have been found to be suitable oxidation inhibitors for use in oils meeting this specification. Preliminary studies indicate both Test Methods D2668 and D4768 are suitable for determining concentration of either inhibitor or their mixture.

APPENDIXES

(Nonmandatory Information)

X1. SUPPLEMENTARY DESIGN INFORMATION

X1.1 The following values are typical for presently used mineral insulating oils. For oils derived from paraffinic or mixed-base crudes, the apparatus designer needs to know that these properties have not changed.

Property	Typical Values	ASTM Test Method
Coefficient of expansion,/°C from 25 to 100°C	0.0007 to 0.0008	D1903
Dielectric constant, 25°C	2.2 to 2.3	D924
Specific heat, J/(kg °C), 20°C	1800	D2766
Thermal conductivity, (m °C), from 25 to 100°C	0.13 to 0.17	D2717

X2. SIGNIFICANCE OF PROPERTIES OF MINERAL INSULATING OIL

X2.1 Physical Properties

X2.1.1 *Aniline Point*—The aniline point of a mineral insulating oil indicates the solvency of the oil for materials that are in contact with the oil. It may relate to the impulse and gassing characteristics of the oil.

X2.1.2 *Color*—A low color number is an essential requirement for inspection of assembled apparatus in the tank. An increase in the color number during service is an indicator of deterioration of the mineral insulating oil.

X2.1.3 *Flash Point*—The safe operation of the apparatus requires an adequately high flash point.

X2.1.4 *Interfacial Tension*—A high value for new mineral insulating oil indicates the absence of undesirable polar contaminants. This test is frequently applied to service-aged oils as an indicator of the degree of deterioration.

X2.1.5 *Pour Point*—The pour point of mineral insulating oil is the lowest temperature at which the oil will just flow and many of the factors cited under viscosity apply. The pour point of −40°C may be obtained by the use of suitable distillates, refining processes, the use of appropriate long life additives, or any combination thereof. If a pour point additive is used, it is necessary to make known the amount and chemical composition.

X2.1.6 *Relative Density (Specific Gravity)*—The specific gravity of a mineral insulating oil influences the heat transfer rates and may be pertinent in determining suitability for use in specific applications. In extremely cold climates, specific gravity has been used to determine whether ice, resulting from freezing of water in oil-filled apparatus, will float on the oil and possibly result in flashover of conductors extending above the oil level. See, for example, "The Significance of the Density of Transformer Oils."[4]

X2.1.7 *Viscosity*—Viscosity influences the heat transfer and, consequently, the temperature rise of apparatus. At low temperatures, the resulting higher viscosity influences the speed of moving parts, such as those in power circuit breakers, switchgear, load tapchanger mechanisms, pumps, and regulators. Viscosity controls mineral insulating oil processing conditions, such as dehydration, degassification and filtration, and oil impregnation rates. High viscosity may adversely affect the starting up of apparatus in cold climates (for example, spare transformers and replacements).

X2.1.8 *Visual Examination*—A simple visual inspection of mineral insulating oil may indicate the absence or presence of undesirable contaminants. If such contaminants are present, more definitive testing is recommended to assess their effect on other functional properties.

X2.2 Electrical Properties

X2.2.1 *Dielectric Breakdown Voltage, 60 Hz*—The dielectric breakdown voltage of a mineral insulating oil indicates its ability to resist electrical breakdown at power frequencies in electrical apparatus.

X2.2.1.1 *Dielectric Breakdown—VDE Electrodes*—The VDE method (Test Method D1816) is sensitive to contaminants, such as water, dissolved gases, cellulose fibers, and conductive particles in oil. Processing involves filtering, dehydration, and degassing, which generally improve the breakdown strength of the oil. As a general guide, the moisture and dissolved gas content by volume in processed oils should be less than 15 ppm and 0.5 % respectively. The minimum breakdown strength for as-received oils is typically lower than that of processed oils because of higher levels of contaminants.

X2.2.2 *Dielectric Breakdown Voltage—Impulse*—The impulse strength of oil is critical in electrical apparatus. The impulse breakdown voltage of oil indicates its ability to resist electrical breakdown under transient voltage stresses (lightning and switching surges). The functional property is sensitive to both polarity and electrode geometry.

X2.2.3 *Dissipation Factor*—Dissipation factor (power factor) is a measure of the dielectric losses in oil. A low dissipation factor indicates low dielectric losses and a low level of soluble contaminants.

X2.2.4 *Gassing*—The gassing tendency of a mineral insulating oil is a measure of the rate of absorption or desorption of hydrogen into or out of the oil under prescribed laboratory conditions. It reflects, but does not measure, aromaticity of the oil.

X2.3 Chemical Properties

X2.3.1 *Oxidation Inhibitor Content*—Oxidation inhibitor added to mineral insulating oil retards the formation of oil sludge and acidity under oxidative conditions. It is important to know if an oxidation inhibitor has been added to the oil and the

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[4] Mulhall, V. R., "The Significance of the Density of Transformer Oils," *IEEE Transactions on Electrical Insulation*, Vol 15, No. 6, December 1980, pp. 498-499. DOI: 10.1520/D3487-09_WIP_#872075.

amount. 2,6-Ditertiary-butyl para-cresol and 2,6-ditertiary butylphenol have been found suitable for use in mineral insulating oils complying with this specification. It is anticipated that other oxidation inhibitors will be accepted.

X2.3.2 *Corrosive Sulfur*—The absence of elemental sulfur and thermally unstable sulfur-bearing compounds is necessary to prevent the corrosion of certain metals such as copper and silver in contact with the mineral insulating oil.

X2.3.3 *Water Content*—A low water content of mineral insulating oil is necessary to achieve adequate electrical strength and low dielectric loss characteristics, to maximize the insulation system life, and to minimize metal corrosion.

X2.3.4 *Neutralization Number*—A low total acid content of a mineral insulating oil is necessary to minimize electrical conduction and metal corrosion and to maximize the life of the insulation system.

X2.3.5 *Oxidation Stability*—The development of oil sludge and acidity resulting from oxidation during storage, processing, and long service life should be held to a minimum. This minimizes electrical conduction and metal corrosion, maximizes insulation system life and electrical breakdown strength, and ensures satisfactory heat transfer. The limiting values in accordance with Table 1, as determined by Test Methods D2112 and D2440, best achieve these objectives.

X2.3.6 Furanic Compounds provide a means to assess the cellulose degradation of an insulation system. The level of these compounds must be at or below the levels stated in Table 1 to ensure the baseline is known for new oil when delivered. Furanic Compounds are typically not found in highly refined oil but might be present due to contamination. The purpose is to ensure future work is not distorted by the presence of these compounds.

X2.3.7 *PCB Content*—Many regulations specify procedures to be followed for the use and disposal of electrical apparatus and electrical insulating fluids containing various PCBs (polychlorinated biphenyls) or aroclors. The procedure to be used for a particular apparatus or lot of insulating fluid is determined from its PCB content. New mineral insulating oil of the type covered by this specification should not contain any detectable PCBs. A non-detectable PCB concentration measured by Test Method D4059 provides documentation to permit the insulating oil and apparatus containing it to be used without the labeling, recordkeeping, and disposal restrictions required of PCB-containing materials.

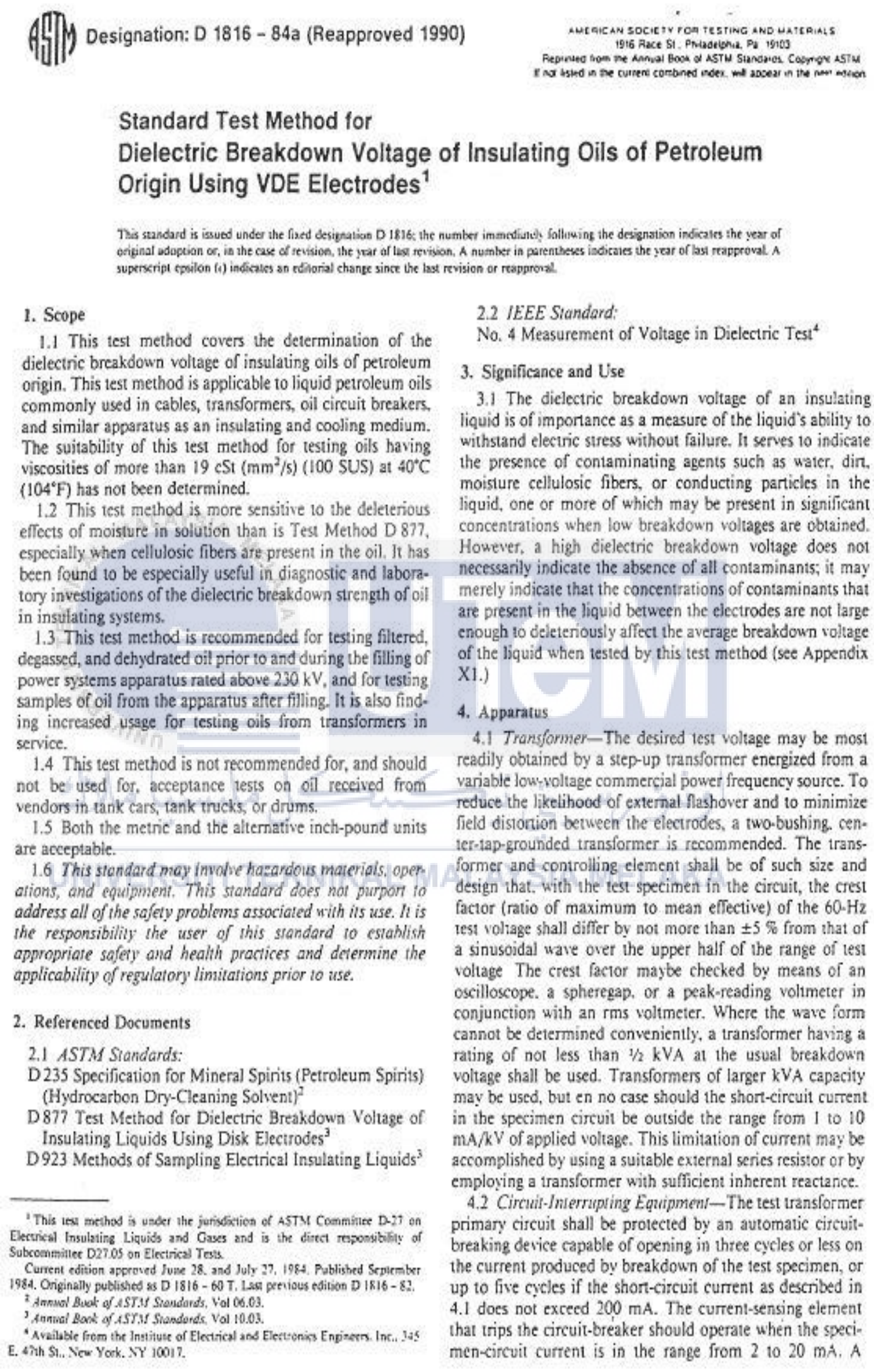
X3. PETROLEUM SOURCES, REFINING PROCESSES, AND SHIPPING CONTAINERS

X3.1 *Petroleum Sources*—Mineral insulating oils are presently refined from predominantly naphthenic crude oils. Paraffinic crudes and new refining technology may be used to provide mineral insulating oil for use in electrical apparatus. As new petroleum sources are developed for this use, additional tests peculiar to the chemistry of these oils may need to be defined.

X3.2 *Refining Processes*—Distillates from crude oils may be refined by various processes such as solvent extraction, dewaxing, hydrotreating, hydrocracking, or combinations of these methods to yield mineral insulating oil meeting the requirements of this specification. The generic process should be specified upon request.

X3.3 *Shipping Containers*—Mineral insulating oil is usually shipped in rail cars, tank trucks (trailers), or drums. Rail cars used for shipping mineral insulating oil are usually not used for shipping other products and are more likely to be free of contamination. Tank trucks may be used for many different products and are more subject to contamination. Oil drums are most often used for shipping small quantities. All shipping containers, together with any attendant pumps and piping, should be cleaned prior to filling with oil and should be properly sealed to protect the oil during shipment.

Appendix C: ASTM D1816



Designation: D 1816 – 84a (Reapproved 1990)

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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If not listed in the current combined index, will appear in the next edition.

Standard Test Method for Dielectric Breakdown Voltage of Insulating Oils of Petroleum Origin Using VDE Electrodes[1]

This standard is issued under the fixed designation D 1816; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the dielectric breakdown voltage of insulating oils of petroleum origin. This test method is applicable to liquid petroleum oils commonly used in cables, transformers, oil circuit breakers, and similar apparatus as an insulating and cooling medium. The suitability of this test method for testing oils having viscosities of more than 19 cSt (mm^2/s) (100 SUS) at 40°C (104°F) has not been determined.

1.2 This test method is more sensitive to the deleterious effects of moisture in solution than is Test Method D 877, especially when cellulosic fibers are present in the oil. It has been found to be especially useful in diagnostic and laboratory investigations of the dielectric breakdown strength of oil in insulating systems.

1.3 This test method is recommended for testing filtered, degassed, and dehydrated oil prior to and during the filling of power systems apparatus rated above 230 kV, and for testing samples of oil from the apparatus after filling. It is also finding increased usage for testing oils from transformers in service.

1.4 This test method is not recommended for, and should not be used for, acceptance tests on oil received from vendors in tank cars, tank trucks, or drums.

1.5 Both the metric and the alternative inch-pound units are acceptable.

1.6 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry-Cleaning Solvent)[2]
D 877 Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes[3]
D 923 Methods of Sampling Electrical Insulating Liquids[3]

2.2 *IEEE Standard:*

No. 4 Measurement of Voltage in Dielectric Test[4]

3. Significance and Use

3.1 The dielectric breakdown voltage of an insulating liquid is of importance as a measure of the liquid's ability to withstand electric stress without failure. It serves to indicate the presence of contaminating agents such as water, dirt, moisture cellulosic fibers, or conducting particles in the liquid, one or more of which may be present in significant concentrations when low breakdown voltages are obtained. However, a high dielectric breakdown voltage does not necessarily indicate the absence of all contaminants; it may merely indicate that the concentrations of contaminants that are present in the liquid between the electrodes are not large enough to deleteriously affect the average breakdown voltage of the liquid when tested by this test method (see Appendix X1.)

4. Apparatus

4.1 *Transformer*—The desired test voltage may be most readily obtained by a step-up transformer energized from a variable low-voltage commercial power frequency source. To reduce the likelihood of external flashover and to minimize field distortion between the electrodes, a two-bushing, center-tap-grounded transformer is recommended. The transformer and controlling element shall be of such size and design that, with the test specimen in the circuit, the crest factor (ratio of maximum to mean effective) of the 60-Hz test voltage shall differ by not more than ±5 % from that of a sinusoidal wave over the upper half of the range of test voltage. The crest factor maybe checked by means of an oscilloscope, a spheregap, or a peak-reading voltmeter in conjunction with an rms voltmeter. Where the wave form cannot be determined conveniently, a transformer having a rating of not less than ½ kVA at the usual breakdown voltage shall be used. Transformers of larger kVA capacity may be used, but en no case should the short-circuit current in the specimen circuit be outside the range from 1 to 10 mA/kV of applied voltage. This limitation of current may be accomplished by using a suitable external series resistor or by employing a transformer with sufficient inherent reactance.

4.2 *Circuit-Interrupting Equipment*—The test transformer primary circuit shall be protected by an automatic circuit-breaking device capable of opening in three cycles or less on the current produced by breakdown of the test specimen, or up to five cycles if the short-circuit current as described in 4.1 does not exceed 200 mA. The current-sensing element that trips the circuit-breaker should operate when the specimen-circuit current is in the range from 2 to 20 mA. A

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[1] This test method is under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.05 on Electrical Tests.

Current edition approved June 28, and July 27, 1984. Published September 1984. Originally published as D 1816 – 60 T. Last previous edition D 1816 – 82.

[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 10.03.

[4] Available from the Institute of Electrical and Electronics Engineers, Inc., 345 E. 47th St., New York, NY 10017.

prolonged flow of current at the time of breakdown causes carbonization of the liquid and pitting and heating of the electrodes, and thereby increases the electrode and test cell maintenance, and time of testing.

4.3 *Voltage-Control Equipment*—The rate of voltage rise shall be ½ kV/s ± 20 %. The rate-of-rise may be calculated from measurements of the time required to raise the voltage between two prescribed values. Voltage control may be secured by a motor-driven variable-ratio-autotransformer. Preference should be given to equipment having an approximate straight-line voltage-time curve over the desired operating range. Motor drive is preferred to manual drive because of the ease of maintaining a reasonably uniform rate-of-voltage rise with this test method. When motor driven equipment is used, the speed control rheostat should be calibrated in terms of rate-of-voltage rise for the test transformer used.

4.4 *Voltmeter*—The voltage shall be measured by a method that fulfills the requirements of IEEE Standard No. 4, giving rms values, preferably by means of:

4.4.1 A voltmeter connected to the secondary of a separate potential transformer, or

4.4.2 A voltmeter connected to a well-designed tertiary coil in the test transformer, or

4.4.3 A voltmeter connected to the low-voltage side of the testing transformer if the measurement error can be maintained within the limit specified in 4.5.

4.5 *Accuracy*—The combined accuracy of the voltmeter and voltage divider circuit should be such that measurement error does not exceed 5 % at the rate-of-voltage rise specified in 4.3.

5. Electrodes

5.1 The electrodes shall be polished brass spherically-capped electrodes of the VDE (Verband Deutscher Elektrotechniker, Specification 0370) type having the dimensions shown in Fig. 1. They shall be mounted with axes horizontal and coincident.

5.2 The test cell shall be designed to permit easy removal of the electrodes for cleaning and polishing.

6. Test Cell

6.1 The test cell shall be approximately cubical. A cell having a capacity of approximately 0.95 L has been found to be satisfactory for an electrode spacing of 2 mm or 0.080 in. A cell having a capacity of approximately 0.5 L has been found to be satisfactory for an electrode spacing of 1 mm or 0.040 in. Electrodes shall be mounted rigidly from opposite sides with the gap approximately centered. Clearance from all other sides and any part of the stirring device shall be at least 13 mm (½ in.). The test cell shall be provided with a motor-driven two-bladed impeller measuring approximately 35 mm (1⅜ in.) between the blade extremities, having a pitch of approximately 40 mm or 1.57 in. (blade angle of approximately 20°), operating at a speed between 200 and 300 rpm. The impeller, located below the lower edge of the electrodes, shall rotate in such a direction that the resulting liquid flow is directed downward against the bottom of the test cell. The test cell shall be made of a material of high dielectric strength, shall be insoluble in and unattacked by any of the cleaning or test liquids used, and shall be nonabsorbent to moisture and the cleaning and test liquids. So that the breakdown may be observed, transparent materials are desirable, but not essential. In order to preclude stirring air with the sample, the cell should be provided with a cover or baffle that will effectively prevent air from contacting the circulating liquid.

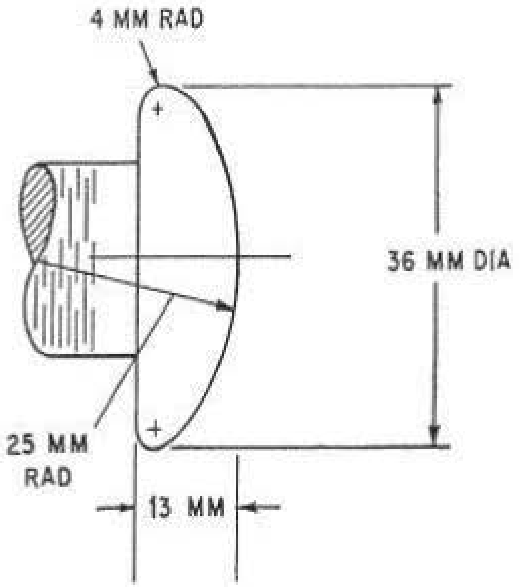

FIG. 1 VDE Electrode

7. Adjustment and Care of Electrodes and Test Cell

7.1 *Spacing*—With the electrodes firmly locked in position, the electrodes shall be checked with a standard round gage for 2-mm or 0.080-in. spacing, when a voltage source of a suitable range is available, or for 1-mm or 0.040-in. spacing when the test transformer voltage limit is restricted to approximately 50 kV. Flat "go" and "no-go" gages may be substituted having thicknesses of the specified value ±0.03 mm for electrode spacings of 1 or 2 mm, or thicknesses of the specified value ±0.001 in. for spacings of 0.040 or 0.080 in. If it is necessary to readjust the electrodes, they shall be relocked, and the spacing again checked.

7.2 *Cleaning*—The electrodes and the cell shall be wiped clean with dry, lint-free tissue paper, or a clean dry chamois. It is important to avoid touching the electrodes or the cleaned gage with the fingers or with portions of the tissue paper or chamois that have been in contact with the hands. After adjustment of the gap spacing, the cell shall be rinsed with a dry hydrocarbon solvent, such as kerosine or solvents of Specification D 235. A low-boiling solvent should not be used, as its rapid evaporation may cool the cell, causing moisture condensation. If this occurs, before using, the cell should be slightly warmed to evaporate the moisture. Care shall be taken to avoid touching the electrodes or the inside of the cell after cleaning. After thorough cleaning, the cell shall be flushed with new, dry, filtered oil of the type to be tested (preferably degassed oil). A voltage breakdown test shall be made on a sample of this oil in the manner specified in this test method. If the breakdown voltage is in the proper range for this conditioned oil, the cell shall be considered as properly prepared for testing other samples. A lower than anticipated value shall be considered as evidence of cell contamination, and it shall again be thoroughly cleaned, and the breakdown test repeated with a clean dry oil.

7.3 *Daily Use*—At the beginning of each day's testing, the electrodes shall be examined for pitting and carbon accumulation, and the spacing checked. If the test of any sample is below the breakdown value being used by the operator as a minimum satisfactory value, the cell shall be drained and flushed with a good quality oil of the type being tested before testing the next sample. When not in use, the cell shall be kept filled with good quality oil of the type normally tested.

7.4 *Polishing of Electrodes*—When electrodes show slight etching, scratching, pitting, or carbon accumulation, they should be removed from the test cup and polished by buffing with jeweler's rouge using a soft cloth or soft buffing wheel. The residue from the buffing should be removed by repeated wiping with lint-free tissue paper saturated with a suitable solvent (such as petroleum ether), followed by solvent rinsing or ultrasonic cleaning. After careful inspection, any electrodes from which pitting cannot be removed by light buffing should be discarded or refinished to the original contour and finish, as more severe refinishing would destroy the electrode contour and dimensions shown in Fig. 1. Reinstall the electrodes in the test cup and adjust spacing and clean in accordance with 7.1 and 7.2.

8. Sampling

8.1 A sample of the oil to be tested shall be obtained with appropriate ASTM sampling apparatus. Oil sampling procedures are detailed in Methods D 923. Particular reference should be made to the general precaution statement of this test method. The sample shall be taken in a dry, clean bottle which shall be tightly sealed and shielded from light until ready to be tested.

8.2 The dielectric breakdown voltage of liquids may be seriously impaired by the migration of impurities through the liquid. In order that a representative test specimen containing the impurities may be obtained, the sample container shall be gently inverted and swirled several times before filling the test cell. Rapid agitation is undesirable, since an excessive amount of air may be introduced into the liquid. Immediately after agitation, a small portion of the sample shall be used to rinse the test cell. The cell shall then be filled slowly with the remaining portion of the sample. There shall be an interval of at least 3 min between filling and application of voltage for the first breakdown, and at least 1-min intervals before applications of voltage for successive breakdowns. During these intervals and at the time voltage is being applied, the propeller shall be circulating the oil.

9. Test Temperature

9.1 The temperature of the sample when tested shall be the same as that of the room, but the room temperature shall in no case be less than 20°C (68°F). Testing liquids at temperatures lower than that of the room may give variable and unsatisfactory results.

10. Procedure

10.1 *Rate-of-Rise of Voltage*—Apply the voltage and increase from zero at the rate of approximately ½ kV/s until breakdown occurs, as indicated by operation of the circuit-interrupting equipment; record the value. Occasional momentary discharges may occur which do not result in operation of the interrupting equipment; these shall be disregarded.

10.2 When it is desired to determine the value of the dielectric breakdown voltage of an oil, make five breakdowns on one filling of the cup. Examine the five breakdowns for statistical consistency, and if they meet the criterion described in 10.3, use their average to determine the dielectric breakdown voltage of the sample. If they do not meet this criterion, make five additional breakdowns on the sample, and use the average of all ten breakdowns as the dielectric breakdown voltage of the sample.

10.3 *Criterion for Statistical Consistency:*

10.3.1 Calculate the mean and standard deviation of the five breakdowns as follows:

$$\bar{X} = \tfrac{1}{5} \sum_{i=1}^{5} X_i \quad \text{and} \quad s = \sqrt{\tfrac{1}{4}\left[\sum_{i=1}^{5} X_i^2 - 5\bar{X}^2\right]}$$

where:

$\bar{X}_i$ = mean of the five individual values,

X_i = *i*th breakdown voltage, and

s = standard deviation.

If the ratio $s/\bar{X}$ exceeds 0.1, it is probable that the standard deviation of the five breakdowns is excessive and therefore that the probable error of their average is also excessive.

10.3.2 *Alternative Criterion*—Calculate the range of the five breakdowns (maximum breakdown voltage minus minimum breakdown voltage), and multiply this range by three. If the value so obtained is greater than the next to the lowest breakdown, it indicates that the standard deviation of the five breakdowns, and, therefore, the probable error of their average value, is excessive.

10.4 When it is desired merely to determine if the dielectric strength is above or below a specified level, only five breakdowns are required, provided the five values are all above or all below this level. Otherwise, follow the procedure described in 10.2.

11. Report

11.1 The report shall include the following:

11.1.1 The test method used,

11.1.2 If the number of tests are as specified in 10.2, the volts (rms value) at each breakdown, and the average of all breakdowns reported to the nearest 1 kV,

11.1.3 If the test is made in accordance with 10.4, the report shall be made in one of the following ways:

11.1.3.1 "Passed" if all of the five breakdowns were above a previously established value,

11.1.3.2 "Failed" if all of the five breakdowns were below a previously established value,

1.1.3.3 "Not less than the minimum of the five breakdowns" if all were above a previously established value, or

11.1.3.4 "Not greater than the maximum of the five breakdowns" if all were below a previously established value.

11.1.4 The approximate temperature of the oil at the time of the test, and

11.1.5 The electrode spacing.

12. Precision and Bias

12.1 The precision of this test method has not been determined. No statement can be made about the bias of this test method because a standard reference material is not available.

APPENDIX

(Nonmandatory Information)

X1. OTHER FACTORS THAT AFFECT THE DIELECTRIC BREAKDOWN VOLTAGE OF INSULATING LIQUIDS AT COMMERCIAL POWER FREQUENCIES

X1.1 The dielectric breakdown voltage of a liquid at commercial power frequencies is also affected by the degree of uniformity of the electric field, the area of the electrodes or volume of the liquid under maximum stress, the length of time for which the liquid is under stress, the temperature of the liquid (especially as it affects the relative saturation level of moisture in solution), gassing tendencies of the liquid under the influence of electric stress, concentration of dissolved gases (especially if saturation levels are exceeded as a result of sudden cooling or decrease in pressure, which may cause the formation of gas bubbles), incompatibility with materials of construction, and velocity of flow. A decrease in dielectric strength of the liquid can have an accentuated effect on the electric creepage strength of solid insulating materials immersed in the liquid.

X1.2 Because of the separate, cumulative, and in some cases, interacting effects of the influences listed above, the average breakdown voltage of a liquid as determined by this test method cannot be used for design purposes. Alternative test procedures that may yield more meaningful indications of the functional dielectric strength of a liquid are under consideration (for example, a step-by-step method of applying voltage, and using the lowest rather than the average value of breakdown voltage obtained in the prescribed number of tests).

Appendix D: ASTM D1533

Designation: D1533 − 12

Standard Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration[1]

This standard is issued under the fixed designation D1533; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the measurement of water present in insulating liquids by coulometric Karl Fischer titration. This test method is used commonly for test specimens below 100 % relative saturation of water in oil. The coulometric test method is known for its high degree of sensitivity (typically 10 µg H_2O). This test method requires the use of equipment specifically designed for coulometric titration.

1.2 This test method recommends the use of commercially available coulometric Karl Fischer titrators and reagents.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practice and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see 8.1 and A2.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D923 Practices for Sampling Electrical Insulating Liquids

2.2 *IEC Standard:*

IEC 60814: Insulating Liquids—Oil-Impregnated Paper and Pressboard—Determination of Water by Automatic Coulometric Karl Fischer Titration[3]

3. Summary of Test Method

3.1 This test method is based on the reduction of iodine containing reagent according to the traditional Karl Fischer reaction. The proposed reaction mechanism is as follows:[4]

$$SO_2 + CH_3OH + RN = [RNH]SO_3CH_3 \quad (1)$$

$$H_2O + I_2 + [RNH]SO_3CH_3 + 2RN = [RNH]SO_4CH_3 + 2[RNH]I$$

$$(RN = \text{Base})$$

The endpoint is determined amperometrically with a platinum electrode that senses a sharp change in cell resistance when the iodine has reacted with all of the water in the test specimen.

3.2 The coulometric Karl Fischer test method requires the use of an automatic titrator with commercially available reagents. Karl Fischer instruments regenerate iodine coulometrically from the iodide in the Karl Fischer reagent. The test specimen is injected into a titration cell where the iodine consumed by the reaction with water is electrolytically regenerated by anodic oxidation of iodide. The completion of the reaction is detected with a platinum sensing electrode. The coulombs of electricity required to generate the necessary amount of iodine then is converted into the amount of water present in the test specimen by use of the Faraday equation.

3.3 *Titration Cell*—The coulometric titration cell consists of either a sealed vessel containing both an anode and cathode which are separated by a diaphragm or a sealed vessel containing an anode and cathode which are not separated by a diaphragm. In both cells the anode compartment contains a solution consisting of sulfur dioxide, iodide, and an amine in a solvent containing methanol/chloroform or methanol/longer chain alcohol. In the cell with a diaphragm the cathode compartment contains similar reagents optimized for cathodic reduction.

4. Significance and Use

4.1 Electrical characteristics of an insulating liquid may be affected deleteriously by excessive water content. A high water

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[1] This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.06 on Chemical Test.

Current edition approved Dec. 1, 2012. Published December 2012. Originally approved in 1958. Last previous edition approved in 2005 as D1533 – 00(2005). DOI: 10.1520/D1533-12.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] American National Standards Institute, 11 West 42nd Street, New York, NY 10036-8002.

[4] Scholz, E., "Karl-Fischer Titration," Springer-Verlag, Berlin, Heidelberg, New York, Tokyo, 1984, 140 pp.

content may make a dielectric liquid unsuitable for some electrical applications due to deterioration of properties such as the dielectric breakdown voltage.

4.2 These tests are suitable for use in acceptance specifications, in control of processing, and in evaluating the condition of dielectric liquids in service.

5. Interferences

5.1 Compounds such as aldehydes, ketones, free halogens, most acids, and oxidizing or reducing agents may interfere with coulometric Karl Fischer titrators. If a drifting end point is noted, an alternative solvent system or another titration method is warranted. If this drifting end point cannot be corrected, the water values should be regarded as suspect. A detailed discussion of interfering substances can be found in the treatise on aquametry.[5]

5.2 Studies have shown that the water content of an insulating liquid sample may be influenced significantly by the sample container.[6] A sample may either gain or lose water on storage in a glass container depending upon the initial water content of the sample, the manner in which the container is cleaned and dried, and the length of storage time before analysis. In addition, sample bottles should not be dried at temperature in excess of 110°C and should be rinsed with the liquid being tested prior to taking the test specimen.

5.3 Erroneous low readings may be obtained if previous spent oil test specimens are not removed from the solvent system on a routine basis. Excess oil may not mix thoroughly with the solvent system thus preventing the total water content of that test specimen from being measured properly. Stirring should be at such a rate that an oil layer will not form on top of the reagent. If such an oil layer does form while at the instruments maximum stirrer speed, stop testing and remove the oil layer. If accurate results can not be obtained, the solution should be discarded.

5.4 Upon setting up of the titration vessel and solvent system, the walls of the titration vessel should be wetted by swirling the solvent system solution around in the vessel.

6. Apparatus

6.1 *Coulometric Titrator,* consisting of a detector electrode, generator electrode, titration vessel, magnetic stirrer, and control unit.

6.1.1 *Detector Electrodes*—This electrode pair amperometrically determines the end point of titration by measuring a sharp change in cell resistance.

6.1.2 *Generator Electrodes*—This electrode pair performs the cathodic reduction of iodide, which allows the Karl Fischer reaction to take place. It may consist of two platinum meshes or wires separated by a diaphragm within a glass assembly or two platinum meshes or wires not separated by a diaphragm.

6.2 *Titration Flask*—The titration flask will be of suitable capacity and will be protected against atmospheric moisture. A bottom drain cock is desirable but not necessary for removing reagents.

6.3 *Stirrers*—Means for agitation during titration will consist of a magnetic stirrer with a glass or TFE-fluorocarbon-covered stirring bar about 2 to 5 cm long or appropriate to the titration vessel. The bar should be cleaned thoroughly, rinsed with methanol, dried in an oven for 1 h at 100°C, and stored in a desiccator until used. In a sealed system, recleaning and redrying are not necessary for routine use.

6.4 *Transfer Syringes*—Syringes shall be used of a suitable size to accommodate instrument manufacturers' recommendation of sample size. Syringes may be glass or plastic. Glass syringes shall be cleaned and dried for 1 h at 100°C prior to use. Plastic syringes shall be disposed of following each sample use.

6.5 *Needles*—Needles that are to be fitted to the transfer syringes shall be long enough to inject samples directly below the surface of the Karl Fischer reagent. They should be of a large enough gauge to allow for easy transfer of the sample.

6.6 *Septums,* used to seal sample port, allowing the introduction of test specimens with a minimum amount of contamination from atmospheric moisture.

6.7 *Sealing Grease*—If the apparatus does not have a gasket seal, use a sealing grease to seal the titration chamber against atmospheric moisture.

6.8 *Drying Oven,* vacuum or air circulating.

6.9 *Desiccator,* standard laboratory type with color change indicator.

6.10 *Analytical Balance,* capable of weighing to ± 0.001 g.

7. Reagents

7.1 *Purity of Reagents*—Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[7]

7.2 *Coulometric Karl Fischer Reagent,* can be obtained commercially. Refer to Annex A1 for information on alternative solvent systems.

7.2.1 *Anode Reagent,* frequently referred to as vessel solution.

7.2.2 *Cathode Reagent,* frequently referred to as generator solution.

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[5] Mitchell, J., Jr. and Smith, D. M., "Aquametry—A Treatise on Methods for the Determination of Water, Part III—the Karl Fischer Reagent," 2nd ed., J. Wiley and Sons, Inc., New York, NY 1977.

[6] Gedemer, T., "Determination of Water in Oil by Karl Fischer Method, Part II, Changes in Moisture Content During Storage," *American Laboratory 7 (10),* pp. 43-50 (1975).

[7] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7.3 *Verification Solutions*—Verification solutions are available commercially.[8] Verification solutions may be formulated in-house from long chain alcohols.

7.3.1 The moisture content of water-saturated octanol is:

Water-saturated 1-Octanol 39.2 ± 0.85 mg/mL of solution[9]

7.3.2 The water-saturated alcohol can be prepared by adding deionized water to the alcohol (ACS reagent grade) at 25°C such that the final mixture consists of a two-phase system in which the lower water phase is at least 2 cm high. Initially, this solution should be mixed thoroughly and allowed to stand at room temperature for at least three days to achieve complete equilibration.

NOTE 1—For the best accuracy the solution should not be mixed or shaken after standing. Remove the sample aliquot from the top phase and inject it immediately into the titration cell. The degree of saturation of the water-saturated 1-Octanol varies <1 % between 10 and 30°C.

7.3.3 The response of the instrument shall be verified with 1 to 2 µL of water; this can give a response value of 1000 to 2000 µg within the specified precision of the instrument.

8. Safety Precautions

8.1 Pyridine was the organic amine that was traditionally used in Karl Fischer reagents; however, pyridine-free formulations are now available commercially. Pyridine-free reagents titrate faster and are less toxic, less odorous, and more stable than pyridine types. The reagents may contain potentially hazardous chemicals, such as iodine, pyridine, sulfur dioxide, methanol, chloroform, chlorinated hydrocarbons, or other organic materials. Wear chemical resistant gloves when mixing the reagents and removing solution from the titration chamber. Care must be exercised to avoid unnecessary inhalation of reagent vapors or direct contact of the reagents with the skin or eyes. Following accidental spillage, flush the affected area with copious amounts of water.

NOTE 2—Carefully read and follow manufacturers instructions and Material Safety Data Sheet when using commercially available reagent systems.

9. Sampling

9.1 The preferred method for sampling insulating liquids is Practice D923. If the test specimen is cloudy or contains free water, it may be difficult to obtain a representative specimen.

10. Preparation of Apparatus

10.1 Thoroughly clean and dry the titration vessel and then reassemble according to the manufacturer's recommendations. Follow the instructions provided in Annex A2 for detailed instructions on cleaning.

10.2 Fill reagent reservoirs with appropriate reagents according to the manufacturer's instructions.

10.3 Turn the instrument on and allow to stabilize.

11. Verification of System Operation

11.1 The accuracy of titration of the instrument and reagents shall be verified prior to beginning of testing by use of a suitable verification solution (see 7.3) containing a known quantity of moisture. It is desirable to verify the system operation using a solution that approximates the same range of water expected to be in the samples. Verification solutions shall be run with new reagents prior to testing. If verification solution results lie outside parameters established by the manufacturer for acceptable moisture content of the solution, reagents shall be changed and reverified.

12. Procedure

12.1 After verifying the system is operating properly, allow the instrument to restabilize prior to use.

12.2 Follow the manufacturer's instructions for suggested specimen size for an expected range of moisture content.

12.3 Using an appropriate syringe and needle (see 6.4 and 6.5) sample the insulating fluid to be tested. Prior to sampling, rinse the syringe and needle with the liquid to be tested one time.

12.4 Determine the sample mass by difference to three significant figures by weighing the test specimen before and after injection. Alternately, inject a known volume of a sample whose density is known at the test temperature to determine sample size.

12.5 Reagent solutions can be used until verification solutions no longer test accurately. See Section 11 for instructions on the use of verification solutions.

13. Calculation

13.1 Most commercially available coulometric Karl Fischer instruments automatically calculate the water content in ppm or percent. If not, calculate the amount of water in the sample as follows:

$$\text{Water Content, mg/kg (ppm)} = A/B \qquad (2)$$

where:

A = mass of water, (µg) (instrument readout), and

B = mass of test specimen, g.

14. Report

14.1 Report the following information:

14.1.1 Water content of the test specimen in mg/kg (ppm) rounded to the nearest integer.

14.1.2 Sample identification.

14.1.3 Sample temperature when collected, if available.

15. Precision and Bias

15.1 *Precision*:

15.1.1 The following criteria may be used for judging precision of test results on new and used oils at the 95 %

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[8] The sole source of supply of verification solutions, RM 8516 and RM 8507, known to the committee at this time is the National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[9] SRM 2890 Water Saturated 1-Octanol Certificate of Analysis, NIST, Gaithersburg, MD, 25 August 1998.

probability level. The precision statements for this test method were based on 9 samples and 37 laboratories.[10]

15.1.1.1 *Repeatability*—The difference between results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, in the long run, would exceed 7 ppm, only 1 case in 20, in the 0 to 50 mg/kg (ppm) range.

15.1.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, in the long run, would exceed 14 ppm, only 1 case in 20, in the 0 to 50 mg/kg (ppm) range.

15.2 *Bias*—The bias of the procedure in this test method for measuring water content of electrical insulating fluids has not been determined from the collaborative interlaboratory round-robin study, since there is no suitable standard for which the exact water content is known.

15.3 The typical method detection limit (MDL) at the 95 % confidence level has been found to be 8 ppm water for the mineral oil samples.

NOTE 3—The MDL is defined here as the minimum concentration of a substance that can be measured and reported with 95 % confidence using a 7-mL oil sample. The value of 8 ppm has been determined from the reproducibility of the results in interlaboratory test of two new and one used oil sample containing about 11 mg/kg (ppm) water.

NOTE 4—A value of the MDL for an individual laboratory may be calculated from the results of *n* replications of complete analysis of a sample using the following equation:[11]

$$\mathrm{MDL}_{0.95} = t_{(n-1,\,0.95)} \times S \qquad (3)$$

where:

$t_{(n-1,\,0.95)}$ = student's *t* value for $n - 1$ df and a confidence level of 95 %, and

S = standard deviation of *n* replicate analyses.

The MDL for an individual laboratory may differ from the MDL of 8 mg/kg (ppm) calculated for this collaborative study.

16. Keywords

16.1 coulometric Karl Fischer titration; free water; Karl Fisher method; Karl Fischer reagent; moisture content; solvent verification solutions; water content

ANNEXES

(Mandatory Information)

A1. ALTERNATIVE SOLVENT SYSTEMS

A1.1 Some high viscosity oils, as well as silicones, may give erroneous water values when titrated in accordance with this test method. Many of these problems, however, may be overcome by the use of alternative solvent systems.

A1.2 If a problem with erroneous answers is known to exist, or is suspected, check the instrument calibration before and after the introduction of a suspected test specimen. If calibration discrepancies are noted, the solvent system or the applicability of the method is suspect and changing of the solvent system from a methanol-chloroform system to a more appropriate solvent system probably is indicated.

A1.3 For certain high viscosity oils, the addition of a low molecular weight hydrocarbon solvent, such as heptane or toluene, to the titration vessel has been found to be suitable.

A1.4 For dimethylsilicones, a 1:1 mix of Karl Fischer reagent and formamide for the titration solvent has been found to be suitable.

A1.5 If an alternative solvent system is used, instrument or titrant calibration checks must be made before and after the addition of the test specimen. If significant calibration discrepancies are noted, the results should be regarded as suspect.

A1.6 To determine the accuracy of a method when using an alternative solvent system, test specimens of known water content should be analyzed. If such samples are not available, commercial standards[8] or water-saturated octanol[9] should be used to assess the response of the instrument.

A1.7 Because of the wide variety of insulating liquids and solvent systems possible, no precision and bias statement is possible for these modifications.

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[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D27-1012.

[11] Glaser, J. A., Foerst, D. L., McKee, G. D., Quave, S. A., and Budde, W. L., "Trace Analysis for Wastewaters," *Environmental Science and Technology*, Vol 15, pp. 1426-1435 (1981).

A2. CLEANING OF THE TITRATION VESSEL AND ELECTRODES

A2.1 The system must be kept clean to obtain reliable results. (**Warning**—Procedures listed herein involve the use of nitric acid, either hot, concentrated acid or 75 % aqueous solutions. Nitric acid and its vapors can cause severe burns. Handle concentrated nitric acid with extreme care. This procedure must be used by persons knowledgeable in the safe handling and disposal of this material.)

A2.2 Cleaning is best done with an appropriate solvent for the samples analyzed. Since the commercial generators are made of glass, platinum and TFE-fluorocarbon, they will withstand most solvents and strong acids. If a solvent is known that adequately will clean the frit, it may be used. Otherwise, the following method may be used.

A2.3 If the frit is clogged, it generally can be cleaned by the use of methanol followed by a water rinse, and then nitric acid. The use of a water aspirator or vacuum pump to pull the fluid through the frit helps to ensure that the pores in the frit will be cleaned.

A2.4 First disassemble the generator and rinse in water. Fill a small beaker with enough nitric acid to cover the frit as the generator is slowly lowered into it. Attach the aspirator to the generator, and pull enough acid through the frit to at least cover the cathode. The acid initially will be dark brown. Discard this acid and pull additional acid through the frit until the acid is colorless.

A2.5 Most deposits can be removed with the use of 75 % nitric acid. Some deposits may require the use of hot, concentrate nitric acid. An aqueous 75 % nitric acid solution should be tried first, and only if that does not remove the deposits, should concentrated nitric acid be used.

A2.6 After cleaning the deposits from the frit or generator, or both, (with nitric acid, as described in A2.3-A2.5) make sure that all the nitric acid is rinsed from the equipment with water to be followed by methanol.

A2.7 Dry the entire assembly in an oven for at least 30 min at 65°C before assembling and refilling the unit.

A2.8 The entire system will require occasional cleaning while the generator will require periodic cleaning. Inspect the sensing electrode for any cracks in the glass or vapor in the glass tube. If there are vent holes in the cover or generator tubes, these should be kept open and inspected occasionally. Consult the manufacturer's manuals for general cleaning procedures.

Appendix E: RESULT OF BREAKDOWN VOLTAGE TEST

TEST	BREAKDOWN VOLTAGE (kV)				
	FO	VO	NS	MS Bigger pore size(1µm)	MS Smaller pore size (0.22µm)
1	6	8	22	8	16
2	7	8	28	9	19
3	6	8	22	9	21
4	7	7	26	10	17
5	6	10	26	10	23
6	6	8	20	8	18
7	7	8	21	6	20
8	8	11	26	9	20
9	7	8	26	9	19
10	6	8	23	8	22
11	7	8	15	8	19
12	7	11	21	10	16
13	7	8	21	12	15
14	8	8	17	9	20
15	8	8	23	12	19
16	8	11	23	10	14
17	8	8	26	10	18
18	7	8	21	8	20
19	8	8	23	11	19
20	8	8	20	10	20
21	8	9	24	8	19
22	7	9	21	14	16
23	7	11	18	8	16
24	6	9	19	10	16
25	9	9	20	10	17

26	8	9	23	8	20
27	7	9	20	11	22
28	9	9	18	8	16
29	8	11	21	11	16
30	8	10	20	9	19
31	9	10	20	8	19
32	8	9	19	8	14
33	7	9	21	9	16
34	9	10	19	10	15
35	8	8	14	10	14
36	9	11	14	11	16
37	9	8	12	11	14
38	9	9	18	8	16
39	8	11	14	10	18
40	8	8	16	10	18
41	9	11	19	10	17
42	9	10	14	8	19
43	8	9	12	10	13
44	7	10	15	11	15
45	8	9	17	8	14
46	8	10	16	10	17
47	10	9	16	10	13
48	8	11	17	8	14
49	8	10	14	8	15
50	9	9	17	8	17

The Breakdown Voltage (BDV) of Fresh Oil (FO), Vacuum Oven (VO), Nitrogen Saturated (NS), and Molecular Sieve (MS).