ENHANCING THE BREAKDOWN VOLTAGE OF MINERAL OIL MIXED WITH NATURAL ESTER OIL

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A thesis is submitted in fulfillment of the requirements for the degree of Bachelor of Electrical Engineering (Industrial Power)

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“I hereby declare that I have read this report and in my opinion this project is sufficient in terms of scope and quality for the award of the degree of Bachelor of Electrical Engineering (Industrial Power) with Honors”.

Signature : …………………………...
Supervisor’s Name : Imran Bin Sutan Chairul
Date :

"I declare that this report entitled "*Enhancing the Breakdown Voltage of Mineral Oil mixed with Natural Ester Oil*" is the results of my own research except cited in references. The report has not been accepted for any degree and is not currently submitted in candidature of any other degree".

Signature : ……………………………….

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Date :

Dedicated to my beloved father and mother,

Hashim Bin Mat Ali & Ruhaida Binti Sulaiman

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ABSTRACT

Insulating liquid in the power transformer applications functioning as a coolant and an insulator. Till now many researcher do many research and find an alternative in replacement of conventional Mineral Oil (MO) that usually used in the transformer. Besides that, Palm Fatty Acid Ester (PFAE) oil is one of the Natural Ester Oil (NEO) that offers many advantages and because of that reason, PFAE oil is preferred and suggested to be used in mixed with MO in order to enhance the breakdown voltage (BDV) for this project. Thus, the main objective in this study is to obtain the best ratio of mixed insulating oil between MO and PFAE oil without reducing too much quantity of MO in order to enhance the BDV and ensuring the best ratio is non-corrosive and has great flash point. The BDV test is carried out using Megger OTS60PB in accordance with the ASTM D1816 standards with 1 mm electrode gap and Coulometric Karl Fischer Titration in accordance with the ASTM D1533 standard is used for the water measurement. Hence, the measurement of corrosive sulfur is performed using Method B from ASTM D1275 standard while Pensky-Martens closed cup tester according to ISO 2719:2002 standard is used for flash point measurement. Throughout the experiment that conducted, the ratio of 70% MO + 30% PFAE represents the best ratio in enhancing the BDV with the properties for flash point which is 156°C. Besides that, the ratio of 70% MO + 30% PFAE has great properties for corrosive sulfur where the copper strip that immersed in insulating oil for this ratio shows slight tarnish or other word is non-corrosive. Hence, it is take into account of 30% of ester content of PFAE oil can improve the dielectric strength without degrade the heat transfer and gives less significantly BDV when presence of moisture content as well as non-corrosive with great the flash point measurement.

ABSTRAK

Penebat cecair dalam aplikasi kuasa pengubah berfungsi sebagai penyejuk dan penebat. Sehingga kini ramai penyelidik melakukan banyak penyelidikan dan mencari alternatif dalam penggantian minyak mineral (MO) konvensional yang biasanya digunakan dalam pengubah. Selain itu, minyak ester asid lemak sawit (PFAE) adalah salah satu daripada minyak ester semulajadi (NEO) yang menawarkan banyak kelebihan dan kerana sebab itu, minyak PFAE adalah pilihan dan dicadangkan untuk digunakan dalam campuran dengan MO untuk meningkatkan voltan kerosakan (BDV) bagi projek ini. Oleh itu, objektif utama dalam kajian ini adalah untuk mendapatkan nisbah yang terbaik minyak penebat campuran antara MO dan minyak PFAE tanpa mengurangkan terlalu banyak kuantiti MO bagi meningkatkan BDV dan memastikan nisbah yang terbaik adalah bukan menghakis dan mempunyai takat kilat yang bagus. Ujian BDV dijalankan menggunakan Megger OTS60PB mengikut piawaian ASTM D1816 dengan jurang 1 mm elektrod dan Coulometric Karl Fischer Titration mengikut piawaian ASTM D1533 digunakan untuk mengukur air. Oleh itu, pengukuran sulfur menghakis dilakukan dengan menggunakan Kaedah B dari piawaian ASTM D1275 manakala Pensky-Martens tester mengikut piawaian ISO 2719: 2002 digunakan untuk mengukur takat kilat. Sepanjang eksperimen yang dijalankan, nisbah 70% MO + 30% PFAE mewakili nisbah terbaik dalam meningkatkan BDV dengan sifat-sifat untuk takat kilat iaitu 156°C. Selain itu, nisbah 70% MO + 30% PFAE mempunyai ciri-ciri yang bagus untuk sulfur menghakis di mana jalur tembaga yang tenggelam dalam penebat minyak untuk nisbah ini menunjukkan sedikit cemar atau perkataan lain adalah bukan menghakis. Oleh itu, adalah mengambil kira 30% daripada kandungan ester minyak PFAE boleh meningkatkan kekuatan dielektrik tanpa merendahkan pemindahan haba dan memberi kurang ketara BDV apabila kehadiran kandungan kelembapan serta bukan menghakis dengan potensi yang bagus pengukuran takat kilat.

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LIST OF ABBREVIATIONS

AC	-	Alternating Current
ANOVA	-	Analysis of Variance
ASTM	-	American Society for Testing and Materials
BDV	-	Breakdown Voltage
ECT	-	Electrostatic Charging Tendency
IEC	-	International Electro Technical Commission
ISO	-	International Organization for Standardization
MO	-	Mineral Oil
MS	-	Malaysia Standard
NEO	-	Natural Ester Oil
PB	-	Portable
PCB	-	Polychlorinated Biphenyl
PFAE	-	Palm Fatty Acid Ester
PPM	-	Parts Per Million
RBDPO	-	Refined Bleached Deodorized Palm Oil
SOP	-	Standard of Procedure

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CHAPTER 1

INTRODUCTION

1.1 Overview

This chapter will review about the research background and motivations, problem statement, objectives of this study, scopes of works, contribution of research and end with thesis outline. The general overview of this study will briefly explain in research background and motivations. Hence, the issues that need to be solved during this study will be explained in the problem statement. For objectives of this study and scopes of works will be described about the main purpose of this study conducted that need to be achieved in order to solve the problems that have been identified. Lastly, the report outline will shows the summary of chapter provided in this thesis.

1.2 Research Background and Motivations

In the electric power transmission and distribution system, the transformer has very important responsibility in providing a reliable and efficient electricity supply. The transformer is listed as the most critical equipment for high voltage equipment. The liquid that filled in the power transformers applications are working as an electrical insulation and as a cooling medium. Usually, mineral oil is one of the liquid insulation that often used in the high voltage transformer which is associated with the reason of lower cost and good properties besides low in losses of dielectric, high the strength of electric field and good performance for a long term. However, in considering the environmental factor makes the mineral oil has a limited point. The first one is mineral oil is well known as non-biodegradable which can contaminate soil and water when there is serious oil spills. Hence, mineral oil also is non-renewable because mineral oil is produced from the base of petroleum and it will run out in the future because of petroleum is nonrenewable [1].

For that reason, many researchers gives an efforts rapidly on developing a fully biodegradable insulating fluid that were started in the mid-1990s [2]. Natural ester oil (NEO) gives more advantages compared to mineral oil in terms of fire safety, environmental friendly and insulation ageing. In fact, NEO is a non-toxic, more biodegradable, and less flammable and it will be more suitable that can be used in the transformer insulation system [1, 3]. Malaysia is well known in palm plantation and palm oil also gives a lot of impact in a variety of application use. Many researchers [4] are studied to make palm-oil as based transformer oil as one of the alternative. There are a lot of benefits of palm oil which are the cooling stability of palm oil is the highest and very good oxidation stability. A knowledge to know about palm oil, it can be potential to substitute to mineral oil due to has excellent in dielectric properties. Since 2006, Lion Corporation take initiatives to develop Palm Fatty Acid Ester (PFAE) as an insulation oil for transformer. PFAE has superior insulation performances in high voltage transformer and environmental friendly [4, 5].

In recent years, investigations on mixed insulating oil between mineral oil and natural ester oil are being worked out and become interested from many researchers. I. Fofana [6] propose the mixed insulating oil in order to enhance its stability of insulating oil which consists of dielectric, flash point, and thermal properties. Mixed insulating oil which mixtures of mineral and ester oils have been reported that having a good thermal performance in term of improved oxidation stability besides to minimize the lower cost of transformer. Since development of the mixed insulating oil become popular among researcher, a report stated that the optimal ratio of mixed insulating oil is consists of 80% of mineral oil and 20% of ester oil [7].

In the last decade, the presence of corrosive sulfur compound have been reported as typical winding failures in transformers and shunt reactors due to the resultant formation of copper sulfide (Cu_2S). Corrosive sulfur is discussed as unexpected failure in electric power transformer and caused the power grid has great loss [8, 9]. In transformer oil, the corrosive sulfur originates mainly from the refining process of mineral oil in which process there were residual sulfur ingredients. The content of corrosive sulfur in transformer oil is probably 5% or more and sulfur was named due to can occur with the copper wire active sulfur. Copper sulfide is the main component of the deposition on the copper and insulation paper which had experienced the corrosion. The reaction between the corrosive sulfide in the transformer and the copper wire at 80~150°C produces the product which is copper sulfide. On the other hand, Dibenzyl Disulfide (DBDS) is pointed out that the main corrosive sulfide occurred in the transformer oil. The concentration of oxygen in the oils is take into account that affects the deposition of copper sulfide on the paper surface [10].

Since insulating oil is taking into account of safety in terms of flammable liquids and fire hazards, many researchers are carried out the study in terms of physical properties especially conducting the flash point. The requirement to do study the flash point on insulating liquid due to quality control purposes as well as for controlling the flammability risk and classification of liquid where to warn a risk and enable the correct precautions to be taken when using, storing or transporting the insulating oil [11].

Therefore, many researchers have been carried out the research and gives the efforts to identify the optimal ratio of mixing of mineral oil and PFAE oil to produce as a liquid insulating materials for power transformer applications. Besides that, a further studies need to be done to resolve the issue of corrosive sulfur in transformer oil and propose effective suppression measures. From this way can ensure the safe and stable operation of the power transformer.

1.3 Problem Statement

Up to now, recent studies showed that mineral oil (MO) as insulating oil that often used in power transformer application has been reported has weakness in terms of environmental factors, non-biodegradable, non-renewable and lowest moisture content. Meanwhile, previous studies stated that PFAE oil based natural ester oil (NEO) has promoted many advantages in terms of fire safety, environmental friendly, more biodegradable, less flammable, non-corrosive and highest moisture content. Therefore, the best ratio of mixed insulating oil between MO and PFAE oil will be produced in order to enhance its stability which consists of breakdown voltage (BDV) without reducing the quantity of MO too much. Since the effect of corrosive sulfur on mixed insulating oil is not be done research widely by any researchers and it is thus necessary gives opportunity to carry out of study the effect of corrosive sulfur on mixed insulating oil. In ensuring the mixed insulating oil that produced is presents the best ratio of concentration of mixed insulating oil, the study of flash point is conducted on mixed insulating oil in order to determine the minimum temperature at which heated of mixed insulating oil gives off sufficient vapor to form a flammable mixture with air.

1.4 Objectives

There are three objective that need to be accomplished in order to make this project successful which are:

1. To obtain the mixed insulating oil with the best ratio for the concentration of mineral oil mixed with natural ester oil by assessing the AC breakdown voltage.
2. To study the effect of the presence of corrosive sulfur compound that react on contact with copper in the mixed insulating oil.
3. To study the flash point of insulating oil in order to determine which the ratio of concentration of mixed insulating oil that gives the best result for the flash point.

1.5 Scope of Works

The scopes of this study are:

1. The ratio of concentration of mixed insulating oil used the types of mineral oil is Nytro Libra oil and types of natural ester oil used is Palm Fatty Acid Ester (PFAE) oil. The baseline liquid used in this study is 500 milliliter.
2. Breakdown Voltage (BDV) measurement performed by following ASTM D1816 standards which using the gap of electrode 1 mm.
3. The test method for measuring moisture content in insulating oil is complies with ASTM D1533 standards where the test known as Karl Fischer.
4. ASTM D1275 standards is applied as test methods to detect corrosive sulfur in insulating oil while ASTM D130 standards is used as reference of copper strip tarnish level classifications.
5. The test method for the test of flash point is performed in according to ISO 2719:2002 standards which using Pensky-Martens closed cup method.

1.6 Contribution of Research

The expected from this research is by mixing of mineral oil (MO) and natural ester oil (NEO) will give improvement of characteristic and behavior in terms of electrical, chemical and physical properties. This research will be carried out on various sample of mixed insulating oil with differences ratio of concentration in identifying which types of ratio of concentration could enhancing the breakdown voltage (BDV). Since PFAE oil does not contain of any sulfur compound, it could give an advantage for doing mixed with MO in this experiment and thus can provide the best protection from corrosive sulfur in producing the mixed insulating that be used in power transformer application. Hence, the properties of mixed insulating oil can be identified through this experiment which is the value of flash point for mixed insulating oil that conducted in the experiment will be compared with the physical properties which is flash point for both MO and NEO in providing benchmarks. Thus, by this study can acknowledge and identify which type of ratio of concentration of mixed insulating oil will presents the best ratio of mixed insulating oil in terms of dielectric strength, corrosive sulfur and flash point.

1.7 Thesis Outline

In general, this thesis is covers five (5) chapters. Chapter 1 is an overview of the research project. The problem statement, objectives, and scopes of projects are defined in this chapter. The objectives and scopes that have been stated will assist the research of works to be done. Chapter 2 is presents the literature review. This chapter consists of the theory related to this research such as the development of insulating liquid, types of insulating liquid, mixed insulating liquid, corrosive sulfur, flash point, and the importance of transformer insulating liquid testing. It also explained and discussed the review of previous related works regarding to this research. Chapter 3 is illustrated methodologies that apply in order to get required output of the project. This chapter includes the selecting of MO and NEO, pre-processing of MO and NEO, BDV and moisture content of MO and NEO following standard, mixture process between MO and NEO by differences ratio, BDV performed on differences ratio of mixed insulating oil, corrosive sulfur and flash point. Chapter 4 is interpreting and discussion of this study. It is includes present moisture content and the breakdown voltage (BDV) on oil samples with differences ratio of concentration of mixed insulating oil, corrosive sulfur and flash point. Lastly, Chapter 5 is conclusions that have been made throughout of this project.

CHAPTER 2

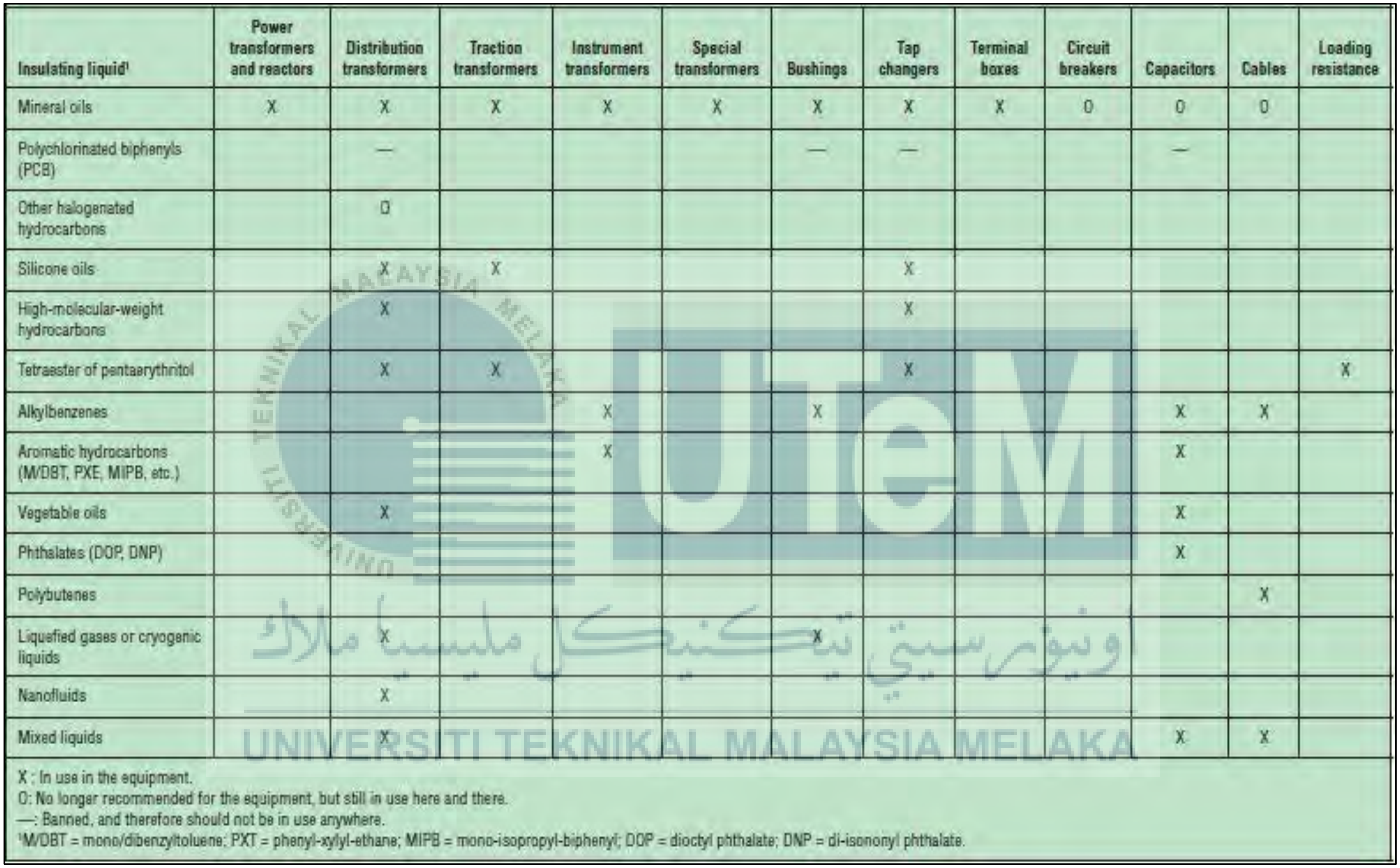
LITERATURE REVIEW

2.1 Introduction

This chapter will described about the literature review that related in this study. Thus, any related theory and relevant information such as development of insulating liquid, types of insulating liquid, mixed insulating liquid, corrosive sulfur, flash point, the importance of transformer insulating liquid testing and Weibull distribution are studied and discussed in this chapter. Meanwhile, the mixed insulating liquid that has been done by from previous researcher also discussed to relate the project conducted.

2.2 Development of Insulating Liquid

Electrical insulation commonly known as critical part in electrical equipment. Insulating liquid has been used worldwide for billion liters in the power electrical equipment for example transformers , resistors, reactors, capacitors, cables, bushings, circuit breakers, tap changers, and etc. Figure 2.1 shows the applications of insulating liquid used in electrical power equipment [6].



Insulating liquid[1]	Power transformers and reactors	Distribution transformers	Traction transformers	Instrument transformers	Special transformers	Bushings	Tap changers	Terminal boxes	Circuit breakers	Capacitors	Cables	Loading resistance
Mineral oils	X	X	X	X	X	X	X	X	O	O	O	
Polychlorinated biphenyls (PCB)		—				—	—			—		
Other halogenated hydrocarbons		O										
Silicone oils		X	X				X					
High-molecular-weight hydrocarbons		X					X					
Tetraester of pentaerythritol		X	X				X					X
Alkylbenzenes				X		X				X	X	
Aromatic hydrocarbons (M/DBT, PXE, MIPB, etc.)				X						X		
Vegetable oils		X								X		
Phthalates (DOP, DNP)										X		
Polybutenes											X	
Liquefied gases or cryogenic liquids		X				X						
Nanofluids		X										
Mixed liquids		X								X	X	

X : In use in the equipment.
O: No longer recommended for the equipment, but still in use here and there.
—: Banned, and therefore should not be in use anywhere.
[1] M/DBT = mono/dibenzyltoluene; PXT = phenyl-xylyl-ethane; MIPB = mono-isopropyl-biphenyl; DOP = dioctyl phthalate; DNP = di-isononyl phthalate.

Figure 2.1: Applications of insulating liquid used in electrical power equipment [6]

In the transformers, insulating liquid plays role as acoustic dampening media and responsible for maintaining the power transformer in good condition. Besides that, insulating liquid has taken responsibility of the functional serviceability of the dielectric insulation system and determine the life span of power transformer application. The information of incipient electrical and mechanical failures can be provided by doing the testing of physicochemical and electrical properties on insulating liquid. Since 1990s, mineral oil based-petroleum has been widely used and become a main insulating liquid in industrial power system due to good aging behavior, low viscosity, low cost and ready availability. In early 1930s, polychlorinated biphenyl (PCB) based insulating liquid has been used in power transformer because of requirement in use high fire security standards. Hence, PCB-free liquid was replaced the PCB since increasing the population density in 1970s. Due to safer nonflammable and environmentally concern, it has been alternative to replace mineral oil and many achievement of significant advances has been made in the last four decades. The properties of insulating liquid used in the last 50 years as shown in Table 2.1 [6].

Table 2.1: The properties of insulating liquid used in the last 50 years [6]

Category	Type of Liquid	Applications	Particular Properties
Mineral oils	Naphthenic, Paraffinic	Liquid-filled power equipment (transformers, circuit breakers, load tap changer, etc.)	• Good resistance to oxidation • Good viscosity index • Relatively low fire point • Low moisture tolerance • Possible sulfur corrosion
High-molecular-weight hydrocarbons	Paraffinic	Transformer, load tap changer	• High flash point
Vegetable oils	Castor, soybean, cotton, palm, etc.	Capacitors, transformers	• Low dielectric losses at frequency higher that 1 kHz • Readily biodegradable • Low oxidation stability
Synthetic hydrocarbons	Polybutenes	Cables	• Low dielectric losses • Adjustable viscosity
	Alkylbenzenes	Bushing, cables, capacitive dividers	• Gas absorbing under partial discharges • Good lightning impulse breakdown strength
	Alkylated hyrdocarbons with condensed aromatic rings (DIPN)	Capacitors	• Good lightning impulse breakdown strength
	Alkylbiphenyls (MIPB)	Capacitors	• Readily biodegradable • Gas absorbing under partial discharge
	Alkyldiarylalcanes (BT, DBT)	Capacitors	• Good lightning impulse breakdown strength

Halogenated hyrdocarbons	Askarels (PCB)	Capacitors and distribution transformers	• Nonflammable • Thermal stability
	Polychoro-diphenyl methanes	Transformers	• Nonflammable • Biodegradable • Thermal stability
	Polychoro-alchanes	Transformers	
Silicone oils	Poly-dimethyl siloxanes or PDMS, Poly-methylphenyl siloxanes	Traction and distribution transformers	• Good viscosity index • High flash point • Gas absorbing under partial discharges • High oxidation stability • Low biodegradability
Organic esters	Simple esters	Capacitors	• Relative permittivity at 20°C higher than 5
	Phtalates	PCB substitute	• Used to increase flash points of some liquids
	Complex esters, tetraester of pentaerythritol	Traction and distribution transformers	• High flash point • High moisture tolerance • Readily biodegradable • High oxidation stability
Other liquids	Ethers (alkyl-diphenyl ether, ditolyl-ether)	Capacitors	• Relative permittivity higher than 3 at 20°C • Adaptability
	Nanofluids	Transformers	• Adaptability
	Mixed liquids	Capacitors, transformers	• Adaptability
	Cryogenic dielectric liquid (nitrogen)	Superconductivity and cryogenic applications	• Reduction or suppression of Joule heating

2.3 Types of Insulating Liquid in Transformer

Insulating liquid that used in the power transformer applications can be divided into different types based on their chemical structures and fire point. Basically, there are (2) types of insulating liquid that commonly used which are; mineral oil (MO) and natural ester oil (NEO). These types have differences of behavior and dielectric properties in the use on its applications.

2.3.1 Mineral Oil (MO)

Since a long time ago mineral oil (MO) has been used widely as oil-immersed transformer due to MO has an excellent insulation performance and lower cost [12]. Basically, MO known as petroleum-based oil and this type of oil is complex mixture where there is hundreds of different compounds consisting of carbon and hydrogen mainly in molecules with different structures [5, 6]. During the distillation of petroleum crude stock, carbon and hydrogen be produced from the refining a fraction of the hydrocarbons that are collected. .The hydrocarbons present in crude petroleum are classified into three (3) general types:

a. Paraffinic

The chemical properties for the paraffinic crude oil is (C_nH_{2n+2}). Paraffinic crude oil contains small amount quantity of naphthenic hydrocarbons. This crude oil can be divided into two (2) types which are normal paraffins and isoparaffins. The molecules of paraffins is straight chain wax type. Meanwhile, isoparaffins is branched molecules. Isoparaffins has lower pour points and this type is more preferred compared to normal paraffins. Besides that, the contain waxes in paraffinic crude oil is much higher than contain wax in naphthenic crude oil [6].

b. Naphthenic

Naphthenic crude oil has chemical properties (C_nH_{2n}) which means this crude oil has high pour points and lower viscosities than paraffinic crude oils. The existence of naphthenic compound in this crude oils is much higher than paraffinic crude oils. Normally, the manufactured of naphthenic crudes oil is from the process of solvent refining with hydroprocessing/hydrotreating or mild hydrofinishing [6].

c. Aromatic

There are many names for aromatic crude oil can be called which are; polynuclear aromatics, polycyclic aromatics, and polyaromatic hydrocarbons. The chemical properties for this crude oil (C_nH_{2n-6}) and the molecules is unsaturated ring. The factor in determine the differences of water solubility of different oils from the aromatic contained. Besides that, polyaromatic hydrocarbons can causes the health concern and naphthenic oils with more than 2% of polyaromatic hydrocarbons also give causes the carcinogenic which dangerous for health [6].

Figure 2.2 shows the form molecules in differences of crude oil. Thus, in producing the new mineral oil have to be following in accordance with the ASTM D3487 or IEC 60296 [13]. Normally, ASTM D3487 standards are covers the criteria of new mineral oil regardless of their crude oil base and origin prior to any processing. This standards also is defines into two (2) types which are: Type I has normal oxidation resistance and Type II has greater oxidation resistance. Hence, Type I does not require any addition of an oxidation inhibitor as it has already contain of naturally occurring inhibitors and Type II are inhibited artificially [14].

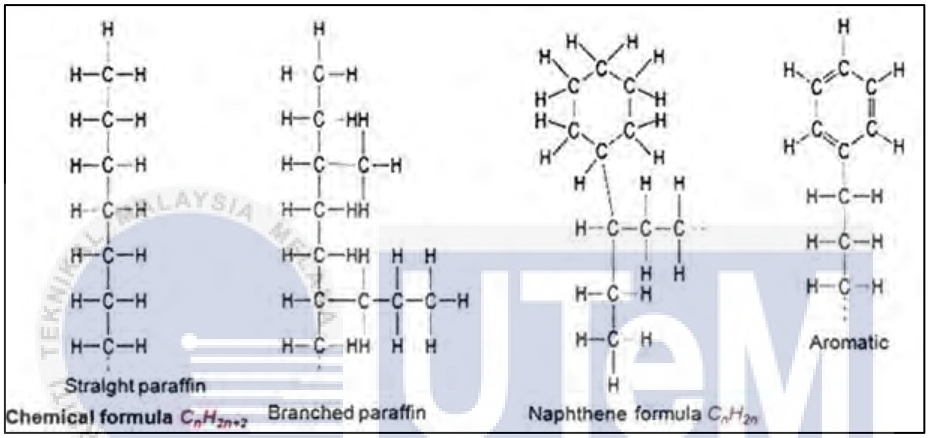


Figure 2.2: Chemical forms of mineral oil [15]

2.3.2 Natural Ester Oil (NEO)

Natural ester (tri-ester) also known as vegetable oil consists of triglycerids which are naturally synthesized by esterification of the tri-alcohol (glycerol) with three fatty acids that obtained from the plant [16, 17]. The chemical composition of vegetable oil is crucial in fully qualify as a dielectric fluid. The oil color of crude vegetable oils that extracted from the seeds is dark. Besides that, crude vegetable oils contains solid constituents such as protein, fibers, and liquids (fats and oils). Triglycerids esters of fatty acids presents both fats and oils where fats has relatively high percentage of saturated triglycerids and can be freeze to solid at below room temperature. Usually, the part of oily remains as liquid when temperature is above 0°C. Besides that, oil with high unsaturation remain as liquid at temperature -15°C and 30°C. The molecules of tryglycerids ester may be presented as; CH_2-$OOCR_1$, CH-$OOCR_2$ and CH-$OOCR_3$ where R_1, R_2 and R_3 are same or different types of fatty acid chains [18]. Basically, the fatty acids are composed through the linear hydrocarbon chains and it is ended by a carboxylic function. Thus, the molecules of these have an even number of carbon atoms which typically from 8 to 22 in tryglycerids and the chain can be saturated or mainly mono-, di- and tri- unsaturated [16]. Transesterification reaction (mono-ester) or mixture of mono and tri-ester is the other process to allow the final product to be obtained [13]. The composition of fatty acid of some vegetable oil as shown in Table 2.2 [6]. In the middle of the nineteen nineties, a fully biodegradable insulating liquids became interesting study and started in development of vegetable oils. Palm Fatty Acid Ester (PFAE) recently has been proposed as a new vegetable-based insulating oil for power transformer [6]. The development of natural ester as insulating liquid as shown in Figure 2.3 [17].

Table 2.2: Typical fatty acid composition of some vegetable oils [6]

Vegetable oil	**Saturated fatty acids (%)**	**Unsaturated fatty acids (%)**		
		Mono-	**Di-**	**Tri-**
Canola oil*	7.9	55.9	22.1	11.1
Corn oil	12.7	24.2	58	0.7
Cottonseed oil	25.8	17.8	51.8	0.2
Peanut oil	13.6	17.8	51.8	0.2
Olive oil	13.2	73.3	7.9	0.6
Safflower oil	8.5	12.1	74.1	0.4
Safflower oil, high oleic content	6.1	75.3	14.2	-
Soybean oil	14.2	22.5	51	6.8
Sunflower oil	10.5	19.6	65.7	-
Sunflower oil, high oleic content	9.2	80.8	8.4	0.2

*Low erucic acid variety of rapeseed oil. Recently canola oil with more than 75% monounsaturated content has been developed.

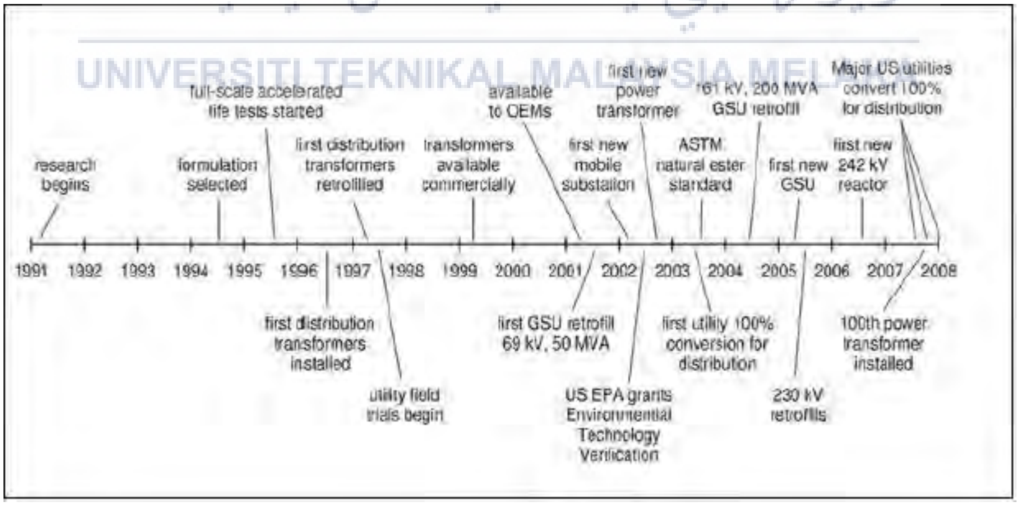


Figure 2.3: The development of natural ester as insulating liquid [17]

2.3.2.1 Palm Fatty Acid Ester (PFAE) Oil

Palm Fatty Acid Ester (PFAE) oil is fatty acid alkyl ester (R^1COOR^2) derived from palm oil and it has great potential for use as insulating medium for transformer such as good biodegradability, superior insulation performance, lower viscosity, high cooling ability, good oxidation stability, high productivity, excellent dielectric properties and environmentally friendly [4, 12]. In the year of 2000s, palm oil and palm kernel oil become the most abundant and consistently supplied among vegetable oil in the world including soybean and rapeseed oil. A photograph of palm nut as shown in Figure 2.4 [19]. Since palm derived shows the highest productivity, its crop yield per unit area is 7 to 10 times higher than other vegetables seed oil [12].

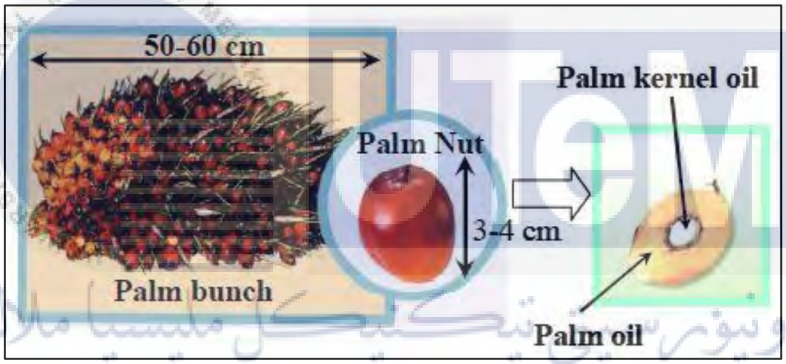
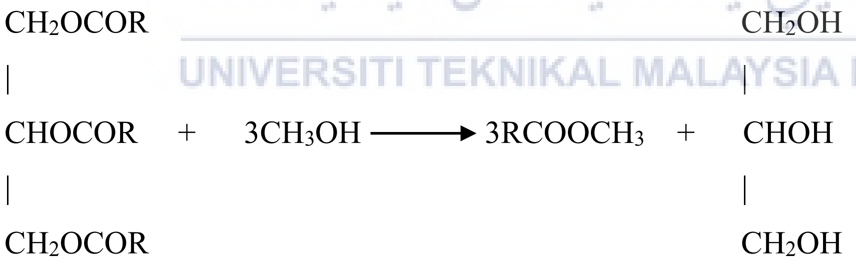


Figure 2.4: Photograph of palm nut [19]

Table 2.3: Composition of fatty acid in oils [19]

Fatty Acid	**Soy-bean oil**	**Rape-seed oil**	**Sunflower oil**	**Palm**		**Coconut oil**	**Beef fat**
				Palm oil	**Palm kernel oil**		
C8: caprylic acid	-	-	-	-	3.3	7.8	-
C10: capric acid	-	-	-	-	3.1	7.6	-
C12: lauric acid	-	-	-	-	45.7	44.8	-
C14: myristic acid	-	-	0.2	1.1	16.4	18.1	2.0
C16: palmitic acid	6.7	5.4	7.1	44.4	8.9	9.5	32.5
C18: stearic acid	3.3	2.0	2.8	4.3	2.3	2.4	14.5
C18: oleic acid	41.7	68.0	30.0	39.9	17.1	8.2	48.3
C18: linoleic acid	41.3	21.0	59.5	9.4	2.7	1.5	2.7
C18: liolenic acid	5.9	-	-	-	-	-	-
Others	1.1	3.6	0.4	0.9	0.5	0.1	-

Basically, palm derived oil has carbons 8 to 18 and wide composition of fatty acids as shown in Table 2.3 [19]. Triglyceride of C16 and C18 fatty acid presents for palm oil. The chemical reaction of PFAE that supplied by Equation (2.1) and (2.2) [12, 19]:



$$\begin{array}{lllll} CH_2OCOR & & & & CH_2OH \\ | & & & & | \\ CHOCOR & + & 3CH_3OH \longrightarrow & 3RCOOCH_3 & + \quad CHOH \\ | & & & & | \\ CH_2OCOR & & & & CH_2OH \end{array} \qquad (2.1)$$

Fat and oil — Methanol — Fatty acid and methyl ester — Glycerin

$$R^1COOCH_3 \quad + \quad R^2OH \longrightarrow R^1COOR^2 \quad + \quad CH_3OH \qquad (2.2)$$

Fatty acid and methyl ester — Alkyl alcohol — Fatty acid alkyl ester — Methanol

Specifically, 99% purity of fatty acid methyl ester is using palm derived oil as a raw material as shown in Equation (2.1) [12, 19]. Fatty acid methyl ester at lower temperature has been through transesterification with other alkyl alcohol in order to improve the liquidity. Hence, fatty acid alkyl ester R^1COOR^2 stands for PFAE oil is synthesized from Equation (2.2) [12, 19] by transesterification from various fatty acid methyl ester with fatty acid moieties of 8 to 18 carbon atoms which means to improve liquidity at low temperature. Besides that, PFAE oil has biodegradability is about 60% which means PFAE oil is more safe for the environment in the event that contamination occurrence of soil or water. PFAE oil has received Eco Mark registration standards due to environmentally friendly [19]. Thus, the properties of PFAE oil and mineral oil as shown in Table 2.4 [5].

Table 2.4: Properties of PFAE oil and mineral oil [5]

Property	**PFAE oil**	**Mineral Oil**
Density at 15℃ [g/cm^3]	0.86	0.88
Kinetic viscosity at 40℃ [mm^2/s]	5.1	8.1
Flash point [℃]	186	152
Pour point [℃]	-32.5	-45
Total acid value [mg KOH/g]	0.005	< 0.010
Moisture [ppm]	15	< 10
Relative permittivity at 80℃	2.95	2.20
Tan δ at 80℃ [%]	0.310	0.001
Volume resistivity at 80℃ [Ω.cm]	1.9×10^{13}	7.6×10^{15}

2.4 Mixed Insulating Liquid

Generally, the importance of mixed insulating liquid to improve the electrical parameters (dielectric strength, specific resistance, and dielectric dissipation factor), chemical parameters (water content and acidity) and physical parameters (viscosity, flash point, and pour point). There are commonly types of insulating liquid that frequently used to be mixed which are; mineral oil, synthetic ester oil, natural ester oil, rapeseed oil. In fact, each oil has their advantage with regard to the other.

2.4.1 Mineral Oil mixed with Synthetic Ester

Mineral oil and synthetic ester are commonly used in producing the mixed insulating oil for improvement of power transformer applications. By mixing these two oil can be improved the characteristic in terms of heat transfer, breakdown voltage (BDV), ageing stability and electrostatic charging tendency (ECT). Basically, the ratio of the concentration used for producing mixed insulating oil is 80% of mineral oil and 20% of synthetic esters due to non-degrading its viscosity and ECT. The types of mineral oil used is naphthenic mineral oil which means this oil is uninhibited and mostly used in large power transformer. Meanwhile, there are two (2) types of synthetic ester used which are; the first type using tetraesters and the second type using based on pentaerythritol (high water solubility) [20]. The properties of naphthenic mineral oil and synthetic ester oil as shows in Table 2.5 [20].

Table 2.5: The properties of naphthenic mineral oil and synthetic ester oil [20]

Type of Oil **Properties (IEC)**	**Naphthenic Mineral Oil**	**Synthetic ester**	
		Tetraesters	**Pentaerythritol**
Density (20℃) g/cm^3	0.89	0.97	0.96
Viscosity (40℃) mm^2/s	8.9	30	35
Pour point ℃	-57	-48	-50
Thermal conductivity (20℃) W/m.K	0.13	0.16	0.15
Flash point ℃ (PM method)	148	257	> 250
Dissipation factor (90℃)	<0.001	0.004	0.02
Dielectric constant (20℃)	2.2	3.2	3.3
Breakdown voltage (kV)	>70	>75	>75
Max. water solubility (25℃) ppm	70	2500	2500

Hence, the previous shows the mixture are homogenous before and after the ageing. The process of miscibility by put the mixed insulating oil in a closing vessel and three ageing stages at 105℃ for 168 hour, after that room temperature for 21 days and the last part at -40℃. Besides that, the BDV test conducted according IEC 60156 standard with gap of the electrode is 2.5 mm and the voltage uniformly increased (2.0 ±0.2 kV/sec) while the oil stirred during the test. The BDV result shows that synthetic ester oil have higher BDV. By mixed 20% of synthetic ester oil enable improvement of BDV average value of mineral oil and avoid its dielectric strength to be reduction after ageing process. In addition, the result of BDV does not follow the "mixture law". Considerably, BDV depends on the water content. Thus, from the BDV result can be analyzed synthetic ester oils have solubility of water so that can reduce the humidity on mineral oils. The probability study on BDV with 32 measurement as shown in Figure 2.5. Then, Figure 2.6 shows the evolution of BDV of oils and mixtures before and after ageing of product alone with 6 measurements [20].

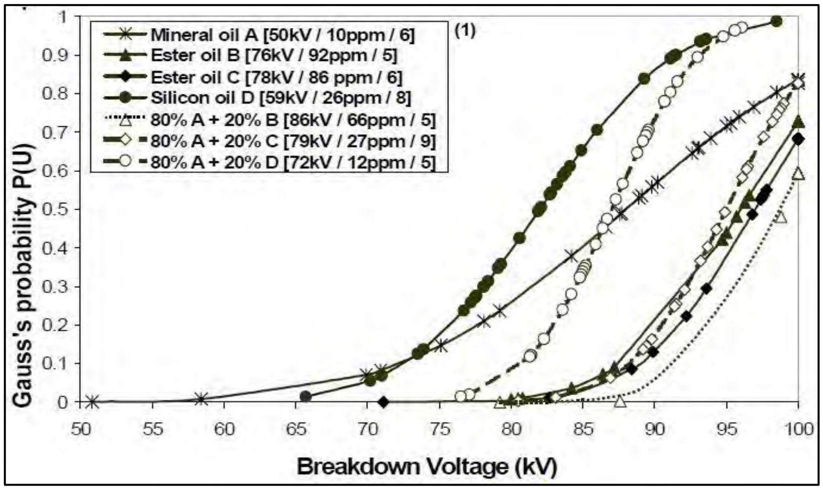


Figure 2.5: The probability study on BDV with 32 measurement [20]

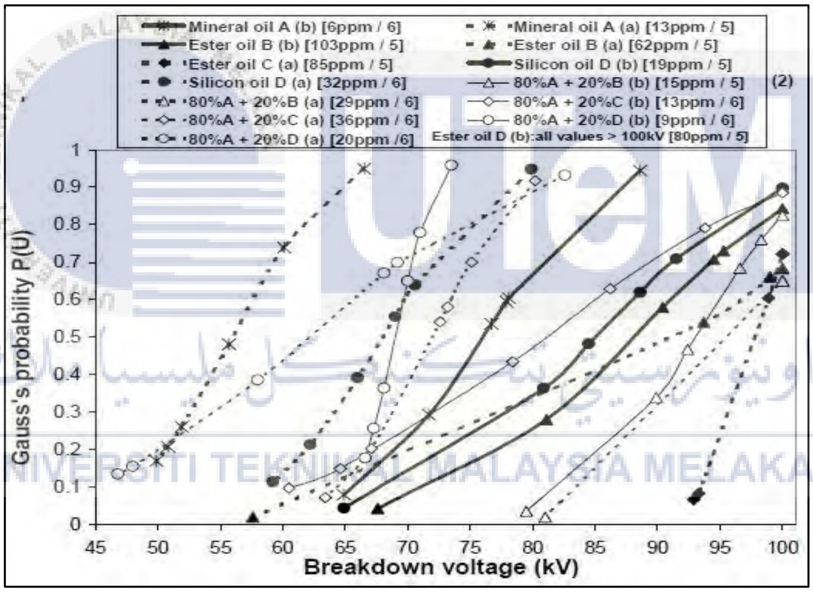


Figure 2.6: The evolution of BDV of oils and mixtures before and after ageing of product alone with 6 measurements [20]

2.4.2 Mineral Oil mixed with Natural Ester

In order to produce new type of liquid insulating material where environmental friendly and excellent in dielectric properties, natural ester is the appropriate liquid in mixture with mineral oil. Natural ester oil is the insulating liquid that has superior characteristic of dielectric strength and water solubility but the losses factor and viscosity are inferior. In addition, mineral oil as mentioned is good thermal properties to evacuate heat from transformer but the factor of ageing and biodegradability are inferior. Thus, through mixing both natural ester oil and mineral oil can be enhanced the dielectric as well as thermal properties of the insulating liquid. The type of mineral oil used is Shell Diala B where its density is 0.873 g/cm^3 meanwhile natural methyl ester oil that obtained from palm oil is presented as natural ester oil. [21].

Hence, by applying the breakdown voltage (BDV) that complies IEC 156 and ASTM D877 standards which the gap of electrode is 2.5mm and the frequency 50 Hz with increasing rate of 2 kV/s on mineral oil mixed with natural ester oil, the result shows that the BDV is increased when the content of natural methyl ester is increased as shown in Figure 2.7. Besides that, the BDV test on pure of natural methyl ester is significantly larger than BDV test on pure mineral oil. It can be conclude that the improvement of the quality of mixture can be improved by increasing the content of natural methyl ester. Besides that, the temperature and the relative water content, W_{tr} can affected the BDV result. The BDV test be reduction due to the increasing the relative water content. Natural ester is known as has much higher saturation water content and thus natural methyl ester oil be able to dissolve much water. Figure 2.8 shows the result of BDV test depends on temperature affected and Figure 2.9 shows the limitation of water saturation for mineral oil and methyl ester that functioning absolute the temperature [21].

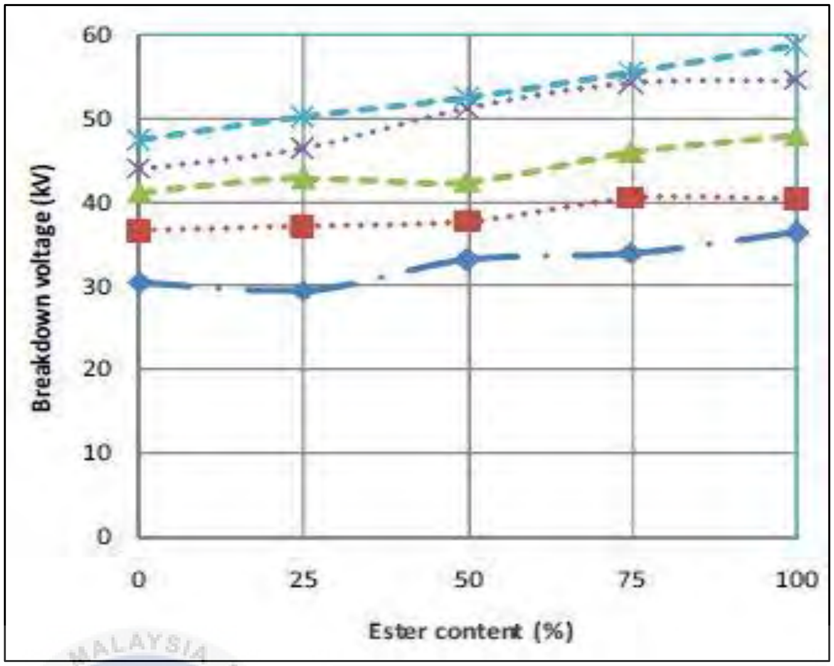


Figure 2.7: Dependence of BDV on the content of ester [21]

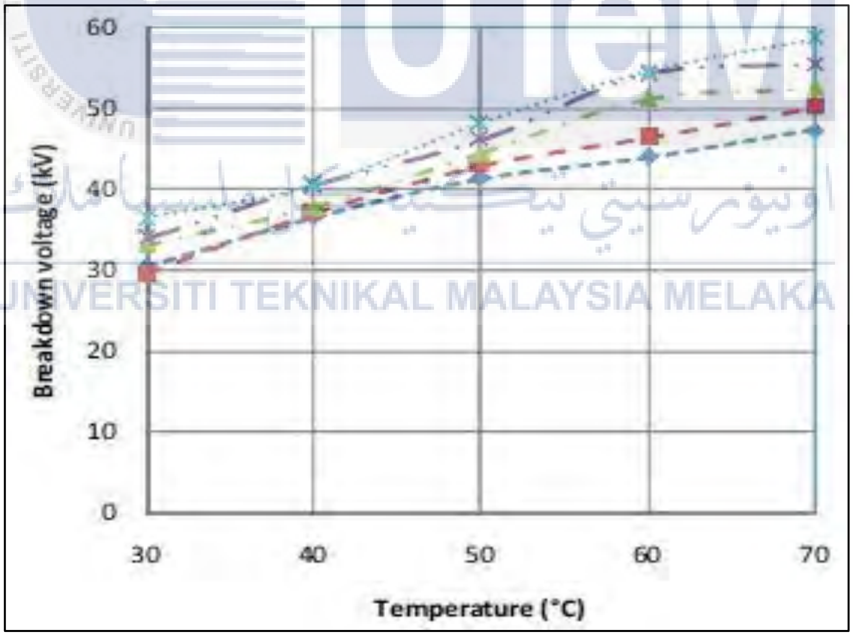


Figure 2.8: Dependence of BDV on temperature [21]

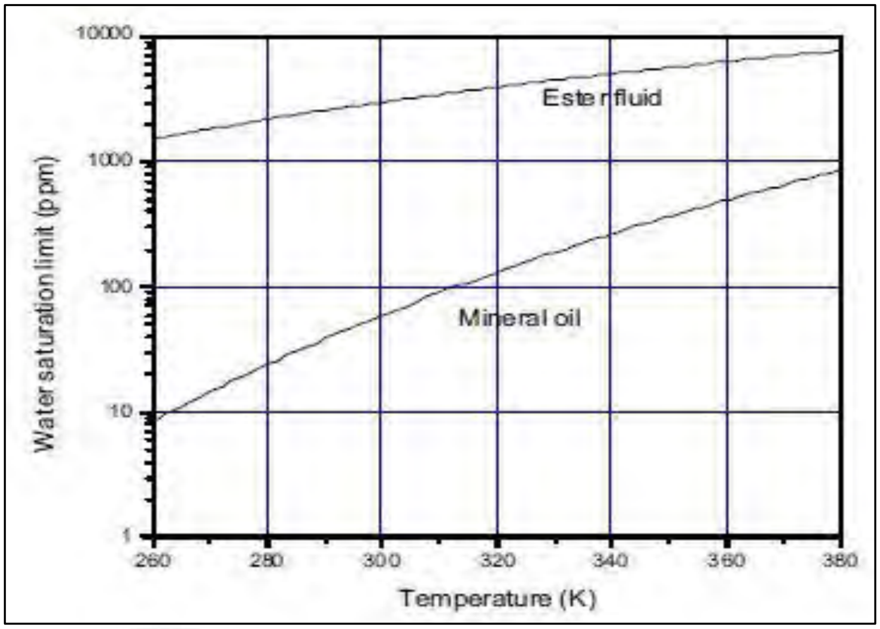


Figure 2.9: The limitation of water saturation for mineral oil and methyl ester that functioning absolute the temperature [21]

The BDV is increased significantly with the temperature at the range of temperature which is from 30°C to 70°C due to the water solubility is increased in the liquid. Equation (2.3) [21] states the temperature dependence of maximum water solubility in the liquid where W_o is in ppm unit and H is in Kelvin unit.

$$W_L = W_O \, exp\left(\frac{-H}{T}\right) \tag{2.3}$$

Table 2.6: The value of W_o and H for mineral oil and methyl ester oil [21]

Parameters **Types of Oil**	**W_o (ppm)**	**H (Kelvin)**
Mineral oil	$19.2x10^6$	3805
Methyl ester oil	$2.61x10^6$	1340

Table 2.6 shows the value of W_o and H for mineral oil and methyl ester oil [21]. Thus, it can be conclude that the ester content is the main factor in affected the properties of mixed insulating liquid between mineral oil and natural ester oil. With the increasing of ester content are be able to enhance the BDV test and 50% of ratio of concentration considered as suitable to be used in high voltage insulating liquid [21].

2.5 Corrosive Sulfur

Corrosive sulfur is defined as an elemental sulfur and thermally unstable sulfur compounds in insulating oil that can cause corrosion of certain transformer metals such as copper and silver [Appendix I]. Since 21st century, ABB, Doble, Toshiba, Areva, Siemens, Sea Marconi, and other factories begun to do the research in finding the solution to deal with corrosive sulfur problem in 2005 as well CIGRE was set up A2-32 with the same purpose [22]. The corrosive sulfur in transformer oil is originates mainly from the refining process of mineral oil (MO) where the process there were residual sulfur ingredients. It can be 5% or more for the content of the corrosive sulfur in transformer oil and sulfur was named in which can happened with the copper wire active sulfur. Copper sulfide (Cu_2S) is the main component deposition on the copper and insulation paper which has experienced the corrosion. In the transformer and the copper wire at 80℃ to 150℃, Cu_2s is the product formed in the reaction between the corrosive sulfides. Besides that, Dibenzyle-disulfide (DBDS) is pointed out that the main corrosive sulfide in the transformer oil. Meanwhile, the oxygen concentration in oils is take into consideration that affects the deposition of Cu_2S on the paper surface [10]. There have been a number of problems and failures of power transformer, shunt reactors, load tap changers and bushings that associated with corrosive sulfur in electrical insulating oil which is using mineral oils (MO) [23]. Besides that, transformer oil is not the only substance in a transformer which may contain sulfur. It could be happened from the gaskets, glues, copper and processing equipment probability contain sulfur as well [24]. Figure 2.10 illustrates the copper sulfide deposits on copper conductor [24].

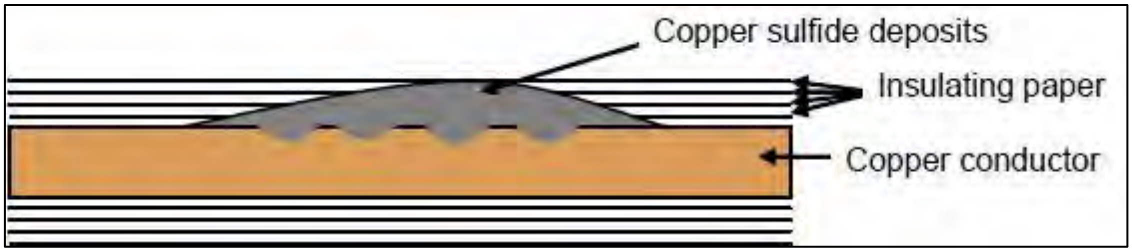


Figure 2.10: Copper sulfide deposits on copper conductor [24]

The testing for corrosive sulfur is usually referring to ASTM D1275 standard which is well known as standard test method for corrosive sulfur in electrical insulating oil. There are two (2) methods for ASTM D1275 standards which are Method A and Method B.

a. ASTM D1275A

ASTM D1275A also can be known as Method A for ASTM D1275 which represents the old parameter and this traditional test has been used for decades. By conducting this method, the oil is exposed to the copper strip and heated to temperature of 140°C for 19 hours in a sealed environment. In determine whether this test is pass or fail is by looking the amount of deposition and discoloration of the copper surface. IEC standards: DIN 51353 is one of the similar test with the Method A for ASTM D1275 which is the oil is exposed to silver strip and heated to temperature of 100°C for 18 hours but the experiment is conducted in an open air environment. It also use the same evaluation with ASTM D1275 which is by looking the amount of deposition and discoloration of the copper surface can be determined the test is pass or fail [24].

b. ASTM D1275B

ASTM D1275B is well known as Method B for ASTM D1275 and this method is the current edition of test method for copper corrosion procedure. Because of traditional test has made several case of transformers failing due to corrosive sulfur, thus it need ASTM D1275A to be replaced with ASTM D1275B. In 2006, the ASTM D1275B is a modified test and has been approved for test method for corrosive sulfur. This method also is similar to ASTM D1275A where the oil is exposed to copper conductor but the temperature to be heated is about 150°C for 48 hours. From this method can give more aggressive test where can enable to better identify corrosive sulfur in oil because of temperature is higher and duration is longer. Thus, some sulfur compounds may be breakdown and create more reactive compounds when exposed to intense heat through this method. Therefore, ASTM D1275B @ Method B for ASTM D1275 is recommended in doing test method for corrosive sulfur for all new transformer oil [24].

2.6 Flash Point

In general, flash point is defined as the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of a test flame causes the vapor of a specimen to ignite under specified conditions of test [Appendix I]. The flash point in simple word is the minimum temperature at which heated oil gives off sufficient vapor to form a flammable mixture with air and it is an indicator of the volatility of the oil. To assess the safety hazard of a liquid or semi-solid with regard to its flammability and then classify the liquid into a group is the fundamental reason for the requirement of flash point measurements is conducted. This classification is then used to warn of a risk and to enable the correct precautions to be taken when using, storing or transporting the liquid [25]. Basically, there is two (2) standard that usually used to perform the flash point test which are; ASTM D92 which known as Cleveland Open Cup Tester as shown in Figure 2.11 [26] and ISO 2719:2002 which known as Pensky-Martens Closed Cup as shown in Figure 2.12 [26, 28].

Figure 2.11: Cleveland open cup tester [26]

Figure 2.12: Pensky-Martens closed cup tester [26, 28]

By using Cleveland open cup method, the measuring flash point is conducted in a vessel where this method is exposed to the air outside. The temperature of the substance is gradually raised and an ignition source is passed over the top of it, until it reaches a point at which it flash and ignites. The precision of open-cup tests is somewhat poorer than closed-cup tests as the vapors produced by heating the test specimen are free to escape to the atmosphere and are more affected by local conditions in the laboratory. When open cup tests are carried out at temperatures above ambient the result is usually higher than a result from a closed cup test due to the reduced concentration of vapors [27, Appendix H].

Besides that, closed cup is mostly used in product specifications and regulations due to its better precision. ISO 2719:2002 standards using the Pensky-Martens closed cup tester for determining the flash point of combustible liquids, liquids with suspended solids, liquids that tend to form a surface film under the test conditions and other liquids. Thus, this standard is suitable be applied for liquids with a flash point above 40°C. Hence, there are two (2) procedures for ISO 2719:2002 standards which are; Procedure A is used for the determination of the flash point of paints and varnishes that do not form a surface film, unused lubricating oils and other petroleum products not covered by Procedure B. Meanwhile, Procedure B is used for the determination of the flash point of residual fuel oils, cutback bitumens, used lubricating oils, liquids that tend to form a surface film, liquids with suspensions of solids and highly viscous materials such as polymeric solutions and adhesives [28].

2.7 The Importance of Transformer Insulating Liquid Testing

2.7.1 Breakdown Voltage

The purpose of Breakdown Voltage (BDV) on insulating oil is to determine the ability of the insulating oil to withstand the electric stress without failure. The standard known that available and frequently used for BDV test on insulating oil which are; ASTM D1816, MS IEC 60156:2012 and ASTM D877 [29]. Megger Transformer Testing Equipment is very popular equipment that used to measure the BDV test on the insulating oil. There are a lot of transformer testing equipment by Megger for example Megger OTS60AF/2, Megger OTS80AF/2, Megger OTS100AF/2, Megger OTS80PB, Megger OTS60PB and etc. [30]. Figure 2.13 shows Megger OTS60PB that made from Megger [30]. Table 2.7 shows the details features of Megger OTS60PB [29, 30].

Figure 2.13: Megger OTS60PB [30]

Table 2.7**:** Details features of Megger OTS60PB [29, 30]

Specifications	**Megger OTS60PB**
Test Voltage	-30 to +30 Vrms / 60 kV (Max)
Operating Temperature	0℃ to 40℃
Operating Humidity	80% Relative Humidity at 40℃
Voltage Resolution	1 kV, ±1%, ±2 digits

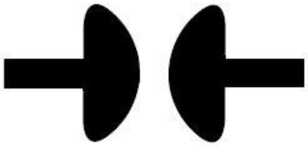
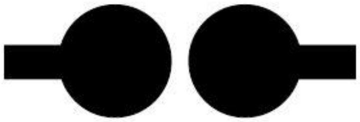
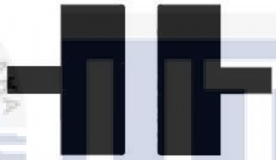
The Megger OTS60PB test set is a portable set and it is a fully automatic testing that has a battery. Table 2.8 shows the test specification parameters for ASTM D1816, ASTM D877 and MS IEC 60156:2012 [29].

Table 2.8: Test specification parameters [29]

Standards	**Electrode Shape**	**Electrode Spacing**	**Initial Stand**	**Rate Increasing of Test Voltage**	**Intermediate Stir Time**	**Number of Test**
ASTM D1816	A	1 mm, 2 mm	3 min	0.5 kV/s	Continuous	5
ASTM D 877	C	2.54 mm	2 min, 20 s	3 kV/s	-	5
MS IEC 60156: 2012	A, B	2.5 mm	3 min	2 kV/s	2 min	6

The electrodes of Megger as shown in Table 2.9 have four (4) types of electrodes shape which are; electrode shape A known as mushroom type, electrode shape B known as spherical type, electrode shape C and electrode D are known as cylindrical type.

Table 2.9: Differences of shape of oil testing electrodes [30]

Types of Electrode	Figures of Electrode Shape	Description
Electrode Shape A		• Mushroom / semispherical • 36 mm diameter
Electrode Shape B		• Spherical • 12.7 mm diameter
Electrode Shape C		• Cylindrical • 25.4 mm diameter
Electrode Shape D		• Cylindrical • 25.44 mm diameter with 0.5 mm edge radius

2.7.2 Moisture

Moisture or known as water (H_2O) is one of the contaminant and enemy that can affect the dielectric properties of insulating oil. Based on Naidu et al. [31] stated that the maximum amount of water present in the oil of a good quality is less than 50 ppm at an atmospheric temperature which about 30°C or less. Usually, certain amount of moisture which is happened from water vapor and a very little amount of water will present in the insulating oil when the oil temperature is decreased. The water content is measured in parts per million (ppm) or mg/l. There are three (3) stages of water can be exist in oil which are; dissolved water, emulsified water, and free water. For the stage of dissolved water, the molecules of water are dispersed alongside oil molecules when the saturation level is below and thus resulting in water in the oil become not visible. Meanwhile, for the stage of emulsified water, the oil is no longer able to absorb more water molecules when the amount of dissolved water exceeds the saturation point. This characterized by a hazy or cloudy that appear of the oil. For the last stage which is known as free water, further increments of water content in oil will result in separate levels between oil and water forming of its higher density, the water forms the lower layer, settling at the bottom of the sump, with the oil floating on top. Figure 2.14 shows the dissolved water, emulsified water and free water [32].

Figure 2.14: Dissolved water, emulsified water and free water [32]

To measure the moisture content in insulating oil is divided into two types of off-line methods which are Karl Fischer reaction method and the pressure gauge method. The Karl Fischer reaction test is selected to be used in this study as shown in Figure 2.15 [33]. ASTM D 1533 test known as Karl Fischer titration method have been used from many previous researchers and this standard test method used to measure the water content in the insulating oil. By using this standard, it is based on the reaction of halogens with water in the presence of anhydrous sulfur dioxide. In addition, high selectivity and sensitivity make this standard is very popular in doing the test of moisture content in oil. [34].

Figure 2.15: Metrohm Coulometric Karl Fischer Titration [33]

2.8 Weibull Distribution

The Weibull distribution provide the best fit of life data and simple graphical solution. The preference of using Weibull because it can provide a reasonable accurate failure forecast and failure analysis with extremely small samples in which the small samples are allowed cost effective component testing.

The mathematical of Weibull distribution can be defined as follows:

$$F(t) = 1 - e^{(t/n)^{\beta}} \tag{2.4}$$

Where;

$F(t)$ = Fraction failing

t = Failure time

n = Characteristic life or scale parameter

β = Slope or shape parameter

From the equation (2.4) [35] can be stated that $F(t)$ is always 63.2% when t is equal to n and it does not matter what the value of β is. This tells that the unique feature of the characteristic life is that it occurs at the 63.2% regardless of the Weibull distribution. Thus, it can be proved that with the Weibull equations which is:

$$\begin{aligned} F(t) &= 1 - e^{(t/n)^{\beta}} \\ &= 1 - 0.368 \\ F(t) &= 0.632 \text{ regardless the value of } \beta \end{aligned}$$

This Weibull statistical technique can be applied after getting the data of AC breakdown voltage for lower failure probabilities at 63.2% in order to estimate the breakdown voltage on the insulating oil that tested. In fact, the Weibull distribution can gives better fits than normal distribution [35].

2.9 Review of Previous Related Works

The focus in this study is more on enhancing the breakdown voltage (BDV) of mineral oil (MO) mixed with natural ester oil (NEO). There is some research conducted by some researchers that related in this study before this study is implemented.

Table 2.10: Paper review on enhanced the BDV of mixed insulating liquid

Author (year)	Paper Review	Findings	Ref
C. Perrier (2006)	• Improvement the characteristic of mineral oil by mixing with synthetic ester • Using Naphthenic as the type of mineral oil and 2 types of synthetic ester in mixture which are; Tetraesters and Pentaerythritol • Other insulating liquid used in mixture which is silicon oil based on polydimethylsiloxane • This paper reports the comparison of heat transfer, BDV, ageing stability and electrostatic charging tendency (ECT)	• Mixture between 80% of mineral oil and 20% of synthetic ester is the good compromise as better mixed insulating liquid and improve the BDV average value of mixture between mineral oil and synthetic ester	[20]
I. S. Darma (2008)	• This paper focused on development of dielectric properties of mixtures between mineral oil and natural ester oil • The mixtures used conventional mineral oil which is Shell Diala B and methyl ester as natural ester oil • The mixtures conducted from 0% to 100% of concentration of ester	• The ester content is strongly affected the properties of mixture between mineral oil and natural ester oil • The increase of ester content in mixtures gives the result of higher acidity, losses factor and enhanced the dielectric breakdown • 50% of ester content considered as the best result of ratio of concentration in mixture	[21]

P. Krishna kumar (2014)	• This paper reports the enhancing of critical parameters which are BDV, flash and fire point of mixed insulating liquid with nano materials and antioxidants. • The investigation of mixture used many types of insulating oil which are; mineral oil (MO), used transformer oil (UTO), sunflower oil (SFO), rice bran oil (RBO) and silicone oil (SO)	• Sample 3 presents mixture between 75% of mineral oil and 25% of sunflower oil gives the highest BDV and low viscosity at higher temperature compared to other mixture samples • Sunflower oil basically based on the natural ester oil	[36]

2.10 Summary and Discussion of the Review

In this chapter, the development and the details types of insulating liquid that currently used in the power transformer as complies with their standard have been discussed. The types of insulating liquid that commonly used are mineral oil (MO) and natural ester oil (NEO) where Palm Fatty Acid Ester (PFAE) oil is usually used among other natural ester oil in nowadays. However, this study is focusing on mixed insulating oil between MO and NEO in order to enhance the breakdown voltage (BDV). In reaching the objective of this research, there is needed a detailed study to find the best ratio of the concentration of MO and NEO by referring to the previous related works in terms of dielectric properties, physical properties, chemical properties, moisture measurement, and etc.. Previous research shows that with the adding of ester content about 20% will give a good compromise and improve the BDV. Thus, the investigation on the mixture of MO and NEO with using the suggestion of ratio given is required in proving the fact including the investigation on differences of the ratio of concentration from 0% to 100% of MO and NEO. Besides that, the content of moisture in the insulating oil will affects the result of BDV. Hence, the corrosive and flash point as the last test process that conducted on the best ratio of mixed insulating oil for identifying the physical and chemical properties for that mixed insulating oil. The next chapter will presents the research methodology used in this study.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter will describe the process involved that will be taken in order to achieve the project objectives and scope. To make the project systematically in procedures, flowchart is illustrated the project implementation.

3.2 Flowchart of Project Implementation

The project implementation is described based on the Figure 3.1. The process involved in this project is divided into several parts which starting from selecting the mineral oil (MO) and natural ester oil (NEO) and ending up with analysis the result. Besides that, the previous research for methods or procedures from previous researchers that have been done are related and will be applied in the case of this study. However, the type of natural ester oil used to be mixed with mineral oil for this study is the main differences from other previous research.

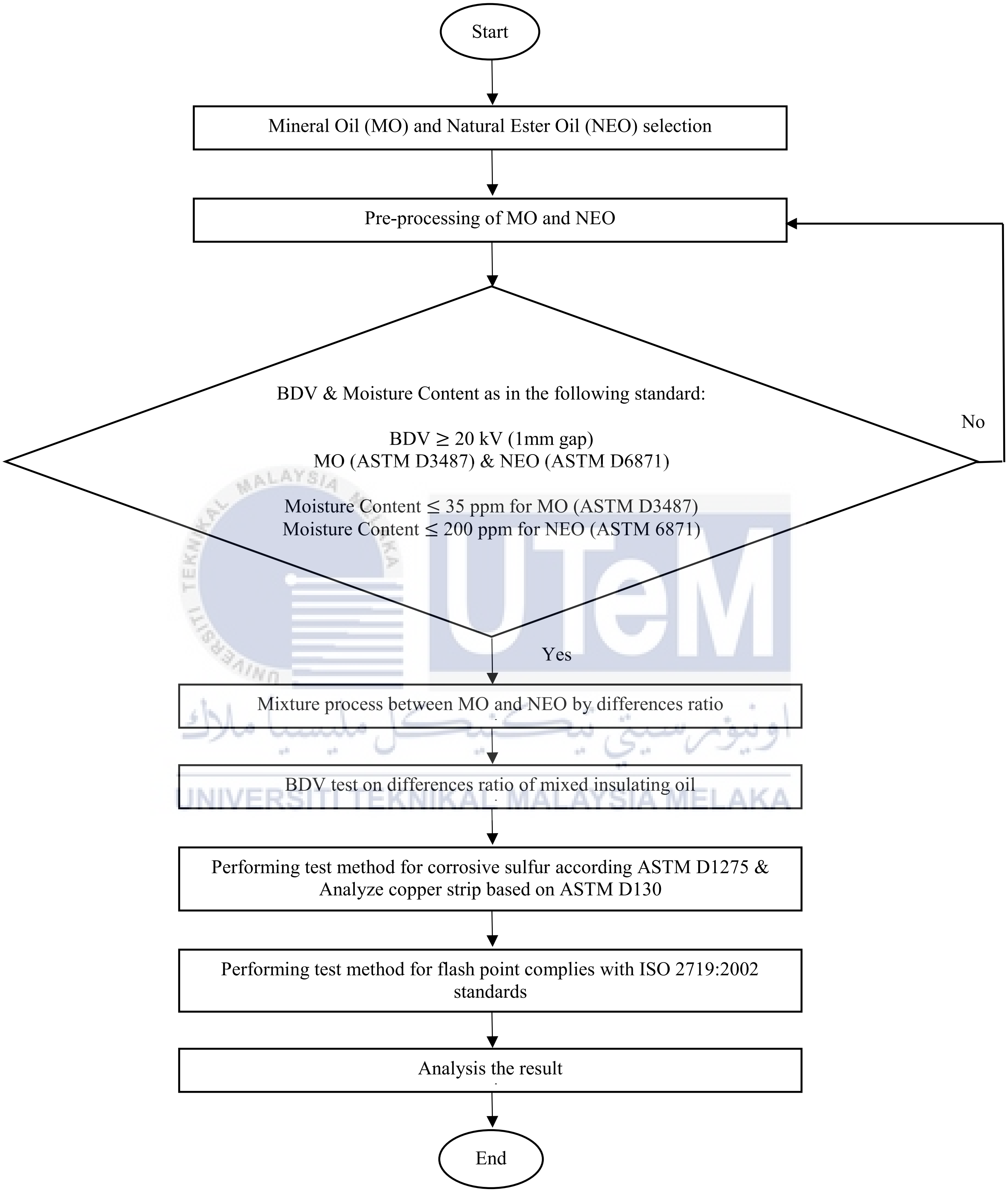


Figure 3.1: Flowchart of project implementation

3.2.1 Mineral Oil (MO) and Natural Ester Oil (NEO) Selection

The mineral oil (MO) that be used in this experiment is Nytro Libra (IEC 60296, Ed. 4) which the properties is uninhibited mineral oil that sourced from Nynas AB (M), Sweden [37] and for natural ester oil (NEO) that be used is Palm Fatty Acid Ester (PFAE) oil sourced from the Lion Corporation, Japan.

Figure 3.2:

Blue barrels which written of Nynas is containing Nytro Libra oil and green barrel is containing PFAE oil

3.2.2 Pre-processing of Mineral Oil (MO) and Natural Ester Oil (NEO)

In this process section, it is very important to mineral oil (MO) and natural ester oil (PFAE oil) to undergo the pre-processing in order to get both in normalized properties before used in the whole experiment. Thus, this second stage of project implementation is required for filtering insulating liquid and standardized to keep the insulation oil purity and composition as well as with parameters of temperature, electric field, electrical strength and water content [38]. The insulating oil filtering treatment method will be performed at the beginning of the experiment. Then, the two (2) types of test will conducted which are; water content and BDV test as complies with the standard will be conducted on MO and PFAE oil for the purpose for keeping both insulating oil in fresh oil.

3.2.2.1 Insulating Oil Filtering Treatment Method

The insulating oil filtering treatment method is as follows:

1. Insulating oil is pumped out from the barrel into a beaker and the beaker is wrapped with plastic for prevent the moisture of insulating oil increased.
2. The apparatus of vacuum pump, filter flask, fritted funnel glass, fritted glass disk, clamper and filter paper with approximately 0.5 cm are set up as in Figure 3.3.

Figure 3.3: Set up the apparatus for filtering treatment process

3. The insulating oil is poured into the fritted funnel glass until the maximum level. Then, the fritted funnel glass is wrapped with plastic to prevent the moisture or unwanted contaminants entered into the insulating oil during filtering process occurred.
4. The switch of vacuum pump is turned on and the process is starting to suck out the air in the filter flask and the insulating oil is starting to filtered.
5. The insulating oil is refill into fritted funnel glass and the process is repeated until the amount of insulating oil want to be used is completed to be filtered.

3.2.2.2 Water Content Test

The water content test on MO and PFAE oil are complies with ASTM D1533 standards using Coulometric Karl Fischer Titration [33, 39]. The procedure to conduct the water content test is as follows:

1. STOP button is pressed to switch on KFC as in Figure 3.4.

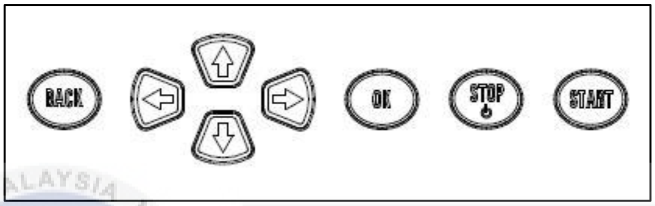


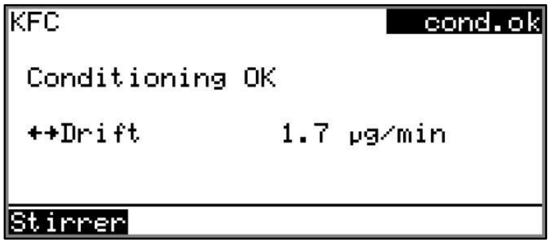
Figure 3.4: Instrument's keypad

2. The desired method (KFC) is selected as in Figure 3.5

```
>Menu                    ready
 Method                    KFC
 ID1
 ID2
 Sample size               1.0
 Unit                        g
```

Figure 3.5: Method selection

3. START button is pressed to start conditioning and waiting until the screen is display "Conditioning OK" as in Figure 3.6.



```
KFC                   cond.ok

 Conditioning OK

 ++Drift         1.7 µg/min

Stirrer
```

Figure 3.6: KFC is in a ready condition

4. A syringe with needle will be loaded with 5 ml of oil sample that need to be tested as in Figure 3.7. For this stage, ensure there is no bubble presence in the syringe.

Figure 3.7: 5 ml of oil sample loaded into a syringe with needle

5. Digital analytical balance is used for weighed the oil sample as shown in Figure 3.8. Hence, the size of oil sample will be used later.

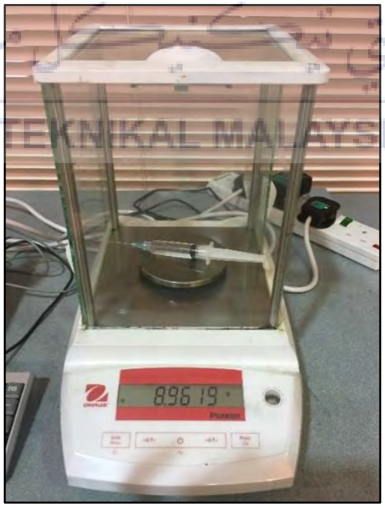
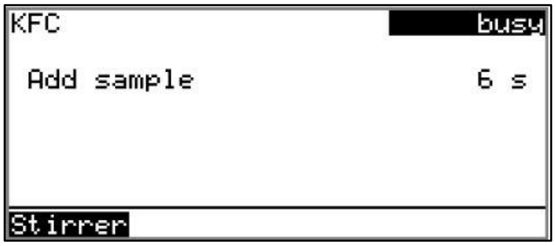


Figure 3.8: Weighing the sample inside syringe

6. Start button is pressed after the screen displayed “Conditioning OK”. Then, oil sample is added as in Figure 3.10 when the screen displayed as in Figure 3.9.



```
KFC                         busy
 Add sample                  6 s

Stirrer
```

Figure 3.9: KFC Screen displayed add sample

Figure 3.10: Oil sample is added

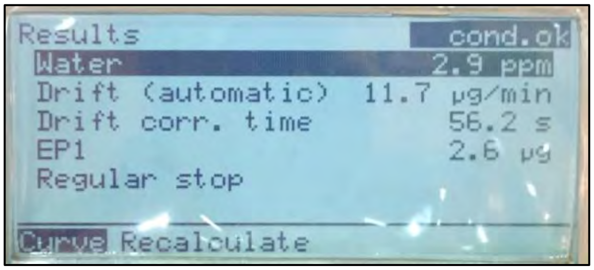
7. Hence, the screen displayed oil sample size which need to be entered as in Figure 3.11.

```
KFC                         hold
 Sample size                 1.0

 Press [START] key to continue
```

Figure 3.11: Sample size is inserted

8. The size of oil sample after weighed by using digital analytical balance is entered and OK button is pressed. Then, START button is pressed to start the titration.
9. The results dialog displayed as shown in Figure 3.12 after the titration is completed.



```
Results                     cond.ok
 Water                      2.9 ppm
 Drift (automatic)   11.7 µg/min
 Drift corr. time          56.2 s
 EP1                        2.6 µg
 Regular stop

Curve Recalculate
```

Figure 3.12: Results dialog displayed the moisture content of the oil sample

10. To ensure the accuracy of the results, the process is repeated for another four oil samples.

3.2.2.3 BDV Test

Breakdown voltage (BDV) test will be performed after the water content test on MO and PFAE oil are conducted. The BDV test on MO and PFAE oil are complies with ASTM D1816 standards using Megger OTS60PB as in Figure 3.13 where the type of VDE electrode used is electrode shape A and the electrode spacing is set to 1 mm.

Figure 3.13: Megger OTS60PB

. The procedure to conduct BDV test is as follows:

1. Oil sample is filled into the vessel where level mark for oil sample is about 500 ml. Ensure there is no formation of bubble in the oil sample and check the temperature of oil sample is at room temperature which is 20°C to 30°C as in Figure 3.14.

Figure 3.14: Measure initial temperature of oil sample

2. The gap spacing of VDE electrode shape A is set to 1 mm as in Figure 3.15.

Figure 3.15: 1 mm of gap spacing of VDE electrode

3. Oil sample is placed in the test chamber as in Figure 3.16.

Figure 3.16: Place the oil sample in the test chamber

4. Megger OTS60PB is switched ON after the chamber cover is closed.
5. Then, ASTM D1816 standard is selected from the main menu as in Figure 3.17.

Figure 3.17: Selection standard from main menu

6. The screen of Megger OTS60PB will display as in Figure 3.18 to confirm the standard and START button is pressed to proceed the next process.

Figure 3.18: Standard confirmation and gap spacing used

7. Next, Megger OTS60PB started the initial stirrer process for 3 minutes as in Figure 3.19. After completed 3 minutes, Megger OTS60PB is readily for the dielectric strength testing as in Figure 3.20.

Figure 3.19: 3 minutes required for initial stirrer process

Figure 3.20: BDV testing is in progress

8. The process in step 5 is repeated to get 30 data of BDV test for each sample for the purpose of large sample of data can give better accuracy.
9. Hence, calculation (3.1) is used for calculation of the mean for 30 data of BDV test for each oil sample.

$$Mean\ BDV = \frac{1}{30}\sum_{i=1}^{30} X_i \tag{3.1}$$

3.2.3 Insulating Oil Following Standard

The standard measurement for mineral oil (MO) and natural ester oil (PFAE oil) that be used in this experiment are ASTM D3487 and ASTM 6871. Table 3.1 shows the comparison of standard properties for both insulating oil in order to ensure to get the best result when conducting the experiment [40]. The maximum water saturation permissible for fresh mineral oil according to ASTM 3487 is 35 ppm while natural ester oil according to ASTM 6871 is 200 ppm. Thus, ASTM D1533 or known as Karl Fischer Titration method is required to test the water content for both insulating oil. Hence, Breakdown Voltage (BDV) test with complies of standard ASTM D1816 will be performed in ensuring the mineral oil and PFAE oil reach their standard for dielectric strength properties.

Table 3.1: Comparison of standard properties for fresh mineral oil and natural ester oil [40]

Properties			**ASTM Method**	**Mineral Oil ASTM D3487**	**Natural Ester ASTM D6871**
Electrical	Dielectric strength (kV)		D1816 (1mm gap)	≥20	≥20
			D1816 (2mm gap)	≥35	≥35
	Dissipation (%)	25 (°C)	D924	≤0.05	≤0.02
		100 (°C)	-	≤0.30	≤4.0
Chemical	Water content (ppm)		D1533	≤35	≤200
	Acid number (mg KOH/g)		D974	≤0.03	≤0.6
Physical	Color		D1500	≤0.5	≤1.0
	Visual examination		D1524	clear & bright	clear & bright
	Relative density		D1298	≤0.91	≤0.96
	Flash point (°C)		D92	≥145	≥275
	Fire point (°C)		D92	-	≥300
	Viscosity	100 (°C)	D445	≤3.0	≤15
		40 (°C)	-	≤12	≤50
Pour point (°C)			D97	≤-40	≤-10
Interfacial tension (dyne/cm)			D971	≥40	-

3.2.4 Mixture Process between MO and NEO by Differences Ratio

The experiment will be carried out with various samples of mixed insulating oil. Each sample has differences ratio of concentration of mineral oil (MO) and PFAE oil. Table 3.2 shows 11 samples with differences ratio of concentration of mixed oil will be tested. In this experiment, 500 ml is selected as based quantity of sample for mixed oil process. Hence, the mixture process between mineral oil (MO) and PFAE oil will be used magnetic stirrer of 400 rotations per minute (RPM) with temperature level is 90°C and time taken for mixture process to complete is about 1 hour [41].

Table 3.2: Ratio of concentration of mixed insulating oil

Samples	Types of Sample
1	100% MO + 0% PFAE
2	0% MO + 100% PFAE
3	90% MO + 10% PFAE
4	80% MO + 20 % PFAE
5	70% MO + 30 % PFAE
6	60% MO + 40% PFAE
7	50% MO + 50% PFAE
8	40% MO + 60% PFAE
9	30% MO + 70% PFAE
10	20% MO + 80% PFAE
11	10% MO + 90% PFAE

3.2.4.1 Mixture Process Procedure

1. A beaker with volume 1000 ml will be used for entire the process
2. Mineral Oil (Nytro Libra oil) is filled into the beaker including the magnetic stirrer rod as in Figure 3.21.

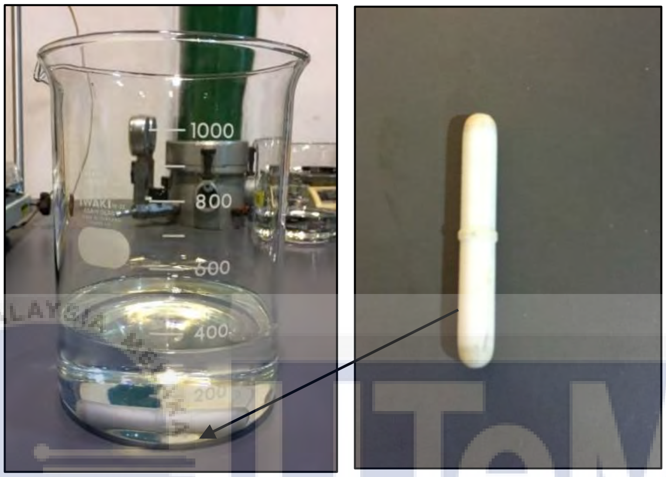


Figure 3.21: Magnetic stirrer rod and MO are filled into the beaker

3. The beaker is put on the plate of magnetic stirrer equipment as in Figure 3.22.

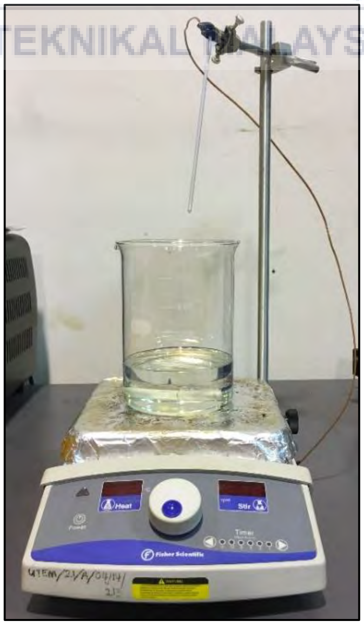


Figure 3.22: Placing the beaker on the plate of magnetic stirrer equipment

4. Rod of temperature is set down to the beaker and ensure the rod does not touch body of the beaker as in Figure 3.23.

Figure 3.23: Set down the rod of temperature to the beaker

5. The temperature is set to 90°C and the speed is set to 400 RPM as in Figure 3.24.

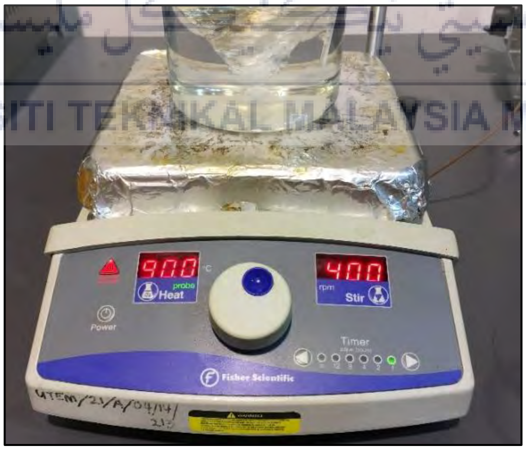


Figure 3.24: Temperature and speed are set to 90°C and 400 RPM

6. After the temperature is reached to 90°C, PFAE oil is started to fill into the beaker and the time is set to 1 hour as in Figure 3.25.

Figure 3.25: PFAE oil is poured into the beaker when temperature is reached 90°C

7. When approaching 1 hour of mixed process, the temperature of oil sample will be measured during the notification of the first beep sound as in Figure 3.26.

Figure 3.26: Taking measurement of oil sample temperature before the mixed process ended

8. After completed 1 hour, the oil sample is filled into the bottle and waiting about 5 minutes to cooling down the sample as in Figure 3.27.

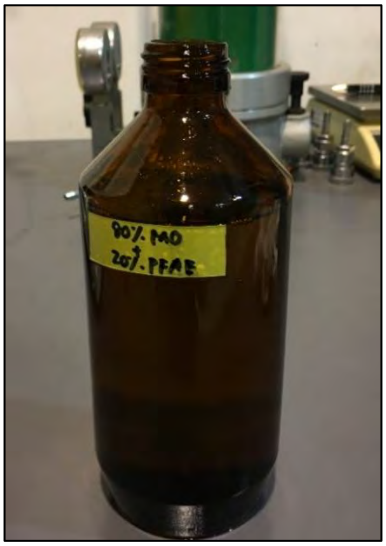


Figure 3.27: Oil sample is filled into the bottle and 5 minutes taken for cooling down process

9. The bottle is blanketed with nitrogen gas and kept in the dark places for overnight as in Figure 3.28.

Figure 3.28: The bottle kept in the dark places for overnight after blanketed with nitrogen gas

3.2.5 BDV Test on Differences Ratio of Mixed Insulating Oil

After completed preparing oil sample for all ratio of concentration of mixed oil, the BDV test is carried out for all of oil sample. Before BDV test is conducted, oil sample for all ratio of concentration of mixed oil are going through the procedure as follows:

1. Oil sample that kept in the bottle which blanketed with nitrogen for overnight is poured into conical flask (Erlenmeyer flask) as shown in Figure 3.29.

Figure 3.29: Pour oil sample into conical flask

2. Oil sample is undergoing water content as in Chapter 3.2.2.1 and the result of water content is recorded.
3. Bubble nitrogen through oil sample in conical flask for 15 minutes as in Figure 3.30.

Figure 3.30: 15 minutes for bubble nitrogen through oil sample in conical flask

4. Then, the test of water content as in Chapter 3.2.2.1 is repeated and the result of water content is recorded.
5. Oil sample is poured into vessel as in Figure 3.31 and BDV test is performed with the same procedure as in Chapter 3.2.2.2.

Figure 3.31: Pour oil sample into vessel

3.2.6 Performing Test Method for Corrosive Sulfur

The test method to detect corrosive sulfur in insulating oil is performed using Method B from ASTM D1275 standards. Based on ASTM D1275 standard, 250 ml non-filtered oil is selected as based quantity of oil sample and copper strip with measurement 6 mm x 25 mm are used in test method for corrosive sulfur process For this experiment, Method B where the main aging parameters about 48 hours at 150°C are applied as test method for corrosive sulfur that conducted on oil sample with ratio of 100% MO, 100% PFAE, 80% MO+20% PFAE and 70% MO+30% PFAE,.

The procedure to conduct test method for corrosive sulfur is as follows:

1. Copper foil as in Figure 3.32 is cut into measurement 6 mm x 25 mm as in Figure 3.33.

Figure 3.32: Copper foil

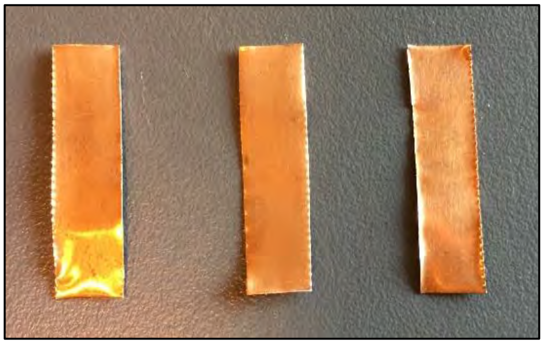


Figure 3.33: Copper strip with measurement 6 mm x 25 mm after being cut

2. 240-grit silicon carbide paper (sandpaper) is used for rubbed both surface of copper strip 6 mm x 25 mm as in Figure 3.34.

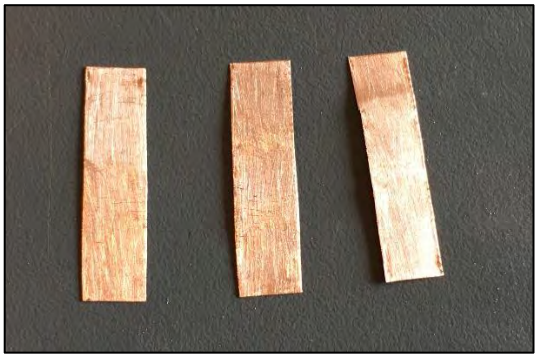


Figure 3.34: Copper strip 6 mm x 25 mm after rubbed with sandpaper

3. Then, copper strip 6 mm x 25 mm is polished with pad of absorbent cotton moistened with a drop of acetone as in Figure 3.35.

Figure 3.35: Polished copper strip with cotton that moistened with acetone

4. Copper strip is wiped with fresh pads of cotton for clean all metal dust and abrasive.
5. Then, copper strip is bent into V-shape at approximately 60° angle as in Figure 3.36 and wash successively in acetone, distilled water, and acetone as in Figure 3.37 and Figure 3.38.

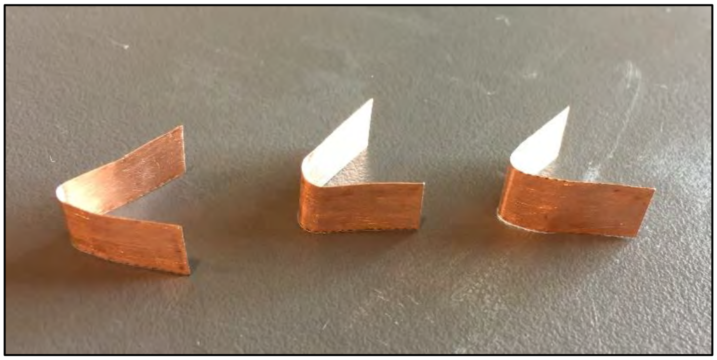


Figure 3.36: Bend copper strip into V-shape at approximately 60° angle

Figure 3.37: Copper strip V-shape washed with acetone

Figure 3.38: Copper strip V-shape washed with distilled water

6. In the meantime, 220 ml of oil sample non-filtered are filled in heavy-walled bottle 250 ml as in Figure 3.39.

Figure 3.39: Heavy-walled bottle 250 ml are filled with 220 ml of oil sample non-filtered

7. Hence, copper strip V-shape is dried in oven for 3 to 5 minutes at 80°C to 100°C as in Figure 3.40 and copper strip V-shape is immediately immersed in heavy-walled bottle that filled with oil sample. Ensure that copper strip V-shape standing on its long edge so that no flat surface lies along the glass bottom.

Figure 3.40: Dry copper strip V-shape in oven for 3 to 5 minutes at 80°C to 100°C

8. Bubble nitrogen through oil sample in the heavy-walled for 5 minutes as in Figure 3.41 and quickly screw with the PTFE plug equipped with fluoro-elastomer O-ring.

Figure 3.41: 5 minutes for bubble nitrogen through oil sample in heavy-walled bottle

9. Heavy-walled bottle are placed in the oven at 150°C as in Figure 3.42. If needed, after 15 minutes of heating at 150°C, partially unscrew the PTFE plug to release the pressure and then screw it back down. Heating heavy-walled bottle in the oven for 48 hours ± 20 minutes at 150 ± 2°C as in Figure 3.43.

Figure 3.42: Placed heavy-walled bottle in the oven at 150°C

Figure 3.43: Heating heavy-walled bottle about 48 hours ± 20 minutes at 150 ± 2°C

10. After completed for heating for 48 hours at 150°C, remove the bottle from the oven and allow to cool down.
11. Copper strip V-shape is taken out from the heavy-walled bottle with carefully as in Figure 3.44.

Figure 3.44: Carefully take out copper strip from heavy-walled bottle

12. Copper strip V-shape washed with acetone to remove all of oil sample and let air dry.
13. Observed by hold the copper strip in such manner so that light reflected from it an angle of approximately 45°
14. ASTM D130 standards as in Figure 3.45 is used for interpretation of results.

Figure 3.45: Copper strip tarnish level classification, test method ASTM D130 standards

15. If the specimen is borderline or unclear which means between noncorrosive and corrosive, the strip is cut in half along the bent portion of the copper strip.
16. Retain one half and take other half of copper strip to submerge completely in a 1:1 solution of hydrochloric acid (HCl) and distilled water at room temperature and wait 20 ± 2 minutes while gently swirling on occasion as in Figure 3.46.

Figure 3.46: Submerge completely copper strip in 1:1 solution of HCl and distilled water

17. If the deposit in question is still present and did no fade or discolor, then the result is reported as corrosive. Meanwhile, if the deposit has been removed or has turned tan/beige, then corrosive sulfur is not present and the result is reported as noncorrosive.

3.2.7 Performing Test Method for Flash Point

In this part, the test method for flash point is performed according ISO 2719:2002 standards using Pensky-Martens closed cup method. Based on ISO 2719:2002 standard, 500 ml non-filtered oil is selected as based quantity of oil sample and Pensky-Martens closed cup tester as in Figure 3.47 are used throughout this experiment [26, 28]. Oil sample with ratio 80% MO+20% PFAE and 70% MO+30% PFAE are selected in flash point test that conducted.

Figure 3.47: Pensky-Martens closed cup tester [26, 28]

The procedure to conduct test method for flash point is as follows:

1. Barometer is set the accuracy to 0.1 kPa. At the test of time, ambient barometric pressure in the vicinity of the apparatus is recorded.
2. Test cup is filled with the test portion to the level indicated by the filling mark. The lid is placed on the test cup and put in the heating chamber. Ensure that the locating or locking device is properly engaged and the thermometer is inserted.
3. The test flame is light up and it adjusted to a diameter of 3mm to 4mm, or the alternative ignition source is switched on. Heat flame is light up or the electric heater and supply heater are switched on at such a rate that the temperature of the test portion as indicated by the thermometer increases at 5°C/min to 6°C/min. This heating rate is maintained

throughout the test. The test portion is stirred in a downward direction at a rate 90 r/min to 120 r/min.

4. When the test portion is expected to have a flash point of 110°C or below, the application of the ignition source is made first when the temperature of test portion is 23°C ± 5°C below the expected flash point and thereafter 1°C intervals. Stirring is ceased and the ignition source is applied by operating the mechanism on the cover which controls the shutter and ignition source so that the source is lowered into the vapour space of the test cup in 0.5 second, left in its lowered position for 1 second and quickly raised to its high position.
5. When the test portion is expected to have a flash point of 110°C or below, the first application of the ignition source is made when the temperature of test portion is 23°C ± 5°C below the expected flash point and thereafter 2°C intervals. Stirring is ceased and the ignition source is applied by operating the mechanism on the cover which controls the shutter and ignition source so that the source is lowered into the vapour space of the test cup in 0.5 second, left in its lowered position for 1 second and quickly raised to its high position.
6. Preliminary test is conducted at a suitable starting temperature when testing a material of unknown flash point. The first ignition source application at 5°C above the starting temperature is made and the procedure as in procedure (4) and procedure (5) are followed.
7. The flash point is observed and recorded on the temperature of test portion read on the thermometer at the time when the ignition-source application causes a distinct flash in the interior of test cup. Ensure does not confuse the true flash point with the bluish halo that sometimes surrounds the ignition source at applications preceding the actual flash point.
8. The result is not valid if the temperature at which the flash point is observed is less than 18°C or greater than 25°C from the temperature of the first application of the ignition source.

9. The test using a fresh test portion is repeated with adjusting temperature of the first application of the ignition source until a valid determination is obtained which is the

flash point is 18°C to 28°C above the temperature of the first application of the ignition source.

10. Lastly, correction of observed flash point to standard atmospheric pressure is calculated using equation (3.2)

$$Tc = To + 0.25\,(101.3 - P) \tag{3.2}$$

where

To is the flash point at ambient barometric pressure in (°C)

P is the ambient barometric pressure in (kPa)

3.2.8 Analysis the Result

The process of analysis the result will be conducted at the last session of experiment after gathered the data and information. The data will be analyzed to determine which ratio of concentration of mixed oil will be selected through the test of water content, BDV, corrosive sulfur and flash point. Thus, the analysis of the experiment should relate to objective in this study and finally conclusion can be made from it.

3.3 Summary

Overall, Nytro Libra oil represents as mineral oil (MO) and Palm Fatty Acid Ester (PFAE) oil represents as natural ester oil (NEO) are selected in this study. The selected method for conducting the experiment is based on research background that has been done. There are 11 samples for ratio of concentration of mixed oil that need to be carried out using the mixture process from previous research method. Hence, oil sample which has been selected is need to undergo the further test such as corrosive sulfur test and flash point test after going through the water content test and BDV test to ensure that oil sample is presents the best ratio for the concentration of mineral oil mixed with natural ester oil. In the next chapter will presents the results and discussion that obtained based on the experiments that have been done.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

In this chapter will be discussed and analyzed the recorded data based on the experiment that has been done. The experiment that successfully conducted which are; breakdown voltage (BDV), moisture content, corrosive sulfur and flash point are comply with the standard in order to fulfill the objectives of this study. The mineral oil (MO) used in this experiment is Nytro Libra which the characteristic is uninhibited MO while Palm Fatty Acid Ester (PFAE) oil is selected to be used as natural ester oil (NEO). The details discussion for analysis the result is as follows.

4.2 Project Achievement and Analysis

The data that collected and recorded are based on the laboratory experiment. The results in this experiment are cover the experimental of moisture content and BDV of fresh MO and fresh PFAE oil, moisture content and BDV of all samples of ratio of concentration of mixed insulating oil, corrosive sulfur and flash point on insulating oil that selected. Following are the analysis of the experimental result.

4.3 Results of the Fresh MO and Fresh NEO

4.3.1 Moisture Content for Fresh MO and Fresh PFAE Oil

In initial stage for the process of experiment procedure, Coulometric Karl Fischer Titration according to ASTM D1533 standards is used for conducted of moisture content test. The result of moisture content for fresh MO before and after filtering treatment are recorded in Table 4.1 and Table 4.2.

Table 4.1: Result of moisture content test for fresh MO before filtering treatment

Types of Samples	No. of Test Conducted	Sample Size (g)	Moisture content (ppm)	Average (ppm)
Fresh MO	Test 1	0.9627	37.6	37.3
	Test 2	0.8917	38.4	
	Test 3	0.8847	35.8	

Table 4.2: Result of moisture content test for fresh MO after filtering treatment

Types of Samples	No. of Test Conducted	Sample Size (g)	Moisture content (ppm)	Average (ppm)
Fresh MO	Test 1	0.9647	27.2	28.4
	Test 2	0.8922	28.8	
	Test 3	0.8907	29.1	

From the result obtained for both Table 4.1 and Table 4.2, it can be seen that there are three (3) reading have been conducted for fresh MO before and after filtering treatment and the average value is calculated to ensure data recorded are accurate and complies with the standard. For Table 4.1, the average value of moisture content for fresh MO before filtering treatment which means the condition of MO after taking out from the barrel is 37.3 ppm. This result of moisture content shows that is not complies with the ASTM D3487 standard for MO in which the value of moisture content must be ≤35 ppm. Since MO has lowest moisture content, this can be justify MO is easily to absorb the water from the surrounding even though MO is kept into the barrel. Thus, insulating oil filtering treatment is performed to treat the MO and to remove the unwanted substances but at the same time it can be reduced the moisture content in MO. From the result shows in Table 4.2, the average value of moisture content for fresh MO after filtering treatment is 28.4 ppm after conducted the three test of moisture content. Hence, the result of moisture content after filtering treatment is meet the requirement and conditions for ASTM D3487 standards.

Hence, the result of moisture content for fresh PFAE oil has been recorded in Table 4.3 and the test conducted is three times to get average value of moisture content.

Table 4.3: Result of moisture content test for fresh PFAE oil before filtering treatment

Types of Samples	**No. of Test Conducted**	**Sample Size (g)**	**Moisture content (ppm)**	**Average (ppm)**
Fresh PFAE	Test 1	0.4537	89.5	82.7
	Test 2	0.4411	82	
	Test 3	0.4073	76.7	

From the ASTM D6871 standards that used for references of NEO standard properties, the water content that permissible for NEO is ≤200 ppm. However, the average value for the result of moisture content for fresh PFAE oil before filtering treatment is 82.7 ppm. Thus, it shows that the moisture content of PFAE oil after taking out from the barrel is meets the requirement and conditions for ASTM D6871 standards for NEO. From the result obtained for the moisture content of PFAE oil can be conclude that PFAE oil has capability to absorb much more moisture content compared to MO due to PFAE oil has the highest moisture content absorption. Thus, the PFAE oil that used in this experiment is not necessary for undergoing the insulating oil filtering treatment.

4.3.2 BDV for Fresh MO and Fresh PFAE Oil

The test of breakdown voltage (BDV) in this experiment is conducted by using Megger OTS60PB according to ASTM D1816 standards with electrode gap spacing 1mm. The result of BDV test for fresh MO and PFAE oil are plotting using Weibull probability plot as in Figure 4.1 and Figure 4.2.

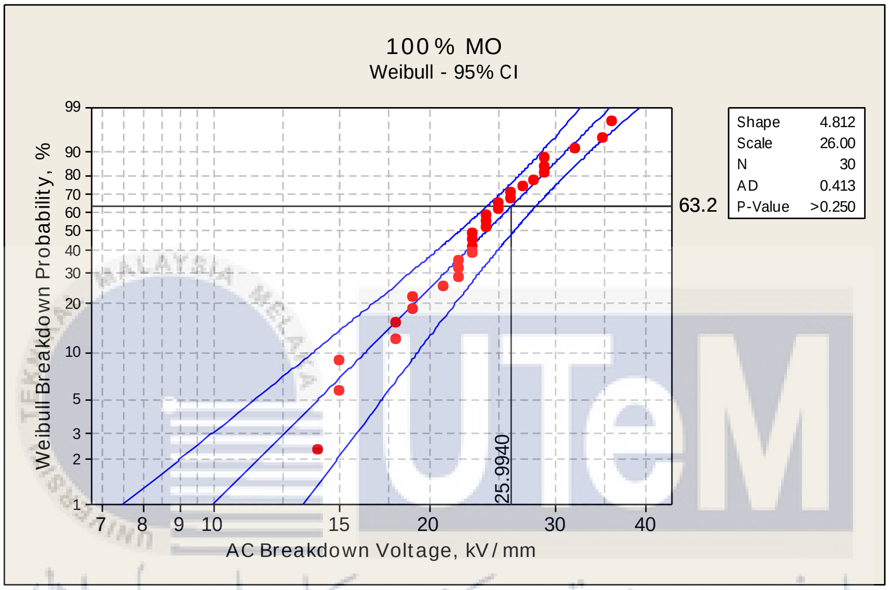


Figure 4.1: Result of BDV using Weibull probability plot for fresh MO

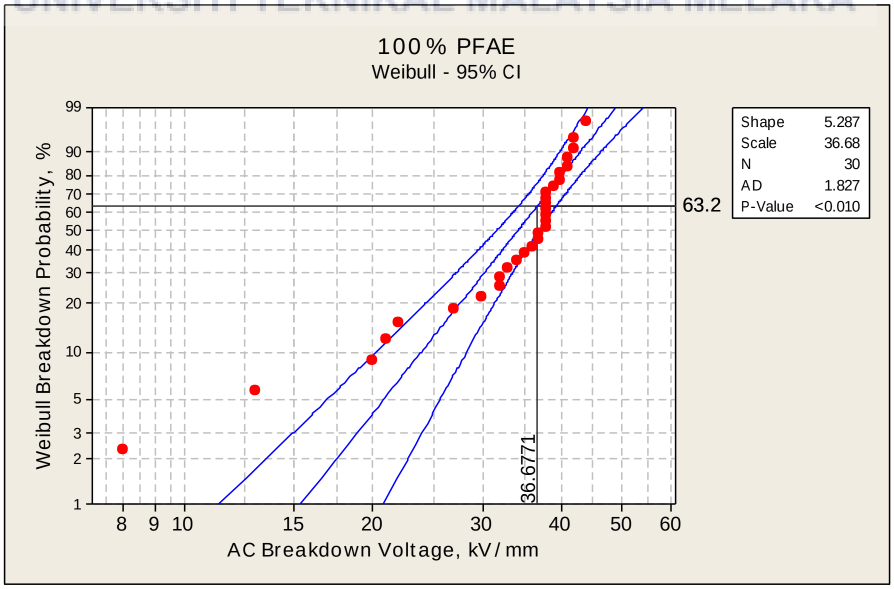


Figure 4.2: Result of BDV using Weibull probability plot for fresh PFAE oil

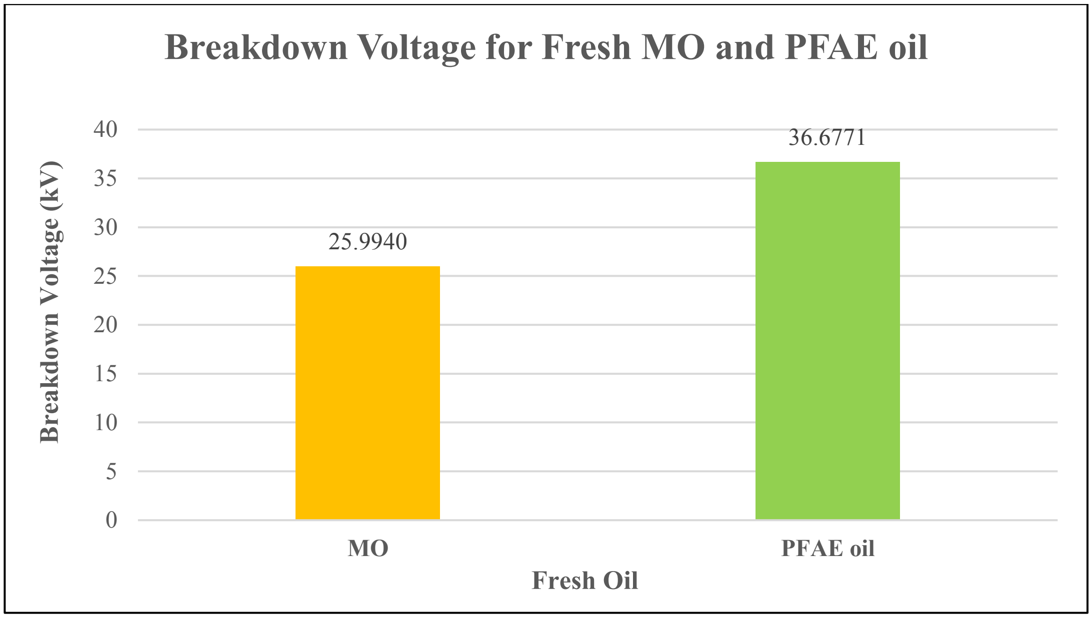


Figure 4.3: BDV for fresh MO and fresh PFAE oil

There are 30 data that recorded in Appendix A in this experiment. Apart from that, the purpose of using Weibull probability plot is to measure the best value of data as it has been widely used for voltage endurance to insulating oil specimens and measured the breakdown voltage in the experiments. The probability value of the data falls at 63.2% where it is the point corresponds to the time of failure of the specimen under test. Based on Table B.1 from Appendix A, the minimum BDV for MO is 14 kV and the maximum is 36 kV. Meanwhile, the minimum BDV for PFAE oil is 8 kV and the maximum 44 kV. By applying Weibull probability plot, the approximate value of BDV for both MO and PFAE oil that intersects the 63.2% unreliability line are 25.9940 kV for MO as in Figure 4.1 and 36.6771 kV for PFAE oil as in Figure 4.2. Thus, from the Figure 4.3, PFAE oil has the highest BDV compared to MO with differences of 10.6831 kV. This is due to PFAE oil has highest absorption of moisture content than MO. Moreover, both MO and PFAE oil are meets the requirement and their standard properties for BDV which are ≥20 kV with using 1 mm of gap spacing of electrode.

4.4 Result of the Differences Ratio of Concentration of Mixed Insulating Oil

4.4.1 Moisture Content for Differences Ratio of Concentration of Mixed Insulating Oil

The measurement of moisture content on differences ratio of concentration of mixed insulating oil are conducted before the process of bubble nitrogen through oil sample in which for undergoing of BDV test by referring to the previous research method. The measurement of moisture content is using Coulometric Karl Fischer Titration in accordance to ASTM D1533 standards. The result of moisture content for all ratio of concentration of mixed insulating oil before the process of bubble nitrogen through oil sample are recorded in Table 4.4 while Table 4.5 shows the result of moisture content for all ratio of concentration of mixed insulating oil after the process of bubble nitrogen through oil sample.

Table 4.4: Average moisture content before the process of bubble nitrogen through oil sample

Samples	**No. of Test Conducted**	**Sample Size (g)**	**Moisture content (ppm)**	**Average (ppm)**
90% MO + 10% PFAE	Test 1	0.8754	33.6	35.3
	Test 2	0.8716	35.3	
	Test 3	0.8332	37.0	
80% MO + 20% PFAE	Test 1	0.5457	47.3	46.0
	Test 2	0.8871	42.5	
	Test 3	0.6575	48.1	
70% MO + 30% PFAE	Test 1	0.9776	61.3	56.3
	Test 2	0.9077	54.8	
	Test 3	0.8266	52.7	
60% MO + 40% PFAE	Test 1	0.9232	72.9	71.2
	Test 2	0.8629	70.3	
	Test 3	0.8574	70.5	
50% MO + 50% PFAE	Test 1	0.8555	65.3	67.4
	Test 2	0.8793	65.2	
	Test 3	0.6601	71.7	

40% MO + 60% PFAE	Test 1	0.9753	94.2	91.5
	Test 2	0.9162	92.1	
	Test 3	0.8783	88.2	
30% MO + 20% PFAE	Test 1	1.1088	109.9	114.1
	Test 2	0.9902	112.8	
	Test 3	0.7384	119.6	
20% MO + 80% PFAE	Test 1	0.9635	116.6	112.8
	Test 2	0.9195	111.8	
	Test 3	0.845	110.0	
10% MO + 90% PFAE	Test 1	0.9204	134.1	131.0
	Test 2	0.8035	129.9	
	Test 3	0.7731	128.9	

Table 4.5: Average moisture content after the process of bubble nitrogen through oil sample

Samples	**No. of Test Conducted**	**Sample Size (g)**	**Moisture content (ppm)**	**Average (ppm)**
90% MO + 10% PFAE	Test 1	0.9071	10.8	10.3
	Test 2	0.8836	9.9	
	Test 3	0.8474	10.3	
80% MO + 20% PFAE	Test 1	0.7605	10.7	12.93
	Test 2	0.7392	12.2	
	Test 3	0.776	15.9	
70% MO + 30% PFAE	Test 1	1.0012	16.4	15.0
	Test 2	0.877	13.6	
	Test 3	0.8284	15.1	
60% MO + 40% PFAE	Test 1	0.9752	16.5	16.8
	Test 2	0.8651	16.7	
	Test 3	0.7896	17.2	

50% MO + 50% PFAE	Test 1	0.7557	15.9	16.0
	Test 2	0.7922	16.5	
	Test 3	0.7365	15.6	
40% MO + 60% PFAE	Test 1	0.7557	25.2	25.1
	Test 2	0.7922	23.3	
	Test 3	0.7365	26.8	
30% MO + 70% PFAE	Test 1	1.0457	30.3	28.2
	Test 2	0.895	28	
	Test 3	0.8928	26.4	
20% MO + 80% PFAE	Test 1	1.0428	26.7	26.2
	Test 2	1.0018	16.2	
	Test 3	0.9207	35.6	
10% MO + 90% PFAE	Test 1	1.0531	49.5	42.4
	Test 2	0.8705	46.6	
	Test 3	0.8256	31.2	

From Table 4.4, it shows that the result of moisture content of all oil samples which are after kept in the dark places for overnight before the process of bubble nitrogen through oil sample conducted. The ratio of 90% MO + 10% PFAE shows the lowest average of moisture content while the ratio of 10% MO + 90% PFAE shows the highest average of moisture content. The ratio of 10% MO + 90% PFAE gives the advantages compared with other oil samples due to this ratio has a lot of ester content to be mixed with MO. This reinforces the reason of NEO can absorb much water and can withstand until 200 ppm of moisture content. Meanwhile, Table 4.5 shows that result of moisture content after under through the process of bubble nitrogen through oil sample. The ratio of 10% MO + 90% PFAE is remain shows the highest average of moisture content even though the process of bubble nitrogen through oil sample is conducted. Thus, from the Table 4.4 and Table 4.5, it can be conclude that the more ester content that added to be mixed with MO, the average of moisture content for oil sample is significantly increased.

4.4.2 BDV for Differences Ratio of Concentration of Mixed Insulating Oil

There are eleven (11) samples with differences ratio of concentration of mixed insulating oil that conducted in this experiment and 500 ml is set as based quantity of each samples. The samples are prepared as implement by previous research method which are 400 RPM, 90°C for temperature level and the duration for mixing between MO and PFAE oil is about 1 hour. Then, the experiment for conducting the BDV test is performed by using Megger OTS60PB in according ASTM D1816 standard with 1mm gap spacing. The result of BDV for all ratio of concentration of mixed insulating oil are plotting using Weibull probability plot as shown in Figure 4.4, Figure 4.5, Figure 4.6, Figure 4.7, Figure 4.8, Figure 4.9, Figure 4.10, Figure 4.11 and Figure 4.12.

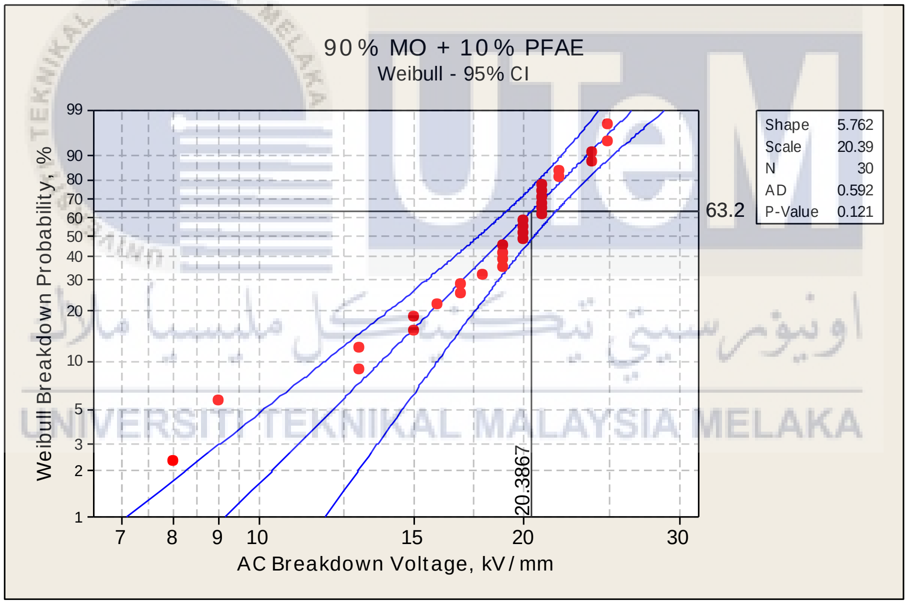


Figure 4.4: Result of BDV using Weibull probability plot for 90% MO + 10% PFAE

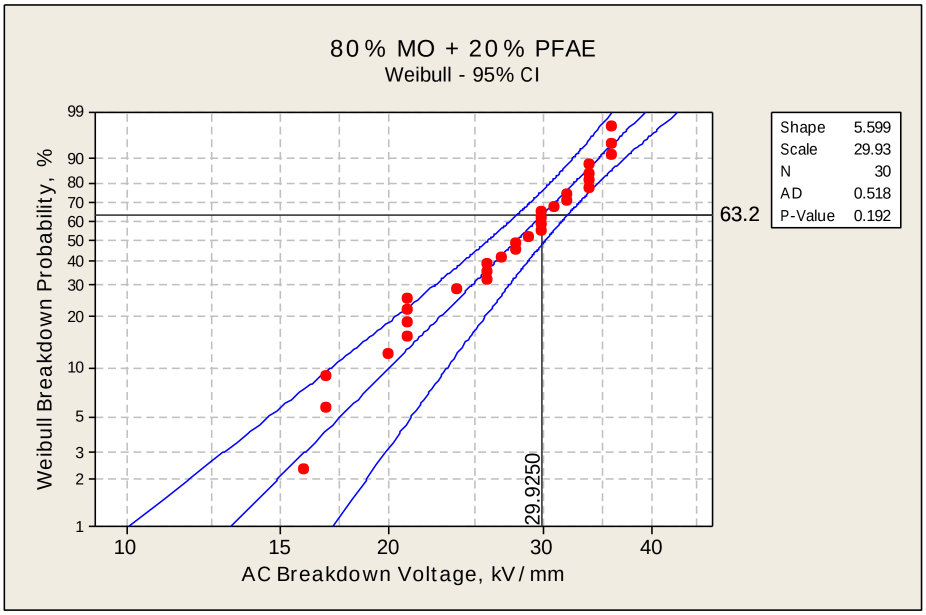


Figure 4.5: Result of BDV using Weibull probability plot for 80% MO + 20% PFAE

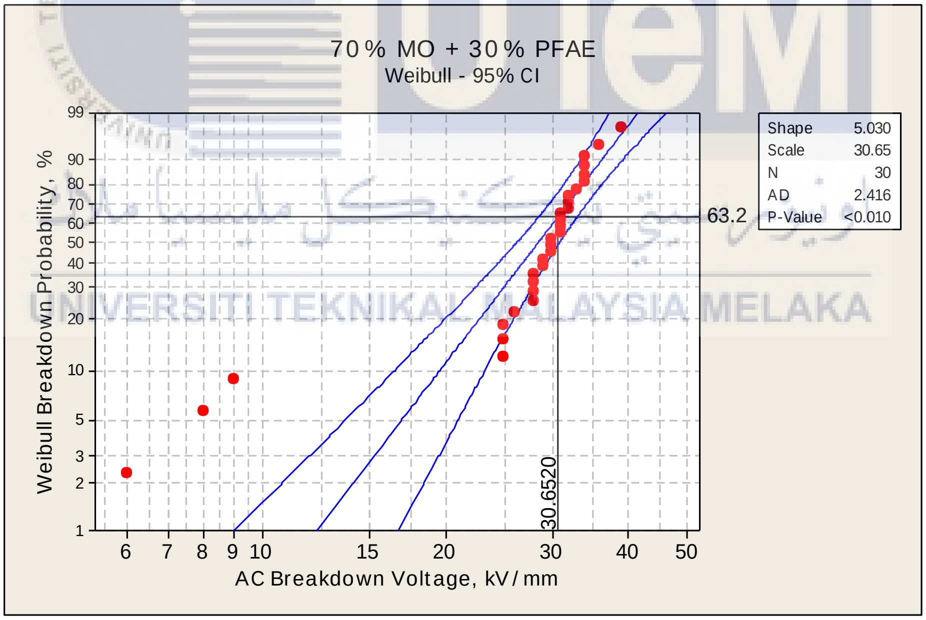


Figure 4.6: Result of BDV using Weibull probability plot for 70% MO + 30% PFAE

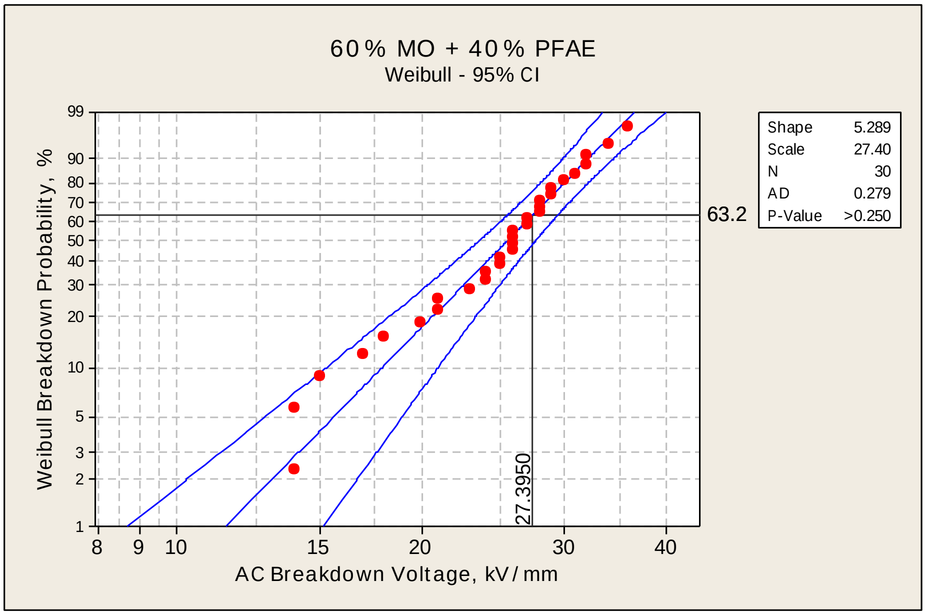


Figure 4.7: Result of BDV using Weibull probability plot for 60% MO + 40% PFAE

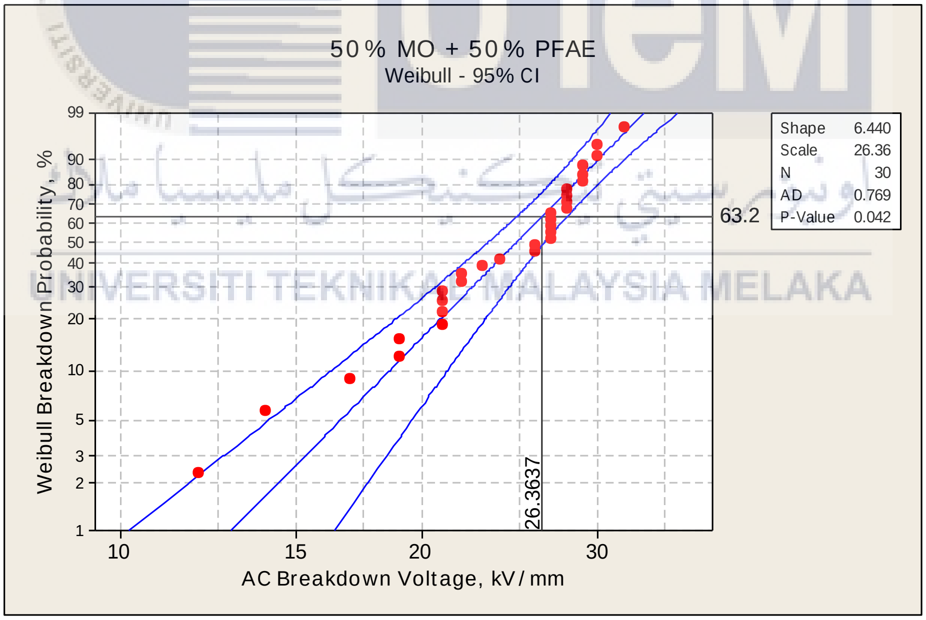


Figure 4.8: Result of BDV using Weibull probability plot for 50% MO + 50% PFAE

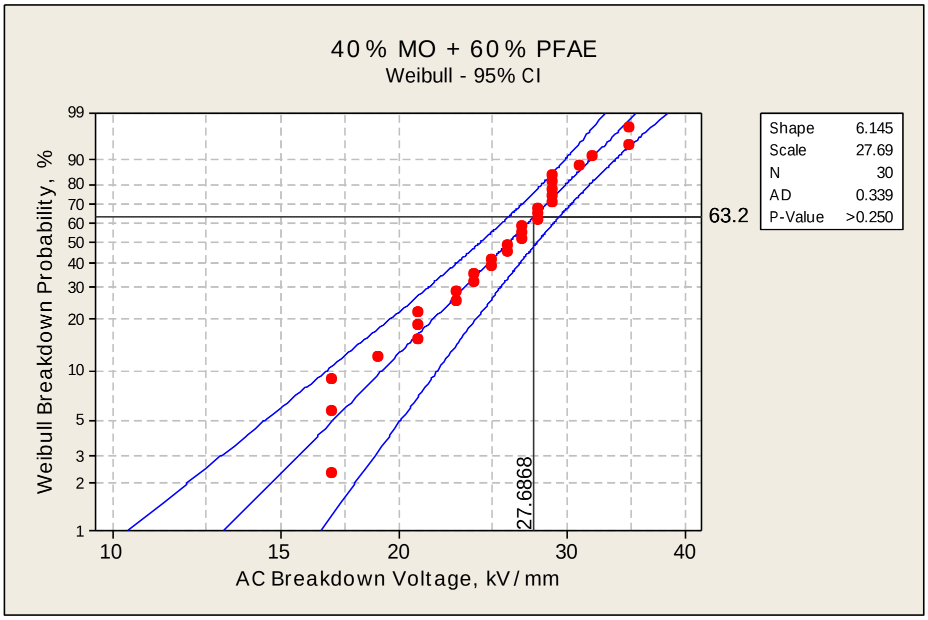


Figure 4.9: Result of BDV using Weibull probability plot for 40% MO + 60% PFAE

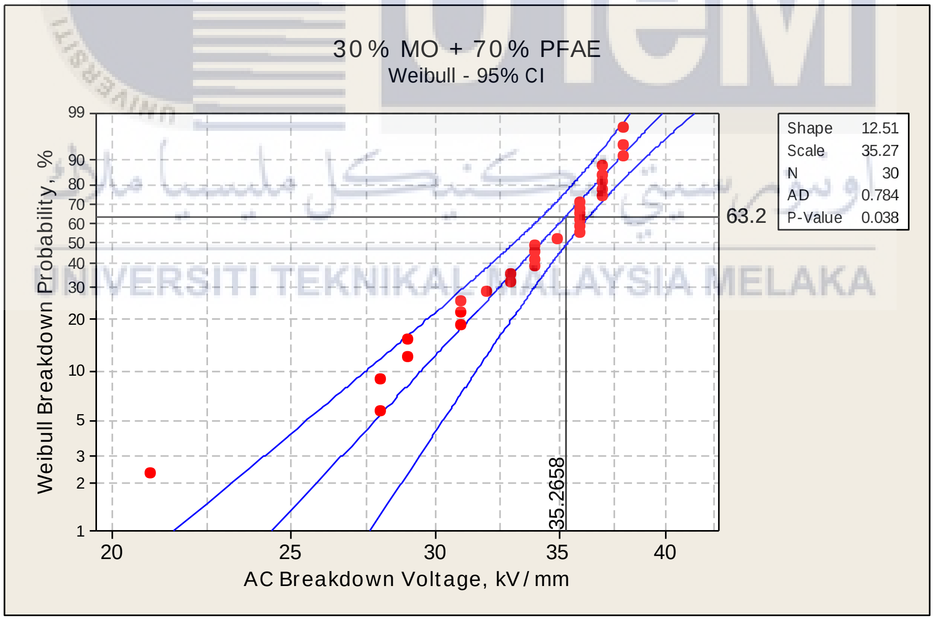


Figure 4.10: Result of BDV using Weibull probability plot for 30% MO + 70% PFAE

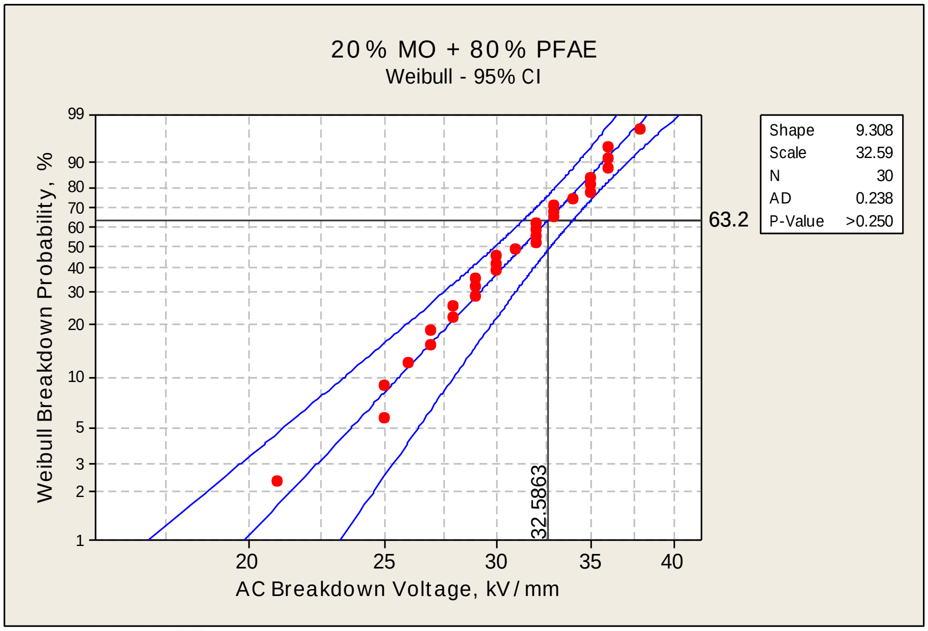


Figure 4.11: Result of BDV using Weibull probability plot for 20% MO + 80% PFAE

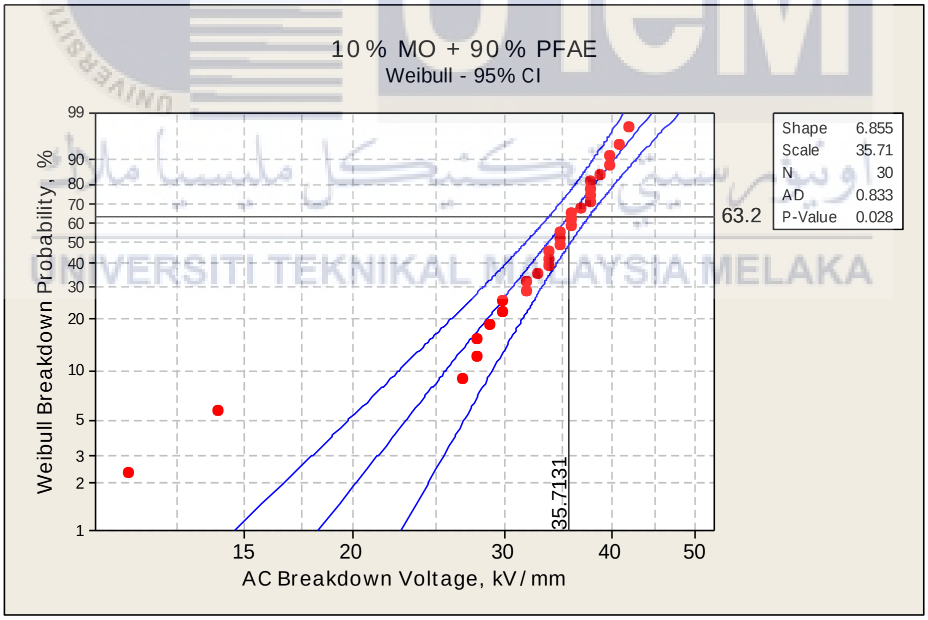


Figure 4.12: Result of BDV using Weibull probability plot for 10% MO + 90% PFAE

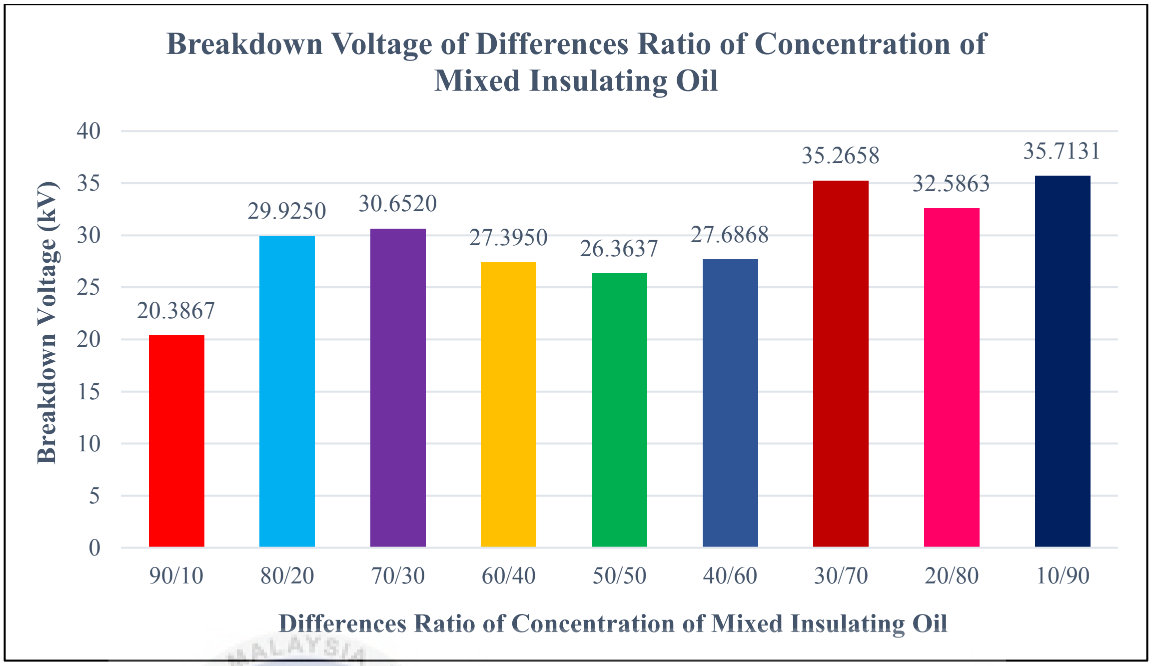


Figure 4.13: BDV of all ratio of concentration of mixed insulating oil

The breakdown voltage (BDV) plays an important role in determine the best ratio of concentration of mixed insulating oil. Since the mixed insulating oil between MO and NEO is not done research widely, through this study have displayed out the result of all ratio of concentration of MO by mixing with PFAE oil with increment 10% of PFAE oil. Hence, from the Figure 4.13 shows that the result of all concentration of mixed insulating oil using Weibull probability plot. Besides that, the result of breakdown voltage for the ratio of 100% MO and 100% PFAE are referring to the fresh MO and fresh PFAE oil as in Chapter 4.3.2. There are 30 data that recorded for each ratio of concentration of mixed insulating oil in Table B.2 from Appendix A in this experiment.

Based on the Figure 4.13 of Weibull probability plot, the approximate value of BDV that intersects the 63.2% unreliability line for all ratio of concentration are as follows; 20.3867 kV for ratio of 90% MO + 10% PFAE as shown in Figure 4.4, 29.9250 kV for ratio of 80% MO + 20% PFAE as shown in Figure 4.5, 30.6520 kV for ratio of 70% MO + 30% PFAE as shown in Figure 4.6, 27.3950 kV for ratio of 60% MO + 40% PFAE as shown in Figure 4.7, 26.3637 kV for ratio of 50% MO + 50% PFAE as shown in Figure 4.8, 27.6868 kV for ratio of 40% MO + 60% PFAE as shown in Figure 4.9, 35.2658 kV for ratio of 30% MO + 70% PFAE as shown in

Figure 4.10, 32.5863 kV for ratio of 20% MO + 80% PFAE as shown in Figure 4.11, and 35.7131 kV for ratio of 10% MO + 90% PFAE as shown in Figure 4.12.

From the experimental result, the mixed insulating oil of ratio of 10% MO + 90% PFAE has the result of BDV is 35.7131 kV which means this ratio shows the highest BDV among other samples. This is due to PFAE oil is higher absolute water inside and much higher of water solubility in which can gives the best combination when mixing with MO. By putting much ester content to be mixed with MO can affects the properties of the sample and then significantly enhance the BDV.

However, as focus to the objective for conducting this study is to find the best mixed insulating oil between MO and NEO without reducing the quantity of MO too much. Thus, the ratio of 50% MO + 50% PFAE is set as reference for enhancement of BDV and below than that ratio is considered as does not meet the purpose for the objective of this study. Then, the ratio of concentration of mixed insulating oil that selected for measuring the enhancement of BDV are as in Table 4.6.

Table 4.6: Enhancement of BDV for sample with differences ratio of concentration of mixed insulating oil

Samples	BDV (kV)	Enhancement (%)
50% MO + 50% PFAE	26.3637	References
90% MO + 10% PFAE	20.3867	-22.67
80% MO + 20% PFAE	29.9250	13.51
70% MO + 30% PFAE	30.6520	16.27
60% MO + 40% PFAE	27.3950	3.91

From the analysis that recorded in Table 4.6, it shows that the ratio of 70% MO + 30% PFAE has 16.27% for enhancement of BDV which is represents the greatest for enhancing the BDV compared with other samples by set the 50% MO + 50% PFAE as references of enhancement of BDV. The ratio of 70 % MO + 30% PFAE has differences of BDV enhancement with the ratio of 80% MO + 20% PFAE which is 0.727 kV and 2.76% for enhancement of BDV.

Thus, it is interesting to note that by adding 30% of PFAE oil enable to improve the BDV average value of MO and to avoid the reduction of its dielectric strength after mixing. In addition, PFAE oil is listed as NEO which is has high water solubility and thus enable to reduce the influence of humidity on MO. Thus, the factors of the chemical interactions is take into account which is the result of BDV is considerably depends on the presence of contaminants, such as moisture content, dirt, or other conducting particles in the oil.

4.4 Result of the Corrosive Sulfur

The test method that applied for conducting corrosive sulfur detection in this experiment is complies with Method B in ASTM D1275 standards which are using copper strip with measurement 6 mm x 25 mm, 250 mL non-filtered as based quantity of oil sample and the main aging parameters is about 48 hours @ 2880 minutes at 150°C. On the other hand, the Method B in ASTM D1275 is applied due to this method is more sensitive, long of exposure time and enable to indicate correctly the presence of corrosive sulfur in the evaluated oils. There are four (4) samples that selected for under through the corrosive sulfur test which are; 100% MO, 100% PFAE, 80% MO + 20 PFAE and 70% MO + 30% PFAE. These 4 samples are selected because of to make 100% MO and 100% PFAE as reference for reaction of sulfur corrosion while 80% MO + 20 PFAE and 70% MO + 30% PFAE as the sample which have the highest BDV and thus it is important in determining which of the 2 samples will shows the best result under this test. Hence, the result of reaction of sulfur corrosion is referring to ASTM D130 standards for classification of copper strip tarnish level. The result of corrosive sulfur four 4 samples are as in Figure 4.14, Figure 4.15, Figure 4.16 and Figure 4.17

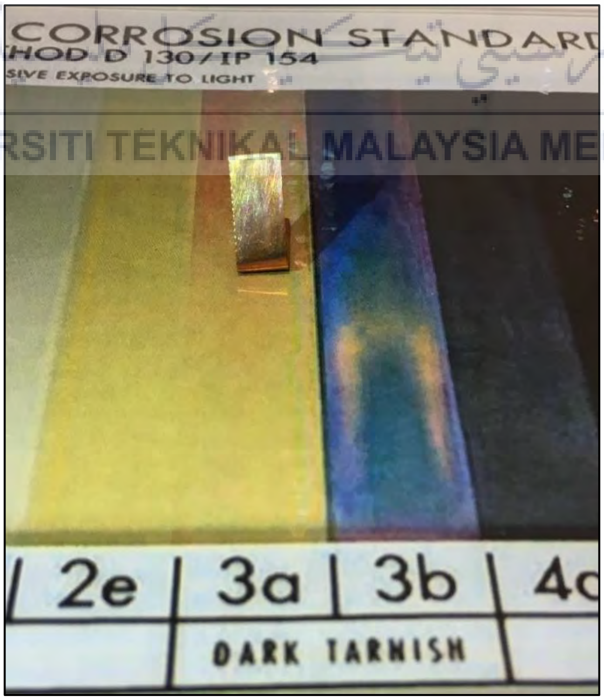


Figure 4.14: Result of corrosive sulfur for copper strip after immersed in 100% MO

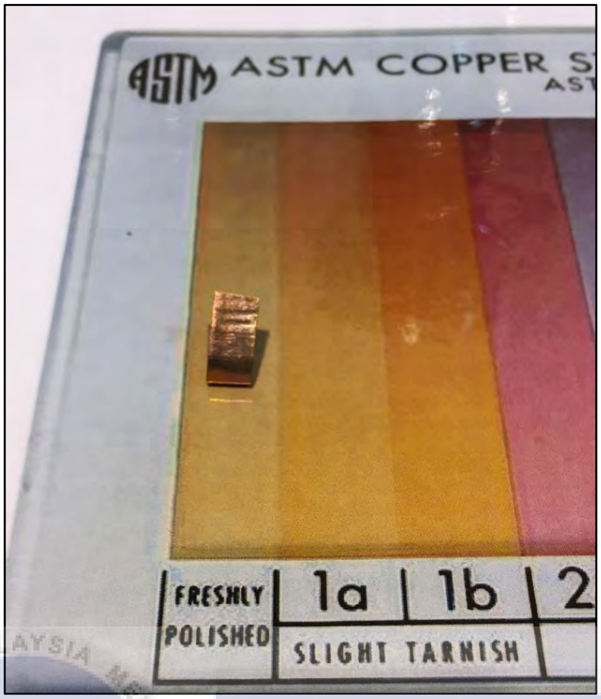


Figure 4.15: Result of corrosive sulfur for copper strip after immersed 100% PFAE

Figure 4.16: Result of corrosive sulfur for copper strip after immersed in 80% MO + 20% PFAE

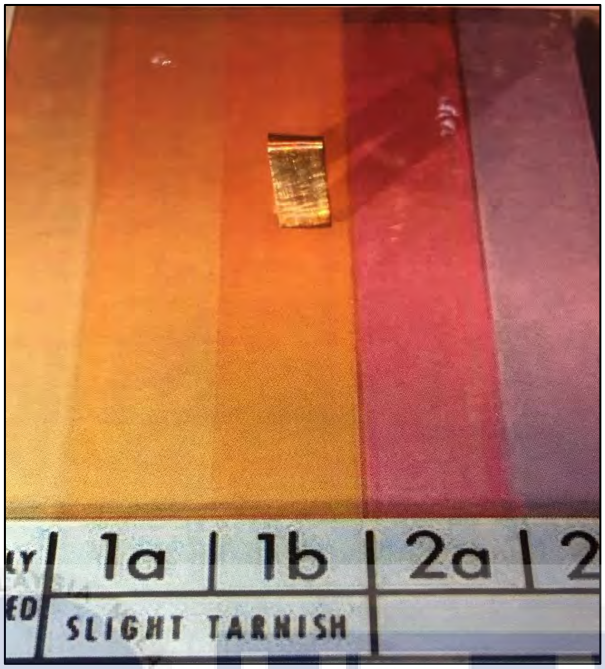


Figure 4.17: Result of corrosive sulfur for copper strip after immersed in 70% MO + 30% PFAE

Basically, the resultant formation of copper sulfide (Cu_2S) and particulate matters are the factors of typical winding failures in transformers and shunt reactors. Normally, MO is referred as the insulating oil which contains of sulfur compound and thus it supported with the reason of the corrosive sulfur in transformer oil originates mainly from the refining process of MO in which process there were residual sulfur ingredients [10]. Thus, through the experimental result shows that the above statement is proved in Figure 4.14 which is the copper strip that immersed in MO after the corrosive sulfur test is classified as 3a in classification of copper strip tarnish level of ASTM D130. The result of copper strip color that immersed in MO is dark tarnish with the description is the color of copper strip is magenta overcast on brassy strip. Table 4.7 shows the summarized the result of copper strip that immersed in the samples after corrosive sulfur test is classified in classifications of copper strip tarnish level in ASTM D130

Table 4.7: Result of copper strip that immersed in the samples after corrosive sulfur test is classified in classifications of copper strip tarnish level in ASTM D130

Classification	Designation	Description		Results of copper strip
Freshly polished strip			Unable to reproduce upon aging so no description is provided	Copper strip for 100% PFAE
1	Slight tarnish	a	Light orange, almost the same as freshly polished strip	
		b	Dark orange	Copper strip for 70% MO + 30% PFAE
2	Moderate tarnish	a	Claret red	
		b	Lavender	
		c	Multicolored with lavender blue or silver or both, overlaid on claret red	
		d	Silvery	
		e	Brassy or gold	
3	Dark tarnish	a	Magenta overcast on brassy strip	Copper strip for 100% MO Copper strip for 80% MO + 20% PFAE
		b	Multicolored with red and green showing (peacock), but no gray	
4	Corrosion	a	Transparent black, dark gray or brown with peacock green barely showing	
		b	Graphite or lusterless black	
		c	Glossy or jet black	

The result of copper strip that immersed in PFAE oil after corrosive sulfur test is classified as freshly polished as in Figure 4.15 due to PFAE oil is categorized as NEO where it is derived directly from renewable natural sources. Thus, the characteristic of NEO is biodegradable and free form corrosive sulfur compounds [42]. Hence, the result of copper strip for the ratio of 80% MO + 20% PFAE after conducted the corrosive sulfur test is classified as dark tarnish as in Figure 4.17 with the description which is the color of copper strip after immersed is magenta overcast on brassy strip. This shows that PFAE has less ester content which is about 20% that be mixed with MO. Thus, the possibility can be made that the ester content does not affects the properties of mixing between MO and PFAE oil since the quantity of MO is much highest than PFAE oil. It can be conclude that there is still the presence of corrosive sulfur compound for this ratio. Meanwhile, the result of copper strip for 70% MO + 30% PFAE after conducted the corrosive sulfur test is classified as slight tarnish. This is because of the color of copper strip after been through the test is dark orange. Thus, it shows that more ester content that added to be mixed with MO can affects the properties of mixed insulating oil and reduced the presence of corrosive sulfur compound.

Based on the experimental result that has been made, the ratio of 70% MO + 30% PFAE is represents the best mixed insulating oil in terms of corrosive sulfur and this mixed insulating oil is suitable for used in the power transformer applications.

4.5 Result of the Flash Point

The test method of flash point is performed according ISO 2719:2002 standards using Pensky-Martens closed cup method. There are four (4) mixed insulating oil with differences ratio that selected to be tested of flash point in order to get the best mixed insulating oil after many test are conducted in this study. The result of flash point for 4 oil samples as shown in Figure 4.18.

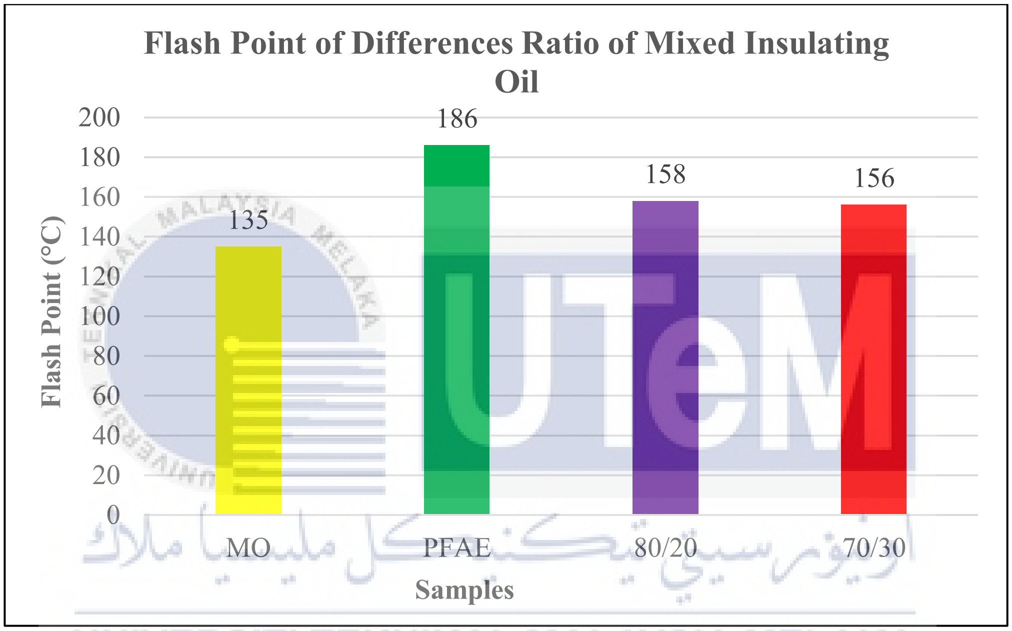


Figure 4.18: Flash point of differences ratio of mixed insulating oil

Generally, the purpose of doing flash point is to determine the minimum temperature at which heated oil gives off sufficient vapor to form a flammable mixture with air. It is an indicator of the volatility of the oil. The aim to do close cup is to simulate the situation of a liquid spill in a closed environment. A fire or explosion is a possibility when exposed to a potential ignition if the liquid is at or above of its flash point. Hence, the result as shown in Figure 4.18 is the flash point for 100% MO, 100% PFAE, the ratio of 80% MO + 20% PFAE and the ratio of 70% MO + 30% PFAE that be used in this experiment. Moreover, 100% MO and 100% PFAE are act as references in determine to select the best result of flash point for both ratio of 80% MO + 20% PFAE and 70% MO + 30% PFAE.

From the result obtained in Figure 4.18, it shows that the ratio of 80% MO + 20% PFAE with the temperature of 158°C has a higher flash point compared to the ratio of 70% MO + 30% PFAE with temperature of 156°C. Thus, at the temperature of 158°C for the ratio of 80% MO + 20% PFAE shows the lowest temperature corrected to a barometric pressure of 101.3 kPa to cause a flammable. Although the flash point for PFAE is the highest compared to other oil samples, it can gives the conclusion that by adding certain amount of ester content to be mixed with MO gives the possibility to enhance the flash point of mixed insulating oil. Thus, this analysis is taking into account of the flash point result is affects from the factor of contaminants in insulating oil since the oil that need to be tested for this flash point are not undergoing the process of filtering after taking out from the barrel. Apart from that, to conclude that the higher the value of flash point can give the best properties for the insulating oil. Otherwise, lower the flash point can gives the greater the fire hazard.

4.6 Summary

Table 4.8: Results for the four test that conducted on oil samples

Test / Oil samples	Moisture content (ppm)	Breakdown voltage (kV)	Corrosive sulfur (classification of strip)	Flash point (°C)
100% MO (set as reference)	28.4	25.994	Dark tarnish (3a)	135
100% PFAE (set as reference)	82.7	36.6771	Freshly polished strip	186
80% MO + 20% PFAE	12.93	29.9250	Dark tarnish (3a)	158
70% MO + 30% PFAE	15.0	30.6520	Slight tarnish (2b)	156

From this chapter, it has displayed the results of mixed insulating oil between Mineral Oil (MO) and Palm Fatty Acid Ester (PFAE) oil since the research on both oil is not done widely by many researcher. Many test are conducted throughout this study in order to find the best ratio of concentration of mixed insulating oil in which will give contribution to be applied in power transformer application. Based on the Table 4.8, it found that the mixed insulating oil with the ratio of 70% MO + 30% PFAE gives the best result in terms of breakdown voltage (BDV), moisture content and corrosive sulfur although the disparity of flash point with the flash point other oil sample is little differences. Then, the conclusion that conclude all the project implementation that have been done and the recommendation to improve for future studies are discussed in the next chapter.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Overall, each oil sample with differences ratio of concentration of mixed insulating oil have been through the experiment of moisture content and breakdown voltage (BDV). Meanwhile, the mixed insulating oil which shows the best ratio is selected for undergoing the further test which is the measurement process of corrosive sulfur and flash point. ASTM D3487 and ASTM D6871 standards are applied from the beginning of the experiment that conducted in selecting the insulating oil due to for ensure the fresh MO and the fresh NEO are complies with their standard properties. Meanwhile, all the experiment that conducted in this study are performed according to the standard that has been set such as the test of breakdown voltage (BDV), moisture content, corrosive sulfur and the flash point are referring to ASTM D1816, ASTM D1533, ASTM D1275 and ISO 2719:2002 standards.

Throughout the mixture process conducted in this study, the ratio of 70% MO + 30% PFAE represents the best compromise of the mixed insulating oil between MO and NEO in which this ratio enable to enhance the breakdown voltage (BDV) and thus fulfill the objective of this study which is to produce best ratio of the mixed insulating oil without reducing too much quantity of MO. Due to the characteristic of PFAE oil can withstand the moisture until 200 ppm, superior insulating performance, lower viscosity, biodegradability, good oxidation stability and excellent dielectric properties, thus, it interesting to note that by added 30% of ester content of PFAE oil to be mixed with MO in this study can be improved the electrical, chemical and physical parameter for example high dielectric strength, more absorption of water content, non-corrosive for corrosive and great potential for flash point. Since there is no standard properties for mixed insulating oil between MO and NEO to be referred and thus through this study can acknowledge the properties for each of ratio of concentration of mixed insulating oil in terms of moisture content, dielectric strength, corrosive sulfur and flash point. Lastly, the investigation on the mixture between MO and NEO, it can be conclude that the ester content is strongly affects the properties of mixture between MO and PFAE oil due to each MO and NEO shows that it has one advantage with regard to the others

5.2 Recommendation

Based on this study, it clearly shows that ester content is affecting the properties of mixture between MO and NEO. There a few suggestions for future plans study related to this project:

1. Study the investigation of the measurement of corrosive sulfur and flash point on all ratio of concentration of mixed insulating oil that has not been done.
2. Study the Total Acid Number (TAN) measurement on all ratio of concentration of mixed insulating oil for identifying the properties of each of mixed insulating oil.
3. Study the effect of variation of heat of mixed insulating oil on the value of breakdown voltage (BDV).

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APPENDIX A: Result of Breakdown Voltage (BDV)

Table B.1: Breakdown voltage for fresh MO and fresh PFAE

Samples	Breakdown Voltage (kV)	
	Fresh MO	Fresh PFAE
Test 1	29	13
Test 2	29	8
Test 3	25	20
Test 4	35	30
Test 5	26	27
Test 6	28	21
Test 7	36	22
Test 8	29	32
Test 9	32	37
Test 10	25	38
Test 11	27	42
Test 12	24	33
Test 13	24	35
Test 14	26	40
Test 15	22	32
Test 16	18	38
Test 17	15	42
Test 18	18	36
Test 19	15	38
Test 20	14	34
Test 21	22	38
Test 22	24	38
Test 23	23	37
Test 24	23	44
Test 25	23	40
Test 26	19	41
Test 27	21	38
Test 28	23	38
Test 29	19	41
Test 30	22	39

Table B.2: Breakdown voltage for all ratio of concentration of mixed insulating oil

Samples	**Breakdown Voltage (kV)**								
	90/10	**80/20**	**70/30**	**60/40**	**50/50**	**40/60**	**30/20**	**20/10**	**10/90**
Test 1	21	17	25	25	21	29	21	28	14
Test 2	20	17	6	20	14	29	31	26	36
Test 3	15	21	8	27	27	35	33	27	36
Test 4	9	21	9	14	17	31	29	29	38
Test 5	8	16	34	14	23	32	29	36	37
Test 6	25	20	25	15	21	28	28	38	40
Test 7	24	21	28	21	27	35	32	30	38
Test 8	22	36	28	21	21	28	34	33	38
Test 9	21	34	34	17	12	27	35	36	38
Test 10	21	34	30	28	28	29	36	35	11
Test 11	22	30	33	26	29	29	37	36	39
Test 12	25	32	36	26	26	27	34	34	42
Test 13	24	34	34	26	29	26	34	31	34
Test 14	19	36	31	25	30	29	38	35	32
Test 15	17	28	31	23	28	24	37	32	35
Test 16	13	30	26	32	29	24	38	29	35
Test 17	13	31	28	28	27	25	36	35	41
Test 18	20	34	32	31	27	23	34	33	36
Test 19	21	36	29	27	26	17	36	25	29
Test 20	19	29	39	30	28	23	37	32	40
Test 21	18	28	32	18	28	25	28	33	34
Test 22	17	26	31	34	27	26	31	32	27
Test 23	19	30	30	36	21	21	33	32	33
Test 24	20	26	32	24	30	21	37	29	32
Test 25	19	30	34	29	19	21	31	25	35
Test 26	21	32	30	26	22	28	38	30	34
Test 27	20	26	28	32	32	17	36	30	30
Test 28	21	24	25	24	19	17	36	28	30
Test 29	16	27	29	29	22	27	36	27	28
Test 30	15	21	31	28	24	19	37	21	28

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 Absorp
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 Gasesand 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.

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, mm^2/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			D3300
negative polarity point, min, kV	145	145	
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 20 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_#872079.

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.

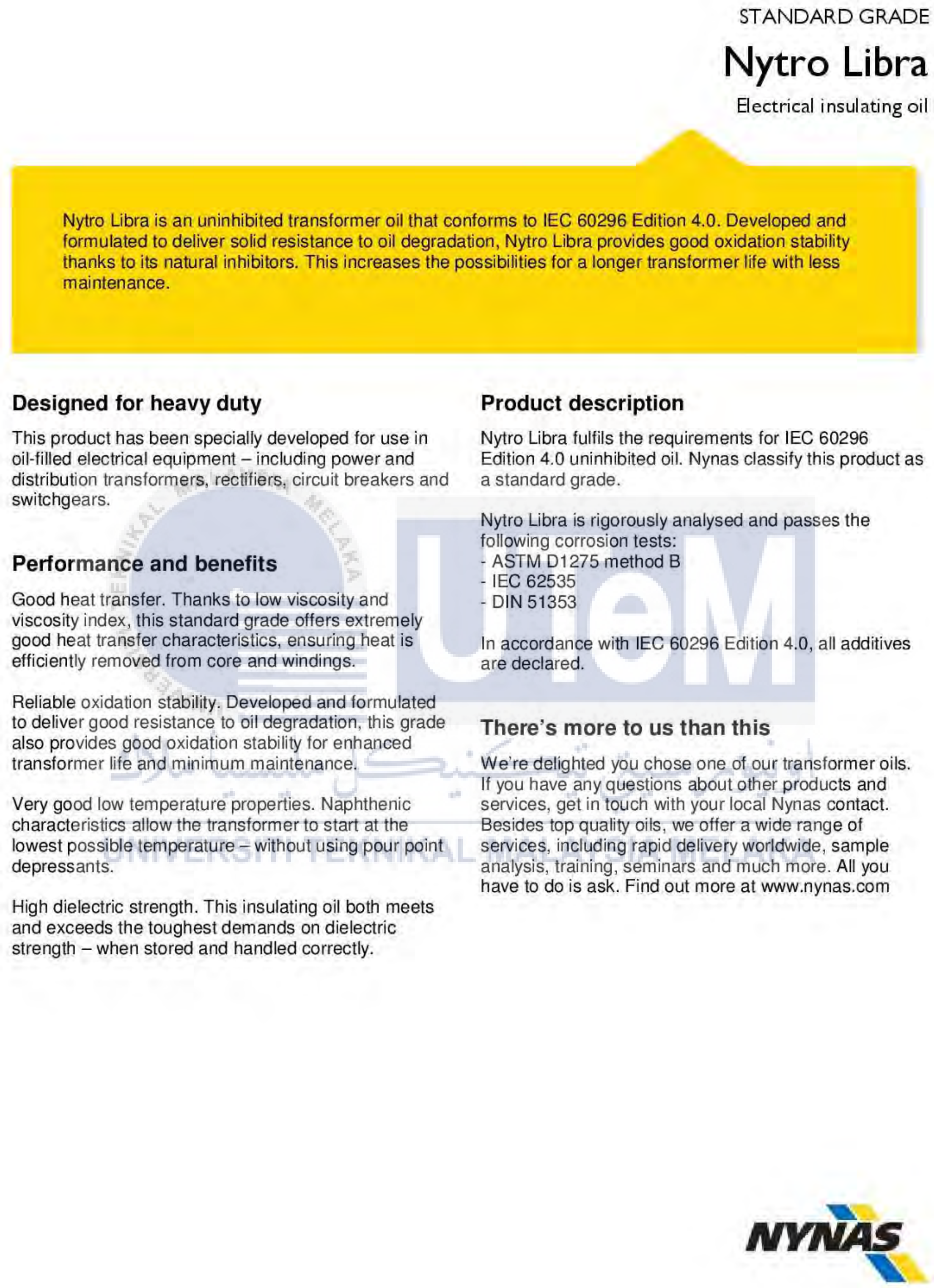
X3. PETROLEUM SOURCES, REFINING PROCESSES, AND SHIPPING CONTAINERS

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: Nytro Libra Datasheet



STANDARD GRADE

Nytro Libra

Electrical insulating oil

Nytro Libra is an uninhibited transformer oil that conforms to IEC 60296 Edition 4.0. Developed and formulated to deliver solid resistance to oil degradation, Nytro Libra provides good oxidation stability thanks to its natural inhibitors. This increases the possibilities for a longer transformer life with less maintenance.

Designed for heavy duty

This product has been specially developed for use in oil-filled electrical equipment – including power and distribution transformers, rectifiers, circuit breakers and switchgears.

Performance and benefits

Good heat transfer. Thanks to low viscosity and viscosity index, this standard grade offers extremely good heat transfer characteristics, ensuring heat is efficiently removed from core and windings.

Reliable oxidation stability. Developed and formulated to deliver good resistance to oil degradation, this grade also provides good oxidation stability for enhanced transformer life and minimum maintenance.

Very good low temperature properties. Naphthenic characteristics allow the transformer to start at the lowest possible temperature – without using pour point depressants.

High dielectric strength. This insulating oil both meets and exceeds the toughest demands on dielectric strength – when stored and handled correctly.

Product description

Nytro Libra fulfils the requirements for IEC 60296 Edition 4.0 uninhibited oil. Nynas classify this product as a standard grade.

Nytro Libra is rigorously analysed and passes the following corrosion tests:
- ASTM D1275 method B
- IEC 62535
- DIN 51353

In accordance with IEC 60296 Edition 4.0, all additives are declared.

There's more to us than this

We're delighted you chose one of our transformer oils. If you have any questions about other products and services, get in touch with your local Nynas contact. Besides top quality oils, we offer a wide range of services, including rapid delivery worldwide, sample analysis, training, seminars and much more. All you have to do is ask. Find out more at www.nynas.com

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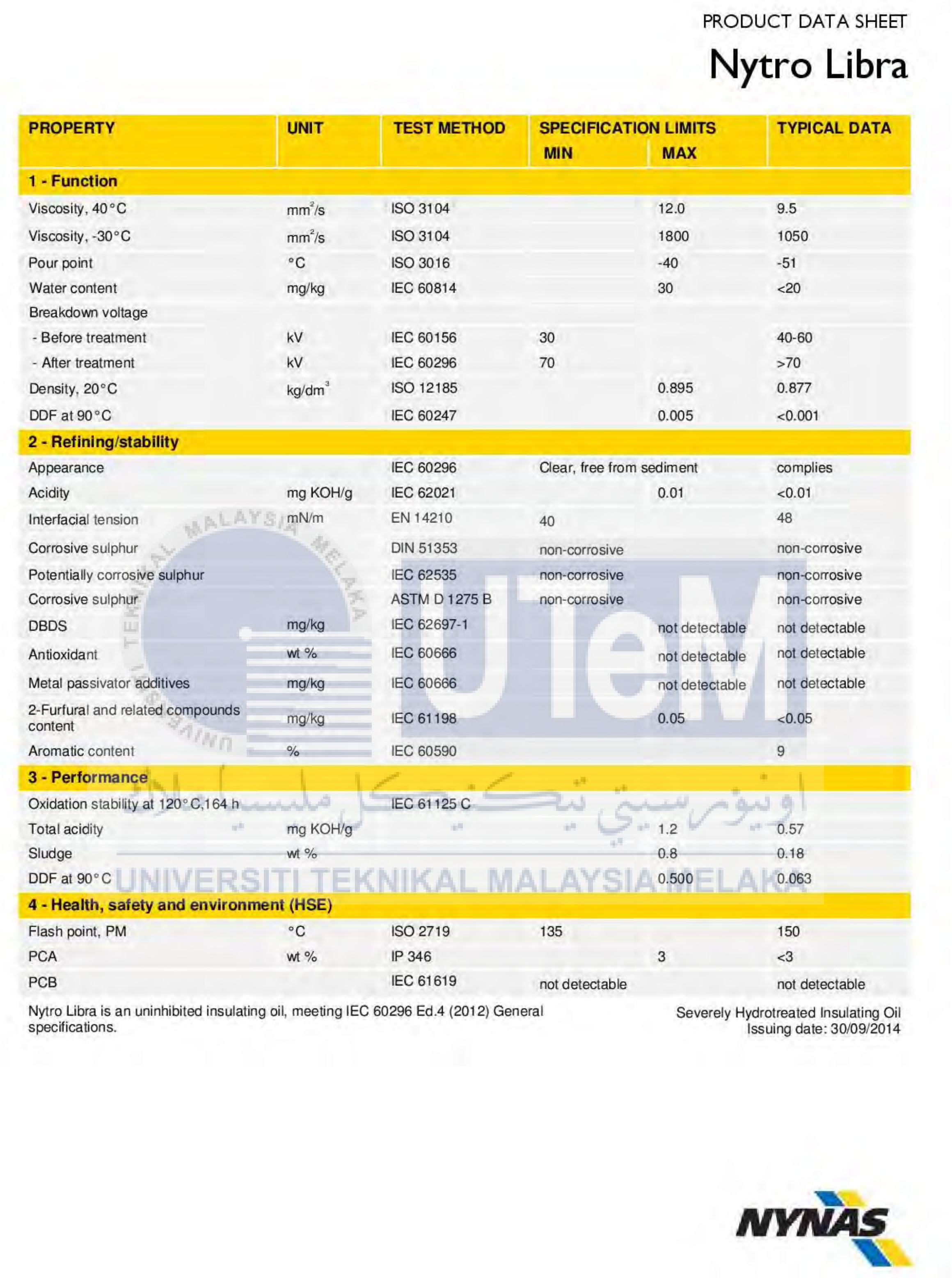

PRODUCT DATA SHEET

Nytro Libra

PROPERTY	UNIT	TEST METHOD	SPECIFICATION LIMITS		TYPICAL DATA
			MIN	MAX	
1 - Function					
Viscosity, 40°C	mm^2/s	ISO 3104		12.0	9.5
Viscosity, -30°C	mm^2/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/dm^3	ISO 12185		0.895	0.877
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		48
Corrosive sulphur		DIN 51353	non-corrosive		non-corrosive
Potentially corrosive sulphur		IEC 62535	non-corrosive		non-corrosive
Corrosive sulphur		ASTM D 1275 B	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	0.57
Sludge	wt %			0.8	0.18
DDF at 90°C				0.500	0.063
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.

Severely Hydrotreated Insulating Oil
Issuing date: 30/09/2014

APPENDIX D: ASTM D6871

Designation: D6871 – 03 (Reapproved 2008)

Standard Specification for Natural (Vegetable Oil) Ester Fluids Used in Electrical Apparatus[1]

This standard is issued under the fixed designation D6871; 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 specification covers a high fire point natural vegetable oil ester insulating fluid for use as a dielectric and cooling medium in new and existing power and distribution electrical apparatus such as transformers and attendant equipment.

1.2 Natural vegetable oil ester insulating fluid differs from conventional mineral oil and other high fire point (or "less-flammable") fluids in that it is an agricultural product derived from vegetable oils rather than refined from petroleum base stocks or synthesized from organic precursors.

1.3 This specification is intended to define a natural vegetable oil ester electrical insulating fluid that is compatible with typical materials of construction of existing apparatus and will satisfactorily maintain its functional characteristic in this application. The material described in this specification may not be miscible with some synthetic electrical insulating liquids. The user should contact the manufacturer of the natural ester insulating fluid for guidance in this respect.

1.4 This specification applies only to new insulating fluid as received prior to any processing. The user should contact the manufacturer of the equipment or fluid if questions of recommended characteristics or maintenance procedures arise.

1.5 *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 practices and determine the applicability of regulatory requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D88 Test Method for Saybolt Viscosity
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)
D877 Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes
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
D974 Test Method for Acid and Base Number by Color-Indicator Titration
D1275 Test Method for Corrosive Sulfur in Electrical Insulating Oils
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 Oils of Petroleum Origin 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
D2300 Test Method for Gassing of Electrical Insulating Liquids Under Electrical Stress and Ionization (Modified Pirelli Method)
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

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[1] This specification is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gasesand is the direct responsibility of Subcommittee D27.02 on Gases and Non-Mineral Oil Liquids.

Current edition approved Oct. 1, 2008. Published December 2008. Originally approved in 2003. Last previous edition approved in 2003 as D6871-03. DOI: 10.1520/D6871-03R08.

[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.

TABLE 1 As-Received New Fluid Property Requirements

Property	Limit	ASTM Test Method
Physical:		
Color, max	1.0	D1500
Fire point, min, °C	300	D92
Flash point, min, °C	275	D92
Pour point, max, °C	−10	D97
Relative density (specific gravity) 15°C/15°C, max	0.96	D1298
Viscosity, max, cSt at:		D445 or D88
100°C (212°F)	15	
40°C (104°F)	50	
0°C (32°F)	500	
Visual Examination	Bright and Clear	D1524
Electrical:		
Dielectric breakdown voltage at 60 Hz		
Disk electrodes, min, kV	30	D877
VDE electrodes, min, kV at:		D1816
1 mm (0.04 in.) gap	20	
2 mm (0.08 in.) gap	35	
Dielectric breakdown voltage, impulse conditions 25°C, min, kV, needle negative to sphere grounded, 1 in. (25.4 mm) gap	130	D3300
Dissipation factor (or power factor) at 60 Hz, max, %		D924
25°C	0.20	
100°C	4.0	
Gassing tendency, max, µl/min	0	D2300
Chemical:		
Corrosive sulfur	not corrosive	D1275
Neutralization number, total acid number, max, mg KOH/g	0.06	D974
PCB content, ppm	not detectable	D4059
Water, max, mg/kg	200	D1533[A]

[A] As stated in Test Methods D1533 Annex A1 "Alternative Solvent Systems," alternate reagents may be needed for certain natural ester formulations. Consult the manufacturer for recommendations. Reagents for aldehydes and ketones (such as Coulomat AK and CG-K) should be used if the additives are unknown. When alternate reagents are needed, using the Test Methods D1533 reagents may yield elevated and erratic water content results.

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

2.2 *National Fire Protection Association Standard:*

National Electrical Code Article 450-23[3]

3. Terminology

3.1 *Definitions*—Definitions of terms related to this specification are given in Terminology D2864. Vegetable oil natural ester: vegetable oil containing ester linkages, typically triglycerides. Most often obtained from seed crops (a "natural" source of esters, as opposed to synthesized esters).

4. Sampling and Testing

4.1 Take all fluid samples in accordance with Test Methods D923.

4.2 Perform each test in accordance with the ASTM test method specified in Table 1.

5. Property Requirements

5.1 Natural ester insulating fluid, as received, shall conform to the requirements of Table 1. The significance of these properties is covered in Guide D117 and Appendixes X2.1-X2.3.

6. Keywords

6.1 electrical insulating fluid; fire point; flammability; insulating fluid; natural ester

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[3] *National Electrical Code*, NFPA 70, National Fire Protection Association Inc.

APPENDIXES

(Nonmandatory Information)

X1. SUPPLEMENTARY DESIGN INFORMATION

X1.1 The following values are typical for natural ester insulating fluids.

Property	Typical Values	ASTM Test Method
Coefficient of Expansion, $^\circ C^{-1}$	0.0007 to 0.0008	D1903
Dielectric Constant, 25°C	3.1 to 3.3	D924
Specific Heat, cal/g, 20°C	0.45 to 0.60	D2766
Thermal Conductivity, (cal/cm·s·°C)	0.00035 to 0.00045	D2717

X2. SIGNIFICANCE OF PROPERTIES PARTICULAR TO NATURAL ESTER INSULATING OIL

NOTE X2.1—The significance of common electrical insulating fluid properties not listed here may be found in Guide D117.

X2.1 Physical Properties

X2.1.1 *Aniline Point*— The aniline point of natural ester insulating fluids vary dramatically from source to source. The variation is such that the aniline point may not be a dependable indicator of solvency of some materials in natural ester insulating fluids.

X2.1.2 *Interfacial Tension*—Interfacial tension performance criteria are being assessed; therefore, requirements have not been established. Interfacial tension of new natural ester insulating fluids, typically in the 26 to 30 mN/m range, is lower than that of new mineral oil.

X2.1.3 *Pour Point*— The pour point of a natural ester insulating fluid is important as an index of the lowest temperature to which the material may be cooled without seriously limiting the degree of circulation. Some natural ester fluids are sensitive to prolonged storage at low temperatures, and their pour points may not adequately predict their low temperature flow properties.

X2.2 Electrical Properties

X2.2.1 *Dissipation Factor*—Dissipation factor (power factor) is a measure of the dielectric losses in fluid. A low dissipation factor indicates low dielectric losses and a low level of soluble contaminants. Natural ester insulating fluids usually have higher dissipation factors than mineral insulating oils, especially at elevated temperatures.

X2.3 Chemical Properties

X2.3.1 *Water Content*— A low water content of natural ester insulating fluid is necessary to achieve adequate electrical strength and low dielectric loss characteristics, to maximize the insulation system life, and to minimize metal corrosion. Reagents for aldehydes and ketones should be used. The standard reagents for mineral oil may yield both elevated and erratic results. The amount of water required to saturate natural ester insulating fluid at room temperature is roughly 20 times that of mineral insulating oil. Comparisons to mineral insulating oil should be made on a percent saturation (relative saturation) basis rather than using absolute water content.

X2.3.2 *Neutralization Number*—A low total acid content of a natural ester insulating fluid is necessary to minimize electrical conduction and metal corrosion and to maximize the life of the insulation system. The neutralization numbers typical of natural ester insulating fluids are usually higher than those of mineral insulating oils. Natural ester insulating oils tend to form long-chain fatty acids. Mineral insulating oils tend to form shorter chain organic acids.

X2.3.3 *Oxidation Inhibitor Content*—The oxidation inhibitor additives that may be present in natural ester insulating fluids vary by manufacturer. The manufacturer should be contacted to obtain information and recommended analysis methods for additives.

X2.3.4 *Oxidation Stability*—Oxidation stability requirements and appropriate test methods for natural ester insulating fluids have not been established. Oxidation stability performance criteria are being assessed, and may differ for sealed systems, conservators, and free-breathing apparatus.

APPENDIX E: 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.

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]

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

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

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

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 Gasesand 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.

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]

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

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

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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).

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.

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:

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

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 8506 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.

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 F: ASTM D1816

INTERNATIONAL

Designation: D1816 – 12

Standard Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using VDE Electrodes[1]

This standard is issued under the fixed designation D1816; 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 liquids (oils of petroleum origin, silicone fluids, high fire-point mineral electrical insulating oils, synthetic ester fluids and natural ester fluids). This test method is applicable to insulating liquids commonly used in cables, transformers, oil circuit breakers, and similar apparatus as an insulating and cooling medium. Refer to Terminology D2864 for definitions used in this test method.

1.2 This test method is sensitive to the deleterious effects of moisture in solution especially when cellulosic fibers are present in the liquid. It has been found to be especially useful in diagnostic and laboratory investigations of the dielectric breakdown strength of insulating liquid in insulating systems.[2]

1.3 This test method is used to judge if the VDE electrode breakdown voltage requirements are met for insulating liquids. This test method should be used as recommended by professional organization standards such as IEEE C57.106.

1.4 This test method may be used to obtain the dielectric breakdown of silicone fluid as specified in Test Method D2225 and Specification D4652, provided that the discharge energy into the sample is less than 20 mJ (milli joule) per breakdown for five consecutive breakdowns.

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

1.6 *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 practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)
D923 Practices for Sampling Electrical Insulating Liquids
D2225 Test Methods for Silicone Fluids Used for Electrical Insulation
D2864 Terminology Relating to Electrical Insulating Liquids and Gases
D3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus
D4652 Specification for Silicone Fluid Used for Electrical Insulation
D6871 Specification for Natural (Vegetable Oil) Ester Fluids Used in Electrical Apparatus

2.2 *IEEE Standard:*

Standard 4 IEEE Standard Techniques for High Voltage Testing[4]
C57.106 Guide for Acceptance and Maintenance of Insulating Oil in Equipment[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. The dielectric breakdown voltage serves to indicate the presence of contaminating agents such as water, dirt, 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.)

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[1] This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gasesand is the direct responsibility of Subcommittee D27.05 on Electrical Test.

Current edition approved June 15, 2012. Published July 2012. Originally approved in 1960 as D1816 – 60 T. Last previous edition approved in 2004 as D1816 – 04. DOI: 10.1520/D1816-12.

[2] Supporting data is available from ASTM Headquarters. Request RR:D27-1006.

[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.

[4] Available from the Institute of Electrical and Electronic Engineers, Inc., PO Box 1331, Piscataway, NJ 08855.

*A Summary of Changes section appears at the end of this standard

3.2 This test method is used in laboratory or field tests. For field breakdown results to be comparable to laboratory results, all criteria including room temperature (20 to 30°C) must be met.

4. Electrical Apparatus

4.1 In addition to this section, use IEEE Standard 4 to determine other requirements necesary for conducting test measurements, and maintaining error limits using alternating voltages. Procedures to ensure accuracy should follow the requirements of IEEE Standard 4. Calibration(s) shall be traceable to national standards and calibration should be verified annually or more often to ensure accuracy requirements. IEEE Standard 4 is required during the manufacturing of the test apparatus and utilized during calibration of the equipment.

4.1.1 *Test Voltage*—The test voltage shall be an alternating voltage having a frequency in the range from 45 to 65 Hz, normally referred to as power-frequency voltage. The voltage wave shape should approximate a sinusoid with both half cycles closely alike, and it should have a ratio of peak-to-rms values equal to the square root of 2 within ±5 %.

4.1.2 *Generation of the Test Voltage*— The test voltage is generally supplied by a transformer or resonant circuit. The voltage in the test circuit should be stable enough to be unaffected by varying current flowing in the capacitive and resistive paths of the test circuit. Non-disruptive discharges in the test circuit should not reduce the test voltage to such an extent, and for such a time, that the disruptive discharge (breakdown) voltage of the test specimen is significantly affected. In the case of a transformer, the short-circuit current delivered by the transformer should be sufficient to maintain the test voltage within 3 % during transient current pulses or discharges, and a short circuit current of 0.1 A may suffice.

4.1.3 *Disruptive Voltage Measurement*— Design the measurement circuit so the voltage recorded at the breakdown is the maximum voltage across the test specimen immediately prior to the disruptive breakdown, with an error no greater than 3 %.

4.2 *Circuit-Interrupting Equipment*— Design the circuit used to interrupt the disruptive discharge through the specimen to operate when the voltage across the specimen has collapsed to less than 100 V. It is recommended that the circuit design limit the disruptive current duration and magnitude to low values that will minimize damage to the electrodes and limit formation of non-soluble materials resulting from the breakdown, but consistent with the requirements of 4.1.2, but in no case should the short-circuit current exceed 1 mA/kV of applied voltage.

4.3 *Voltage Control Equipment*—Use a rate of voltage rise of 0.5 kV/s. The tolerance of the rate of rise should be 5 % for any new equipment manufactured after the year 2000. Automatic equipment should be used to control the voltage rate of rise because of the difficulty of maintaining a uniform voltage rise manually. The equipment should produce a straight-line voltage-time curve over the operating range of the equipment. Calibrate and label automatic controls in terms of rate-of-rise.

4.4 *Measuring Systems*—The voltage shall be measured by a method that fufills the requirements of IEEE Standard No. 4, giving rms values.

4.5 Connect the electrode such that the voltage measured from each electrode with respect to ground during the test is equal within 5 %.

4.6 *Accuracy*—The combined accuracy of the voltmeter and voltage divider circuit shall be such that measurement error does not exceed 3 % at the rate-of-voltage rise specified in 4.3. For equipment manufactured prior to 1995 the maximum allowable error is 5 %.

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 ±1 %, mounted with axes horizontal and coincident within ±1 mm.

6. Test Cell

6.1 The test cell shall be designed to permit easy removal of the electrodes for cleaning and polishing, verification that the shape is within the specified tolerance, and to permit easy adjustment of the gap spacing. The vector sum of the resistive and capacitive current of the cup, when filled with oil meeting the requirements of Specification D3487, shall be less than 200 µA at 20 kV, at power frequency. Mount the electrodes rigidly from opposite sides with the spacing axially centered within ±1 mm. Clearance from the electrodes to all sides, bottom, cover or baffle, and any part of the stirring device is at least 12.7 mm (½ in.). Provide the test cell with a motor-driven two-bladed impeller and drive shaft, constructed of a material having high dielectric strength. The two-bladed impeller is 35 mm (1⅜ in.) ±5 % between the blade extremities, having a

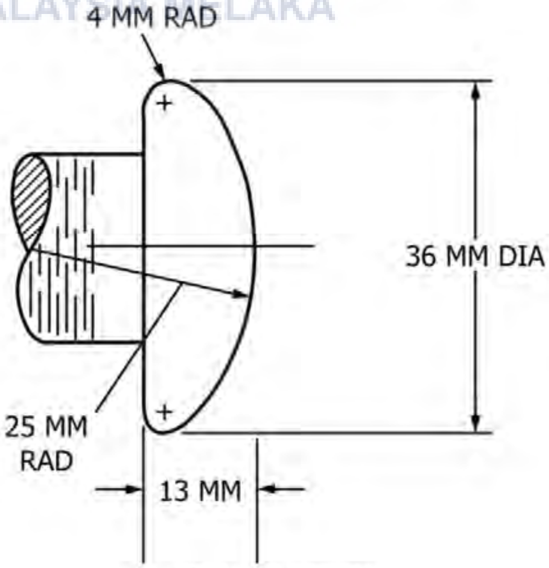

FIG. 1 VDE Electrode

pitch of 40 mm (1.57 in.) ±5 % (blade angle of twenty degrees (20°) ± 5 %), operating at a speed between 200 and 300 rpm. The impeller, located below the lower edge of the electrodes, rotates in such a direction that the resulting liquid flow is directed downward against the bottom of the test cell. Construct the test cell of a material of high dielectric strength, that is not soluble in or attacked by any of the cleaning or test liquids used, and is 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, provide the cell with a cover or baffle that will effectively prevent air from contacting the circulating liquid.

7. Adjustment and Care of Electrodes and Test Cell

7.1 *Electrode Spacing*—With the electrodes held firmly in place, check the electrodes with a standard round gage for 2 ± 0.03-mm (0.079-in.) spacing. If a dielectric breakdown does not occur during any of the consecutive breakdown tests using the 2 mm spacing or the sample is not adequate for the 2 mm spacing test cell a 1 ± 0.03-mm (0.039-in.) spacing should be used to determine the breakdown voltage and the spacing reported. Flat "go" and "no-go" gages may be substituted having thicknesses of the specified value ±0.03 mm for electrode spacing of 1 or 2 mm. If it is necessary to readjust the electrodes, set the electrodes firmly in place and check the spacing. For referee tests or tests that will be used for close comparisons, the laboratories shall agree in advance on the spacing for the tests and ensure that all other requirements of this test method are met. The spacing agreed upon shall be measured with the gage that corresponds exactly to the selected spacing within tolerance stated above for the gage.

7.2 *Cleaning*—Wipe the electrodes and cell 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 spacing, rinse the cell with a dry hydrocarbon solvent, such as kerosine or solvents of Specification D235. Do not use a low boiling point solvent, as its rapid evaporation may cool the cell, causing moisture condensation. If this occurs, before using, warm the cell to evaporate the moisture. Avoid touching the electrodes or the inside of the cell after cleaning. After thorough cleaning, flush the cell with new insulating liquid of the type to be tested that is filtered through a 5-micron filter or smaller and containing less than 25 ppm moisture. Conduct a voltage breakdown test on a specimen of this insulating liquid in the manner specified in this test method. If the breakdown voltage is in the expected range for this conditioned insulating liquid, the cell is considered properly prepared for testing other samples. A lower than anticipated value is considered as evidence of cell contamination; then repeat the cleaning and the breakdown test with clean dry insulating liquid.

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, drain the cell and flush the cell with new insulating liquid of the type to be tested that is filtered through a 5-micron filter and containing less than 25 ppm moisture before testing the next specimen. When not in use, keep the cell filled with oil that meets the requirements of Specification D3487 of the type normally tested. Alternatively, the cell may be stored empty in a dust-free cabinet. At the beginning of each days testing, clean according to 7.2.

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, followed by solvent rinsing or ultrasonic cleaning. After careful inspection, any electrodes from which pitting cannot be removed by light buffing should be discarded, as more 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 Obtain a sample of the insulating liquid to be tested using appropriate ASTM sampling apparatus. Insulating liquid sampling procedures are detailed in Practice D923. Particular reference should be made to the general precaution statement of this test method. The sample shall be taken in a dry, clean, non-permeable bottle. Tightly seal and shield from light until ready to be tested. Plastic bottles are permeable and moisture content of the sample may change resulting in a measurable difference when compared to samples collected in non-permeable containers. Prior to starting the test, the sample shall be inspected for the presence of moisture, sludge, metallic particles, or other foreign matter. If the sample shows evidence of free water, the dielectric breakdown test should be waived, and the sample reported as unsatisfactory.

9. Test Procedure

9.1 Allow the sample and the test cup to equilibrate to ambient temperature. Laboratory and referee tests shall be conducted at room temperature (20 to 30°C).

9.2 To ensure a homogenous sample, gently invert and swirl the sample container several times. Rapid agitation is undesirable, since an excessive amount of air may be introduced into the liquid. Within 1 min after agitation, use a small portion of the sample to rinse the test cell. Drain the rinse. Within 30 s of the rinse, fill the cell slowly with the remaining portion of the sample. The cell is full when closing the cover or baffle allows no air to be in contact with the insulating liquid.

9.3 Wait at least 3 min but no more than 5 min between filling the cup and application of voltage for the first breakdown. For high fire-point electrical insulating oils, natural esters and synthetic esters, the hold time before the initial application of voltage shall be at least 30 min. Apply the voltage increasing from zero at the rate of 0.5 kV/s ± 5 % until breakdown occurs as indicated by operation of the circuit-interrupting equipment; record the highest rms voltage value that occurred immediately prior to each breakdown. Occasional momentary discharges may occur which do not result in operation of the interrupting equipment; these shall be disregarded until the voltage across the specimen collapses to less than 100 V. Conduct four additional breakdowns waiting at least 60 s but no more than 90 s before applications of voltage for successive breakdowns. (During the intervals before voltage application, between breakdowns, and at the time voltage is being applied, the propeller shall be circulating the insulating liquid.)

9.4 Calculate the mean of the five breakdowns as follows:

$$\bar{X} = \frac{1}{5}\sum_{i=1}^{5} X_i$$

where:

$\bar{X}$ = mean of the five individual values,
X_i = ith breakdown voltage.

9.5 Using the breakdown voltage values determined in 9.3, determine that the range of the five (5) breakdown voltages does not exceed the values indicated as follows:

$$\text{Range} = X_{Highest} - X_{Lowest}$$

where:

$X_{Highest}$ = the highest breakdown voltage of the five readings, and
X_{Lowest} = the lowest breakdown of the five readings.

1 mm gap setting	Range must be less than 120 % of the mean of the five (5) breakdown voltages
2 mm gap setting	Range must be less than 92 % of the mean of the five (5) breakdown voltages

If this range is exceeded and there is sufficient volume of test specimen, repeat the analysis on a new cup filling. If an insufficient volume of test specimen is available, the test result can be reported with an additional comment that the range of the breakdowns exceeds the allowable range.

9.6 When it is desired merely to determine if the dielectric strength is above or below a specified level, five breakdowns are required, provided the five values are all above or all below this level. Otherwise, follow the procedure described in 9.3 – 9.5

10. Report

10.1 Include in the report the following:

10.1.1 The test method used,

10.1.2 The volts (rms value) at each breakdown, and the mean of all breakdowns reported to two significant digits,

10.1.3 The approximate temperature of the insulating liquid at the time of the test, and

10.1.4 The electrode spacing.

11. Precision and Bias[5]

11.1 *Single-Operator Precision*—The single operator percent coefficient of variance of a single test result comprised of 5 breakdowns has been found for the 1 mm gap to be 14 % and for the 2 mm gap 11 %. Therefore, results of two properly conducted tests by the same operator on the same sample should not differ by more than 40 % and 30 % respectively. The maximum allowable range for the series of 5 breakdowns should not exceed 120 % for the 1 mm gap and 92 % for the 2 mm gap.

11.2 *Multilaboratory Precision*—The multilaboratory percent coefficient of variance has been found for the 1 mm gap to be 17 % and for the 2 mm gap 14 %. Therefore, two separate test results conducted by separate laboratories on the same sample of insulating liquid should not differ by more than 47 % of the mean of the two tests if the 1 mm gap was used or differs by more than 40 % of the mean of the two tests if the 2 mm gap was used. The precision statement for the 1 mm gap is temporary, because sufficient laboratories were not available and testing is planned to replace the precision statement before 2009.

11.3 *Bias*—No statement can be made about the bias of this test method because a standard reference material is not available.

12. Keywords

12.1 breakdown voltage; dielectric strength; insulating liquids; insulating oils; test cell; VDE electrodes

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[5] Supporting data is available from ASTM International Headquarters. Request RR:D27-1014.

APPENDIX

(Nonmandatory Information)

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

X1.1 The dielectric breakdown voltage of an insulating liquid at commercial power frequencies is also affected by:

X1.1.1 The degree of uniformity of the electric field,

X1.1.2 The area of the electrodes,

X1.1.3 Volume of the insulating liquid under maximum stress,

X1.1.4 Insulation on the electrodes,

X1.1.5 Water content of the insulating liquid,

X1.1.6 Size and number of particles in the insulating liquid,

X1.1.7 Length of time for which the insulating liquid is under stress,

X1.1.8 The temperature of the insulating liquid as it affects the relative saturation level of moisture in solution.

X1.1.9 Gassing tendencies of the insulating liquid under the influence of electric stress,

X1.1.10 Concentration of dissolved gases if saturation levels are exceeded as a result of a sudden cooling or decrease in pressure, which may cause the formation of gas bubbles,

X1.1.11 Incompatibility with materials of construction, and

X1.1.12 Velocity of flow.

X1.1.13 A decrease in dielectric strength of the insulating liquid can have an accentuated effect on the electric creepage strength of the solid insulating materials immersed in the insulating liquid.

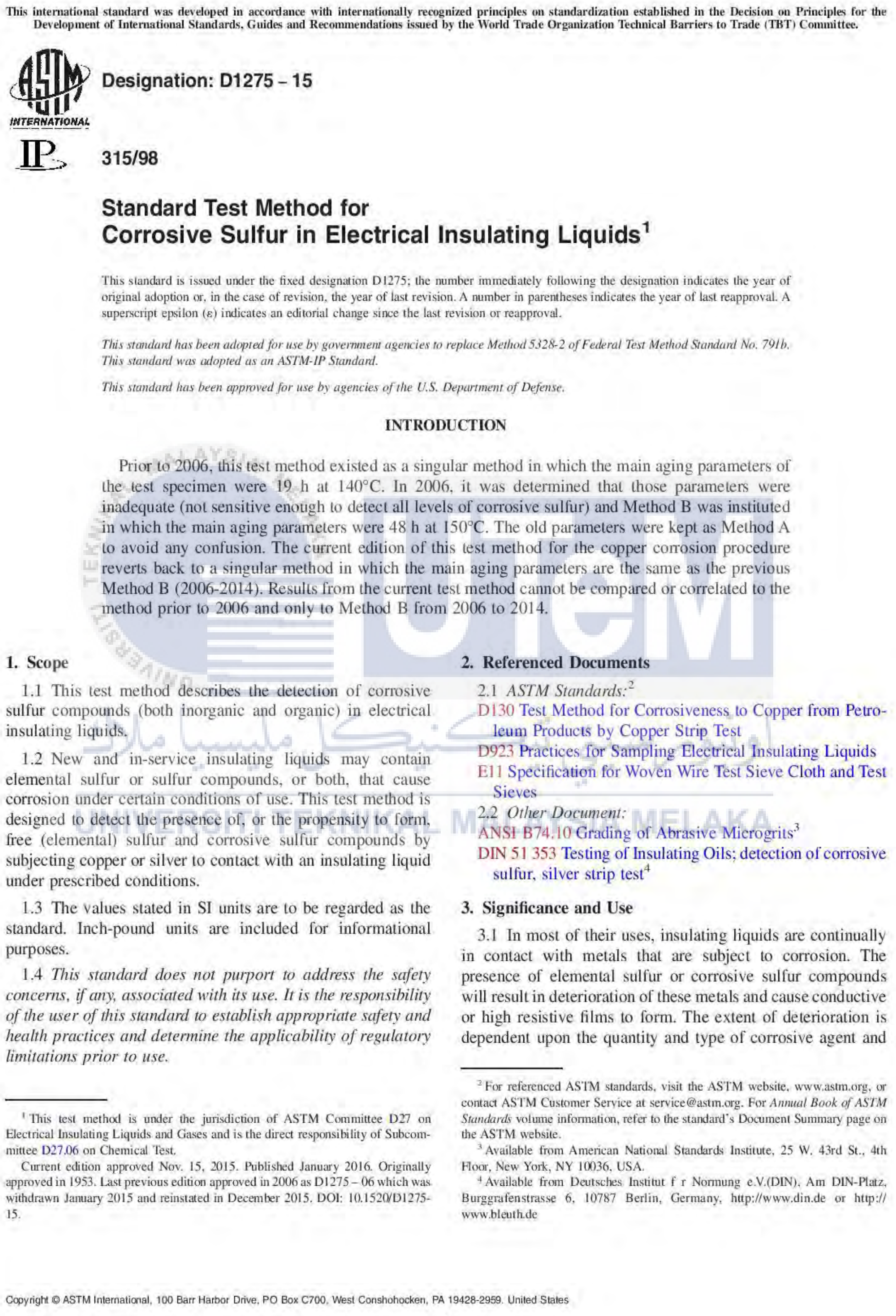
X1.2 Because of the separate, cumulative, and in some cases, interacting effects of the influences listed above, the average breakdown voltage of a liquid as determined by this test method cannot be used directly for design purposes. Procedures utilizing electrode shapes and configurations similar to those used in insulating liquid filled apparatus are used to determine design data and to study the influence of the variables. However, test results utilizing the rounded electrodes in this test method do give an indication of the relative dielectric strength of the insulating liquid in the insulation system.

SUMMARY OF CHANGES

Committee D27 has identified the location of selected changes to this standard since the last issue (D1816-04) that may impact the use of this standard.

(1) Revised title to include other insulating liquids (silicone fluids, natural and synthetic esters).
(2) Changed the volume requirement for the test cell in 6.1.
(3) Added Requirements for handling samples containing free water in 8.1.
(4) Added hold time for testing natural and synthetic esters and high fire point fluids in 9.3.
(5) Removed the standard deviation calculation and added Range calculation in 9.4 and 9.5.
(6) Added Instructions for handling test results when they exceed the allowable range and there is insufficient sample to retest.

APPENDIX G: ASTM D1275



This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D1275 − 15

315/98

Standard Test Method for Corrosive Sulfur in Electrical Insulating Liquids[1]

This standard is issued under the fixed designation D1275; 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 adopted for use by government agencies to replace Method 5328-2 of Federal Test Method Standard No. 791b. This standard was adopted as an ASTM-IP Standard.

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

INTRODUCTION

Prior to 2006, this test method existed as a singular method in which the main aging parameters of the test specimen were 19 h at 140°C. In 2006, it was determined that those parameters were inadequate (not sensitive enough to detect all levels of corrosive sulfur) and Method B was instituted in which the main aging parameters were 48 h at 150°C. The old parameters were kept as Method A to avoid any confusion. The current edition of this test method for the copper corrosion procedure reverts back to a singular method in which the main aging parameters are the same as the previous Method B (2006-2014). Results from the current test method cannot be compared or correlated to the method prior to 2006 and only to Method B from 2006 to 2014.

1. Scope

1.1 This test method describes the detection of corrosive sulfur compounds (both inorganic and organic) in electrical insulating liquids.

1.2 New and in-service insulating liquids may contain elemental sulfur or sulfur compounds, or both, that cause corrosion under certain conditions of use. This test method is designed to detect the presence of, or the propensity to form, free (elemental) sulfur and corrosive sulfur compounds by subjecting copper or silver to contact with an insulating liquid under prescribed conditions.

1.3 The values stated in SI units are to be regarded as the standard. Inch-pound units are included for informational purposes.

1.4 *This standard does not purport to address 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 practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D923 Practices for Sampling Electrical Insulating Liquids

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

2.2 *Other Document:*

ANSI B74.10 Grading of Abrasive Microgrits[3]

DIN 51 353 Testing of Insulating Oils; detection of corrosive sulfur, silver strip test[4]

3. Significance and Use

3.1 In most of their uses, insulating liquids are continually in contact with metals that are subject to corrosion. The presence of elemental sulfur or corrosive sulfur compounds will result in deterioration of these metals and cause conductive or high resistive films to form. The extent of deterioration is dependent upon the quantity and type of corrosive agent and

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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 Nov. 15, 2015. Published January 2016. Originally approved in 1953. Last previous edition approved in 2006 as D1275 – 06 which was withdrawn January 2015 and reinstated in December 2015. DOI: 10.1520/D1275-15.

[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] Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036, USA.

[4] Available from Deutsches Institut für Normung e.V.(DIN), Am DIN-Platz, Burggrafenstrasse 6, 10787 Berlin, Germany, http://www.din.de or http://www.bleuth.de

time and temperature factors. Detection of these undesirable impurities, even though not in terms of quantitative values, is a means for recognizing the hazard involved.

3.2 Two methods are provided, one for copper corrosion and one for silver corrosion. Copper is slightly less sensitive to sulfur corrosion than silver but the results are easier to interpret and less prone to error. The silver corrosion procedure is provided especially for those users who have applications where the insulating liquid is in contact with a silver surface.

4. Apparatus

4.1 *Bath*—A hot-air oven or liquid bath provided with suitable means of heating to, and controlling at 140 or 150 ± 2°C. A circulating hot-air oven is preferred.

4.2 *Bottles*[5]—Heavy-walled, 250-mL, bottles of chemically resistant glass constructed with necks to receive a polytetrafluoroethylene (PTFE) threaded plug equipped with a fluoroelastomer O-ring. Bottles of such capacity and design are required in order to allow sufficient space for expansion of the insulating liquid and to eliminate intrusion from atmospheric gases. Flat bottomed bottles are preferred.

4.3 *Copper Foil, 99.9+ % pure,* 0.127 to 0.254 mm (0.005 to 0.010 in.) in thickness.

4.4 *Silver Foil,* 99.99+ % pure, 0.5 mm thick.

4.5 *Polishing Material,* consisting of 240-grit silicon carbide paper or cloth (refer to Specification E11), and also 230-mesh silicon carbide grains and pharmaceutical absorbent cotton.

NOTE 1—It should be noted that 240-grit silicon carbide paper and 230-mesh silicon carbide grains have particle sizes of about the same size (63 µm). In the United States, abrasive papers are classified in accordance with ANSI B74.10. Abrasive powders are classified by ASTM mesh size.

5. Reagents

5.1 *Acetone,* ACS reagent grade.

5.2 *Hydrochloric Acid*—ACS reagent grade, 36.5 to 38.0 % assay.

5.3 *Nitrogen Gas*—Commercial cylinders of nitrogen gas are satisfactory for this purpose.

5.4 *Suitable Solvent*—technical grade acetone, heptane, hexane and pentane.

5.5 *Water,* distilled.

6. Summary of Test Methods

6.1 *Copper Corrosion*—220 mL of insulating liquid is aged in a sealed heavy-walled bottle for 48 h at 150°C in the presence of a copper strip.

6.2 *Silver Corrosion*—220 mL of insulating liquid is aged in a sealed heavy-walled bottle for 48 h at 150°C in the presence of a silver strip.

7. Preparation of Apparatus

7.1 Chemically clean bottles and PTFE screw plugs with solvents to remove insulating liquid residue, then wash the bottles with a suitable solvent such as heptane or hexane, or us a phosphate-type cleaning powder or liquid. Rinse with tap water, then with distilled water, and dry in an oven. Replace the fluoro-elastomer O-ring before each test to avoid cross contamination.

7.2 Polish a larger piece of copper or silver foil from which, after the final polishing, several strips of the proper size may be cut. Remove blemishes from both surfaces of the copper or silver foils with the 240-grit silicon carbide paper. Cut a strip(s) of copper or silver foil 6 by 25 mm (¼ by 1 in.). Strips may be stored in sulfur-free acetone at this point for future use. Do the final polishing of the strip by removing it from the acetone, holding it in the fingers protected with ashless filter paper or nitrile gloves, and rubbing with 230-mesh silicon carbide grains picked up from a glass plate with a pad of absorbent cotton moistened with a drop of acetone. Wipe the strip with fresh pads of cotton and subsequently handle only with stainless steel forceps (do not touch with fingers). Rub in the direction of the long axis of the strip. Clean all metal dust and abrasive from the strip, using successive clean cotton pads until a fresh pad remains unsoiled. Bend the clean strip in a V-shape at approximately a 60° angle and wash successively in acetone, distilled water, and acetone. Dry in an oven for 3 to 5 min at 80 to 100°C and immediately immerse the copper or silver strip in the prepared test specimen of insulating liquid (Note 2). Do not use compressed air or an inert gas to dry the metal strip.

NOTE 2—This method of cleaning has been adapted from Test Method D130.

8. Copper Corrosion Procedure

8.1 Retrieve a sample for testing in accordance with Practices D923. Use the insulating liquid to be tested as received. Do not filter the insulating liquid.

8.2 Promptly place the prepared copper strip in a clean 250-mL bottle to which has been added 220 mL of the insulating liquid to be tested. Place the bent copper strip standing on its long edge so that no flat surface lies along the glass bottom of the vessel. Bubble nitrogen through the insulating liquid in the bottle by means of a 1.5 mm inner diameter (approximately 1⁄16 in. inner diameter) glass or stainless steel tube connected to the reduction or needle valve of the cylinder (connections must be sulfur-free) for 5 min at a rate of 0.5 L/min. Quickly screw in the PTFE plug equipped with fluoro-elastomer O-ring (Note 3).

8.3 Place the bottle with the test specimen in the oven at 150°C. If needed, after 15 min of heating at 150°C, partially unscrew the PTFE plug to release the pressure and then screw it back down. Remove the bottle after heating for 48 h ± 20 min at 150 ± 2°C. Allow to cool. Carefully take the copper strip from the bottle and wash with acetone or other suitable

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[5] The sole source of supply of the Bottles and PTF screw plugs known to the committee at this time is Prism Research Glass, P.O. Box 14187, Research Triangle Park, NC 27709, part number DOB-B-250. 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. Laboratories already using bottles from Ace Glass can continue.

solvent to remove all of the insulating liquid and let air dry. Do not use pressurized air to dry the copper strip.

8.4 To inspect, hold the test strip in such a manner so that light reflected from it at an angle of approximately 45° will be observed. Interpret and report the results as defined in Sections 9 and 10. If there is difficulty in determining whether the copper strip is considered to be noncorrosive or corrosive, refer to 8.5 and 8.5.1.

8.5 If a specimen is borderline or unclear (between noncorrosive and corrosive), cut the strip in half along the bent portion of the copper strip. Retain one half and treat the other half as described in 8.5.1.

8.5.1 Submerge the half of the copper strip completely in a 1:1 solution of HCl and distilled water at room temperature and wait 20 ± 2 min while gently swirling on occasion. If the deposit in question is still present and did not fade or discolor, then interpret and report the result as corrosive in Sections 9 and 10. If the deposit has been removed or has turned tan/beige, then corrosive sulfur is not present and the result is to be reported as noncorrosive. The tarnish level is then determined on the untreated portion of the copper strip. Report the tarnish level of the untreated portion of the copper strip as listed in 9.1 disregarding the color on the strip that made it unclear in the initial inspection of the strip.

NOTE 3—The corrosive effects of unstable sulfur compounds present in the insulating liquid are reduced when aged under strong oxidizing conditions such as when oxygen is present. It is imperative to keep the bottle well sealed after nitrogen bubbling. If the insulating liquid visibly darkens during testing then most likely oxygen has entered the test bottle and oxidized the insulating liquid. In these cases, the test procedure has become compromised and must be repeated.

9. Interpretation of Results

9.1 Classify the insulating liquid as corrosive or noncorrosive in accordance with Table 1. Classification of corrosive or noncorrosive is to be aided through the use of ASTM Copper Strip Corrosion Standards as referenced in Test Method D130. Often there are several different tarnish levels on a copper strip. In these cases, report the most tarnished classification. A description of the tarnish levels is provided in Table 2 for reference only.

10. Report

10.1 Report the following information:
10.1.1 Sample identification,
10.1.2 Test specimen as being corrosive or noncorrosive,
10.1.3 Tarnish level according to Test Method D130,
10.1.4 HCl treated if the strip was treated per 8.5.1, and
10.1.5 This Method, Copper Corrosion.

TABLE 1 Copper Strip Classifications

Classification	Description
Noncorrosive	Orange, red, lavender, multicolored with lavender blue or silver, or both, overlaid on claret red, silvery, brassy or gold, magenta overcast on brassy strip, multicolored with red and green showing (peacock) but no gray
Corrosive	Transparent black, dark gray or dark brown, graphite or lusterless black, glossy or jet black, any degree of flaking

TABLE 2 Copper Strip Tarnish Level Classifications, Test Method D130

Classification	Designation	Description
Freshly Polished Strip	...	Unable to reproduce upon aging so no description is provided
1	Slight tarnish	a. Light orange, almost the same as freshly polished strip
	...	b. Dark orange
2	Moderate tarnish	a. Claret red
	...	b. Lavender
	...	c. Multicolored with lavender blue or silver, or both, overlaid on claret red
	...	d. Silvery
	...	e. Brassy or gold
3	Dark tarnish	a. Magenta overcast on brassy strip
	...	b. Multicolored with red and green showing (peacock), but no gray
4	Corrosion	a. Transparent black, dark gray or brown with peacock green barely showing
	...	b. Graphite or lusterless black
	...	c. Glossy or jet black

11. Silver Corrosion Procedure

11.1 Using a silver strip, instead of a copper strip, repeat the instructions provided in 8.1 – 8.3.

11.2 To inspect after the 48 h heating, hold the test strip in such a manner so that light reflected from it at an angle of approximately 45° will be observed. Interpret and report the results as defined in Table 3 (Note 4). If there is difficulty in determining whether the silver strip is considered to be noncorrosive or corrosive, refer to 11.3.

11.3 On occasion, it can be difficult to accurately determine the coloration of the silver strip and thus whether or not the insulating liquid specimen is corrosive or noncorrosive. In those cases, use energy dispersive X-ray spectroscopy (EDX or EDS) to determine the concentrations of silver and sulfur on the silver strip.

11.3.1 Mount the silver strip on an aluminum electron microscopy stub with conductive adhesive. Do not coat the silver strip as it is already conductive. Place the silver strip into the sample chamber and apply vacuum in accordance with the manufacturer's instructions.

11.3.2 The following parameters have been found satisfactory when performing the analysis:

11.3.2.1 Working distance: 19 to 30 mm.

11.3.2.2 Variable pressure mode at around 30 Pa or column vacuum at 1.7×10^{-7}.

11.3.2.3 Accelerating voltage: variable around 20.0 kV.

11.3.2.4 Probe current: dependent on manufacturer but around 93 µA.

11.3.2.5 QC on daily basis when instrument is in use, using aluminum, copper or cobalt standards.

TABLE 3 Silver Strip Classifications

Classification	Description
Noncorrosive	Silver, yellow, golden yellow, shades of blue
Corrosive	Grays to black, brownish

11.3.2.6 Perform the analysis on any discolored areas of the silver strip.

11.3.3 If the sulfur concentration of any of the discolored areas tested is 1 weight percent or greater, the result is to be reported as corrosive.

NOTE 4—Table 3 is excerpted from the German Method DIN 51 353.

12. Interpretation of Results

12.1 Classify the insulating liquid as corrosive or noncorrosive in accordance with Table 3.

13. Report

13.1 Report the following information:

13.1.1 Sample identification,

13.1.2 Test specimen as being corrosive or noncorrosive,

13.1.3 Weight percentages of silver and sulfur if applicable, and

13.1.4 This Method, Silver corrosion.

14. Precision and Bias

14.1 No statement is made about either the precision or bias of this test method since the result merely states whether there is conformance to the criteria for success specified in the procedure and is not quantitative.

14.2 Results from repeatability testing from one laboratory are provided in Table 4 and Table 5.

15. Keywords

15.1 copper; corrosion; corrosive sulfur; elemental sulfur; free sulfur; insulating; insulating liquid; mineral; natural ester; oils; petroleum; silicone; silver; sulfur ; synthetic ester

TABLE 4 Repeatability on the Copper Corrosion Procedure, Noncorrosive or Corrosive

Sample	Concentration in Test Specimen <1 mg/kg Elemental Sulfur	Concentration in Test Specimen 8 mg/kg Elemental Sulfur	Concentration in Test Specimen 80 mg/kg dibenzyl disulfide
1	noncorrosive	corrosive	corrosive
2	noncorrosive	corrosive	corrosive
3	noncorrosive	corrosive	corrosive
4	noncorrosive	corrosive	corrosive
5	noncorrosive	corrosive	corrosive
6	noncorrosive	corrosive	corrosive
7	noncorrosive	corrosive	corrosive
8	noncorrosive	corrosive	corrosive
9	noncorrosive	corrosive	corrosive
10	noncorrosive	corrosive	corrosive

TABLE 5 Repeatability on the Copper Corrosion Procedure, Test Method D130 Tarnish Level

Sample	Concentration in Test Specimen <1 mkg/kg Elemental Sulfur	Concentration in Test Specimen 8 mg/kg Elemental Sulfur	Concentration in Test Specimen 80 mg/kg dibenzyl disulfide
1	1b	4b	Did not match Test Method D130 tarnish levels as all strips were gray and flaky
2	1b	4b	
3	1b	4b	
4	1b	4b	
5	1b	4b	
6	1b	4b	
7	1b	4b	
8	1b	4b	
9	1b	4b	
10	1b	4b	

APPENDIX H: ASTM D92

Designation: D 92 – 05

An American National Standard
American Association State
Highway and Transportation Officials Standard
AASHTO No.: T48
DIN 51 376

Designation: 36/84 (89)

Standard Test Method for Flash and Fire Points by Cleveland Open Cup Tester[1]

This standard is issued under the fixed designation D 92; 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.

INTRODUCTION

This flash point and fire point test method is a dynamic method and depends on definite rates of temperature increases to control the precision of the test method. Its primary use is for viscous materials having flash point of 79°C (175°F) and above. It is also used to determine fire point, which is a temperature above the flash point, at which the test specimen will support combustion for a minimum of 5 s. Do not confuse this test method with Test Method D 4206, which is a sustained burning test, open cup type, at a specific temperature of 49°C (120°F).

Flash point values are a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can therefore only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods, or with test apparatus different from that specified.

1. Scope*

1.1 This test method describes the determination of the flash point and fire point of petroleum products by a manual Cleveland open cup apparatus or an automated Cleveland open cup apparatus.

NOTE 1—The precisions for fire point were not determined in the current interlaboratory program. Fire point is a parameter that is not commonly specified, although in some cases, knowledge of this flammability temperature may be desired.

1.2 This test method is applicable to all petroleum products with flash points above 79°C (175°F) and below 400°C (752°F) except fuel oils.

NOTE 2—This test method may occasionally be specified for the determination of the fire point of a fuel oil. For the determination of the flash points of fuel oils, use Test Method D 93. Test Method D 93 should also be used when it is desired to determine the possible presence of small, but significant, concentrations of lower flash point substances that may escape detection by Test Method D 92. Test Method D 1310 can be employed if the flash point is known to be below 79°C (175°F).

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 6.4, 7.1, 11.1.3, and 11.2.4.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
D 140 Practice for Sampling Bituminous Materials
D 1310 Test Method for Flash Point and Fire Points of Liquids by Tag Open-Cup Apparatus
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

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[1] This test method is under the joint jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility. In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP standard in 1965.

Current edition approved June 1, 2005. Published July 2005. Originally approved in 1921. Last previous edition approved in 2002 as D 92–02b.

[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.

***A Summary of Changes section appears at the end of this standard.**

D 4206 Test Method for Sustained Burning of Liquid Mixtures Using the Small Scale Open-Cup Apparatus

E 1 Specification for ASTM Liquid-in-Glass Thermometers

E 300 Practice for Sampling Industrial Chemicals[3]

2.2 *Energy Institute Standard:*[4]

Specifications for IP Standard Thermometers

2.3 *ISO Standards:*[5]

Guide 34 Quality Systems Guidelines for the Production of Reference Materials

Guide 35 Certification of Reference Material—General and Statistical Principles

3. Terminology

3.1 *Definitions:*

3.1.1 *dynamic, adj—in petroleum products,* the condition where the vapor above the test specimen and the test specimen are not in temperature equilibrium at the time that the ignition source is applied.

3.1.1.1 *Discussion*—This is primarily caused by the heating of the test specimen at the constant prescribed rate with the vapor temperature lagging behind the test specimen temperature.

3.1.2 *fire point, n—in petroleum products,* the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a test specimen of the sample to ignite and sustain burning for a minimum of 5 s under specified conditions of test.

3.1.3 *flash point, n—in petroleum products,* the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a specimen of the sample to ignite under specified conditions of test.

3.1.3.1 *Discussion*—The test specimen is deemed to have flashed when a flame appears and instantaneously propagates itself over the entire surface of the test specimen.

3.1.3.2 *Discussion*—When the ignition source is a test flame, the application of the test flame can cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash point and shall be ignored.

4. Summary of Test Method

4.1 Approximately 70 mL of test specimen is filled into a test cup. The temperature of the test specimen is increased rapidly at first and then at a slower constant rate as the flash point is approached. At specified intervals a test flame is passed across the cup. The flash point is the lowest liquid temperature at which application of the test flame causes the vapors of the test specimen of the sample to ignite. To determine the fire point, the test is continued until the application of the test flame causes the test specimen to ignite and sustain burning for a minimum of 5 s.

5. Significance and Use

5.1 The flash point is one measure of the tendency of the test specimen to form a flammable mixture with air under controlled laboratory conditions. It is only one of a number of properties that should be considered in assessing the overall flammability hazard of a material.

5.2 Flash point is used in shipping and safety regulations to define flammable and combustible materials. Consult the particular regulation involved for precise definitions of these classifications.

5.3 Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable material. For example, an abnormally low flash point on a test specimen of engine oil can indicate gasoline contamination.

5.4 This test method shall be used to measure and describe the properties of materials, products, or assemblies in response to heat and a test flame under controlled laboratory conditions and shall not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test method may be used as elements of a fire risk assessment that takes into account all of the factors that are pertinent to an assessment of the fire hazard of a particular end use.

5.5 The fire point is one measure of the tendency of the test specimen to support combustion.

6. Apparatus

6.1 *Cleveland Open Cup Apparatus (manual)*—This apparatus consists of the test cup, heating plate, test flame applicator, heater, and supports described in detail in the Annex A1. The assembled manual apparatus, heating plate, and cup are illustrated in Figs. 1-3, respectively. Dimensions are listed with the figures.

6.2 *Cleveland Open Cup Apparatus (automated)*—This apparatus is an automated flash point instrument that shall perform the test in accordance with Section 11 Procedure. The apparatus shall use the test cup with the dimensions as described in Annex A1 and the application of the test flame shall be as described in Annex A1.

6.3 *Temperature Measuring Device*—A thermometer having the range as shown below and conforming to the requirements prescribed in Specification E 1 or in the Specifications for IP Standard Thermometers, or an electronic temperature measuring device, such as a resistance thermometer or thermocouple. The device shall exhibit the same temperature response as the mercury thermometers.

	Thermometer Number	
Temperature Range	ASTM	IP
−6 to +400°C	11C	28C
20 to 760°F	11F	

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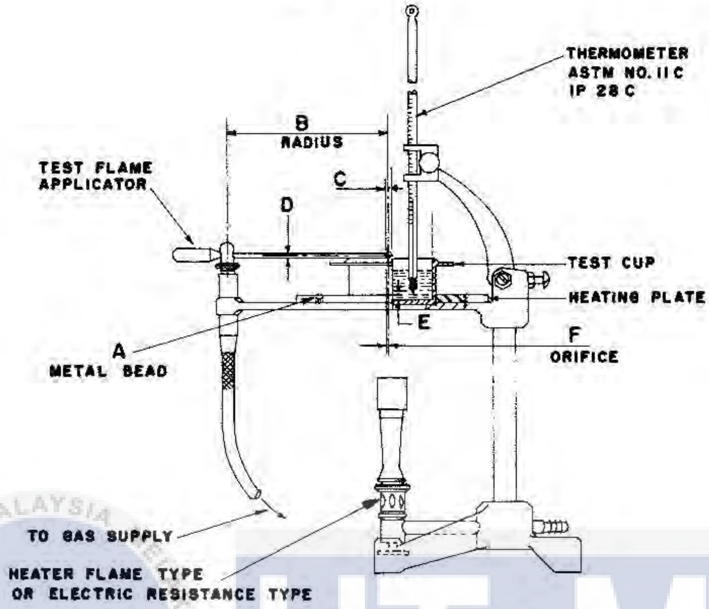
[3] Withdrawn.

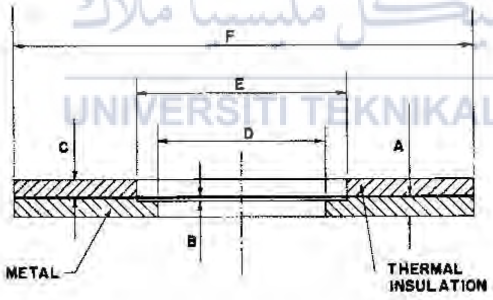
[4] Available from Energy Institute, 61 New Cavendish St., London, W1M 8AR, U.K.

[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.



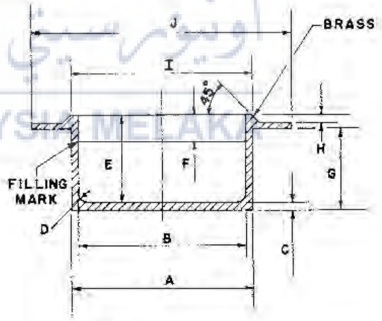
	millimetres		inches	
	min	max	min	max
A—Diameter	3.2	4.8	0.126	0.189
B—Radius	152	nominal	6	nominal
C—Diameter	1.6	nominal	0.063	nominal
D		2		0.078
E	6	7	0.236	0.276
F—Diameter	0.8	nominal	0.031	nominal

FIG. 1 Cleveland Open Cup Apparatus



	millimetres		inches	
	min	max	min	max
A	6	7	0.236	0.276
B	0.5	1.0	0.020	0.039
C	6	7	0.236	0.276
D—Diameter	55	56	2.165	2.205
E—Diameter	69.5	70.5	2.736	2.776
F—Diameter	146	159	5.748	6.260

FIG. 2 Heating Plate



	millimetres		inches	
	min	max	min	max
A	67.5	69	2.658	2.717
B	63	64	2.480	2.520
C	2.8	3.5	0.110	0.138
D—Radius	4	nominal	0.157	nominal
E	32.5	34	1.280	1.339
F	9	10	0.354	0.394
G	31	32.5	1.221	1.280
H	2.8	3.5	0.110	0.138
I	67	70	2.638	2.756
J	97	100	3.819	3.937

FIG. 3 Cleveland Open Cup

6.4 *Test Flame*—Natural gas (methane) flame and bottled gas (butane, propane) flame have been found acceptable for use as the ignition source. The gas flame device is described in detail in Annex A1. (**Warning—**Gas pressure supplied to the apparatus must not be allowed to exceed 3 kPa (12 in.) of water pressure.)

7. Reagents and Materials

7.1 *Cleaning Solvents*—Use suitable technical grade solvent capable of cleaning out the test specimen from the test cup and drying the test cup. Some commonly used solvents are toluene and acetone. (**Warning—**Toluene, acetone, and many solvents are flammable. Health hazard. Dispose of solvents and waste material in accordance with local regulations.)

8. Sampling

8.1 Obtain a sample in accordance with the instructions given in Practices D 140, D 4057, D 4177 or E 300.

8.2 At least 70 mL of sample is required for each test. Refer to Practice D 4057.

8.3 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily; this will prevent loss of volatile material and possible introduction of moisture. Do not make a transfer of the sample unless the sample temperature is at least 56°C (100°F) below the expected flash point. When possible, flash point should be the first test performed on a sample and the sample should be stored at low temperature.

NOTE 3—Typical sample storage temperature is normal room temperature or lower.

8.4 Do not store samples in gas-permeable containers since volatile material may diffuse through the walls of the enclosure. Samples in leaky containers are suspect and not a source of valid results.

8.5 Light hydrocarbons may be present in the form of gases, such as propane or butane, and may not be detected by testing because of losses during sampling and filling of the test cup. This is especially evident on heavy residuums or asphalts from solvent extraction processes.

8.6 Samples of very viscous materials can be warmed until they are reasonably fluid before they are tested. However, no sample shall be heated more than is absolutely necessary. It shall never be heated above a temperature of 56°C (100°F) below its expected flash point. When the sample has been heated above this temperature, allow the sample to cool until it is at least 56°C (100°F) below the expected flash point before transferring.

NOTE 4—Typically, the sample containers for these types of samples will remain closed during the warming process.

8.7 Samples containing dissolved or free water can be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Samples of very viscous materials can be warmed until they are reasonably fluid before they are filtered, but they shall not be heated for prolonged periods or above a temperature of 56°C (100°F) below its expected flash point.

NOTE 5—If the sample is suspected of containing volatile contaminants, the treatment described in 8.6 and 8.7 should be omitted.

9. Preparation of Apparatus

9.1 Support the manual or automated apparatus on a level steady surface, such as a table.

9.2 Tests are to be performed in a draft-free room or compartment. Tests made in a laboratory hood or in any location where drafts occur are not to be relied upon.

NOTE 6—A draft shield is recommended to prevent drafts from disturbing the vapors above the test cup. This shield should cover at least three sides of the test cup vicinity. Some apparatus may already include a built-in draft shield.

NOTE 7—With some samples whose vapors or products of pyrolysis are objectionable, it is permissible to place the apparatus along with a shield into a hood, the draft of which is adjusted so that the vapors may be withdrawn without causing air currents over the test cup during the final 56°C (100°F) rise in temperature prior to the flash point.

9.3 Wash the test cup with the cleaning solvent to remove any test specimen or traces of gum or residue remaining from a previous test. If any deposits of carbon are present, they should be removed with a material such as a very fine grade of steel wool. Ensure that the test cup is completely clean and dry before using again. If necessary, flush the test cup with cold water and dry for a few minutes over an open flame or a hot plate to remove the last traces of solvent and water. Cool the test cup to at least 56°C (100°F) below the expected flash point before using.

9.4 Support the temperature measuring device in a vertical position with the bottom of the device located 6.4 ± 0.1 mm ($\frac{1}{4} \pm \frac{1}{50}$ in.) up from the bottom of the inside of the test cup and located at a point halfway between the center and the side of the test cup on a diameter perpendicular to the arc (or line) of the sweep of the test flame and on the side opposite to the test flame applicator mounting position.

NOTE 8—The immersion line engraved on the ASTM or IP thermometer will be 2 ± 0.1 mm ($\frac{5}{64} \pm \frac{1}{50}$ in.) below the level of the rim of the cup when the thermometer is properly positioned.

NOTE 9—Some automated apparatus is capable of positioning the temperature measuring device automatically. Refer to the manufacturer's instructions for proper installation and adjustment.

9.5 Prepare the manual apparatus or the automated apparatus for operation according to the manufacturer's instructions for calibrating, checking, and operating the equipment.

10. Calibration and Standardization

10.1 Adjust the automated flash point detection system, when used, according to the manufacturer's instructions.

10.2 Calibrate the temperature measuring device according to the manufacturer's instructions.

10.3 Verify the performance of the manual apparatus or the automated apparatus at least once per year by determining the flash point of a certified reference material (CRM), such as those listed in Annex A2, which is reasonably close to the expected temperature range of the samples to be tested. The material shall be tested according to the procedure of this test method and the observed flash point obtained in 11.1.10 or 11.2.5 shall be corrected for barometric pressure (see Section 12). The flash point obtained shall be within the limits stated in Table A2.1 for the identified CRM or within the limits calculated for an unlisted CRM (see Annex A2).

10.4 Once the performance of the apparatus has been verified, the flash point of secondary working standards (SWSs) can be determined along with their control limits. These secondary materials can then be utilized for more frequent performance checks (see Annex A2).

NOTE 10—The verification fluid is a material with a predetermined, interlaboratory tested, flash point temperature that is used to verify proper operation of the apparatus. Calibration is undertaken by the operator according to the apparatus manufacturers' instructions should the result of the verification be outside the stated reproducibility.

10.5 When the flash point obtained is not within the limits stated in 10.3 or 10.4, check the condition and operation of the apparatus to ensure conformity with the details listed in Annex A1, especially in regard to the position of the temperature measuring device, the application of the test flame, and the heating rate. After adjustment of the apparatus, repeat the test with a fresh test specimen (see 10.3) with special attention to the procedural details prescribed in Section 11.

11. Procedure

11.1 *Manual Apparatus*:

11.1.1 Fill the test cup with the sample so that the top of the meniscus of the test specimen is level with the filling mark, and position the test cup on the center of the heating plate. The temperature of the test cup and the sample shall not exceed 56°C (100°F) below the expected flash point. If too much test specimen has been added to the cup, remove the excess using a syringe or similar device for withdrawal of fluid. However, if there is test specimen on the outside of the test cup, empty, clean, and refill it. Destroy any air bubbles or foam on the surface of the test specimen with a sharp knife or other suitable device and maintain the required level of test specimen. If a foam persists during the final stages of the test, terminate the test and disregard any results.

11.1.2 Solid material shall not be added to the test cup. Solid or viscous samples shall be heated until they are fluid before being poured into the test cup; however, the temperature of the sample during heating shall not exceed 56°C (100°F) below the expected flash point.

11.1.3 Light the test flame and adjust it to a diameter of 3.2 to 4.8 mm ($\frac{1}{8}$ to $\frac{3}{16}$ in.) or to the size of the comparison bead, if one is mounted on the apparatus (see Annex A1). (**Warning**—Gas pressure supplied to the apparatus must not be allowed to exceed 3 kPa (12 in.) of water pressure.) (**Warning**—Exercise care when using a gas test flame. If it should be extinguished it will not ignite the vapors in the test cup, and the gas for the test flame that then enters the vapor space can influence the result.) (**Warning**—The operator shall exercise care and take appropriate safety precautions during the initial application of the test flame since test specimens containing low-flash material can give an abnormally strong flash when the test flame is first applied.) (**Warning**—The operator shall exercise care and take appropriate safety precautions during the performance of this test method. The temperatures attained during this test, up to 400°C (752°F), are considered hazardous.)

11.1.4 Apply heat initially at such a rate that the temperature as indicated by the temperature measuring device increases 5 to 17°C (9 to 30°F)/min. When the test specimen temperature is approximately 56°C (100°F) below the expected flash point, decrease the heat so that the rate of temperature rise during the last 28°C (50°F) before the flash point is 5 to 6°C (9 to 11°F)/min.

NOTE 11—With low flash point material or with highly viscous material, it is advised to use the 5 to 6°C/min heating rate from the start of the test to the end.

11.1.5 Apply the test flame when the temperature of the test specimen is approximately 28°C below the expected flash point and each time thereafter at a temperature reading that is a multiple of 2°C. Pass the test flame across the center of the test cup at right angles to the diameter, which passes through the temperature measuring device. With a smooth, continuous motion, apply the test flame either in a straight line or along the circumference of a circle having a radius of at least 150 ± 1 mm (6.00 ± 0.039 in.). The center of the test flame shall move in a horizontal plane not more than 2 mm ($\frac{5}{64}$ in.) above the plane of the upper edge of the test cup and passing in one direction only. At the time of the next test flame application, pass the test flame in the opposite direction of the preceding application. The time consumed in passing the test flame across the test cup in each case shall be approximately 1 ± 0.1 s.

NOTE 12—When determining the flash point of asphalt, it is recommended to carefully move fully to one side, such as with a spatula, any surface film formed before each application of the ignition source. Available data indicate that higher flash point is observed for asphalt samples when surface film formed is not moved aside, compared to the flash point observed when the surface film is moved aside prior to the application of the ignition source.

NOTE 13—An alternative to the moving aside of the formed surface film can be found in Appendix X1.

11.1.6 During the last 28°C (50°F) rise in temperature prior to the expected flash point, care shall be taken to avoid disturbing the vapors in the test cup with rapid movements or drafts near the test cup.

11.1.7 When a foam persists on top of the test specimen during the last 28°C (50°F) rise in temperature prior to the expected flash point, terminate the test and disregard any results.

11.1.8 Meticulous attention to all details relating to the test flame, size of the test flame, rate of temperature increase, and rate of passing the test flame over the test specimen is required for proper results.

11.1.9 When testing materials where the expected flash point temperature is not known, bring the material to be tested and the test cup to a temperature no greater than 50°C (122°F), or when the material required heating to be transferred into the test cup, bring the material to that temperature. Apply the test flame, in the manner described in 11.1.5, beginning at least 5°C (9°F) above the starting temperature. Continue heating the test specimen at 5 to 6°C (9 to 11°F)/min and testing the material every 2°C (5°F) as described in 11.1.5 until the flash point is obtained.

NOTE 14—Flash point results determined in an unknown expected flash point mode should be considered approximate. This value can be used as the expected flash point when a fresh specimen is tested in the standard mode of operation.

11.1.10 Record, as the observed flash point, the reading on the temperature measuring device at the time the test flame causes a distinct flash in the interior of the test cup.

11.1.10.1 The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the entire surface of the test specimen.

11.1.11 The application of the test flame can cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash point and shall be ignored.

11.1.12 When a flash point is detected on the first application of the test flame, the test shall be discontinued, the result discarded, and the test repeated with a fresh test specimen. The first application of the test flame with the fresh test specimen shall be at least 28°C (50°F) below the temperature found when the flash point was detected on the first application.

11.1.13 When the apparatus has cooled down to a safe handling temperature, less than 60°C (140°F), remove the test cup and clean the test cup and the apparatus as recommended by the manufacturer.

NOTE 15—Exercise care when cleaning the apparatus so as not to damage or dislocate the automated flash detection system, when used, or temperature measuring device. See the manufacturer's instructions for proper care and maintenance.

11.1.14 To determine the fire point, continue heating the test specimen after recording the flash point such that the test specimen temperature increases at a rate of 5 to 6°C (9 to 11°F)/min. Continue the application of the test flame at 2°C (5°F) intervals until the test specimen ignites and sustains burning for a minimum of 5 s. Record the temperature of the test specimen when the test flame, which caused the test specimen to ignite was applied. Sustain burning as the observed fire point of the test specimen.

11.1.15 When the apparatus has cooled down to a safe handling temperature, less than 60°C (140°F), remove the test cup and clean the test cup and the apparatus as recommended by the manufacturer.

11.2 *Automated Apparatus*:

11.2.1 The automated apparatus shall be capable of performing the procedure as described in 11.1, including control of the heating rate, application of the test flame, detection of the flash point, or fire point, or both, and recording the flash point or fire point, or both.

11.2.2 Fill the test cup with the sample so that the top of the meniscus of the test specimen is level with the filling mark, and position the test cup on the center of the heating plate. The temperature of the test cup and the sample shall not exceed 56°C (100°F) below the expected flash point. If too much test specimen has been added to the cup, remove the excess using a syringe or similar device for withdrawal of fluid; however, if there is test specimen on the outside of the test cup, empty, clean, and refill it. Destroy any air bubbles or foam on the surface of the test specimen with a sharp knife or other suitable device, and maintain the required level of test specimen. If a foam persists during the final stages of the test, terminate the test, and disregard any results.

11.2.3 Solid material shall not be added to the test cup. Solid or viscous samples shall be heated until they are fluid before being poured into the test cup; however, the temperature of the sample during heating shall not exceed 56°C (100°F) below the expected flash point.

11.2.4 Light the test flame, when necessary, and adjust it to a diameter of 3.2 to 4.8 mm ($\frac{1}{8}$ to $\frac{3}{16}$ in.) or to the size of the comparison bead, if one is mounted on the apparatus. (**Warning—**Gas pressure supplied to the apparatus must not be allowed to exceed 3 kPa (12 in.) of water pressure.) (**Warning—**Exercise care when using a gas test flame. If it should be extinguished it will not ignite the vapors in the test cup, and the gas for the test flame that then enters the vapor space can influence the result.) (**Warning—**The operator shall exercise care and take appropriate safety precautions during the initial application of the test flame since test specimens containing low-flash material can give an abnormally strong flash when the test flame is first applied.) (**Warning—**The operator shall exercise care and take appropriate safety precautions during the performance of this test method. The temperatures attained during this test, up to 400°C (752°F), are considered hazardous.)

NOTE 16—Some automated apparatus can light the test flame automatically and the size of the flame is preset.

11.2.5 Start the automated apparatus according to the manufacturer's instructions. The apparatus shall follow the procedural details described in 11.1.4 through 11.1.15.

12. Calculations

12.1 Observe and record the ambient barometric pressure (see Note 17) at the time of the test. When the pressure differs from 101.3 kPa (760 mm Hg), correct the flash point or fire point, or both, as follows:

$$\text{Corrected flash point} = C + 0.25\,(101.3 - K) \quad (1)$$

$$\text{Corrected flash point} = F + 0.06\,(760 - P) \quad (2)$$

$$\text{Corrected flash point} = C + 0.033\,(760 - P) \quad (3)$$

where:

C = observed flash point, °C,
F = observed flash point, °F,
P = ambient barometric pressure, mm Hg, and
K = ambient barometric pressure, kPa.

NOTE 17—The barometric pressure used in this calculation is the ambient pressure for the laboratory at the time of test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea level readings and would not give the correct reading for this test.

12.2 Using the corrected flash point or fire point, or both, as determined in 12.1, round the values to the nearest 1°C (2°F) and record.

13. Report

13.1 Report the corrected flash point or fire point value, or both, as the Test Method D 92 Cleveland open cup flash point or fire point, or both, of the test specimen.

14. Precision and Bias

14.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results is as follows:

14.1.1 *Repeatability*—The difference between successive results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values in 1 case in 20.

Flash point	8°C (15°F)
Fire point	8°C (15°F)

14.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

Flash point	18°C (32°F)
Fire point	14°C (25°F)

14.2 The precision data for fire point is not known to have been developed in accordance with Precision Manual RR:D02-1007.[6]

NOTE 18—The precisions for fire point were not determined in the current interlaboratory program. Fire point is a parameter that is not commonly specified, although in some cases, this temperature may be desired.

NOTE 19—The precision for asphalt type samples which have had any formed surface film removed has not been determined.

NOTE 20—The precision for asphalt type samples which have utilized the procedure in Appendix X1 have not been determined.

14.3 *Bias*—The procedure of this test method has no bias because flash point and fire point can be defined only in terms of this test method.

14.4 *Relative Bias*—Statistical evaluation of the data did not detect any significant difference between the reproducibility variances of manual and automated Cleveland flash point results for the samples studied with the exception of multi-viscosity lubricating oil and white mineral oil. Evaluation of the data did not detect any significant difference between averages of manual and automated Cleveland flash point for the samples studied with the exception of multi-viscosity lubricating oil, which showed some bias. In any case of dispute, the flash point as determined by the manual procedure shall be considered the referee test.

14.5 The precision data for flash point were developed in a 1991 cooperative test program using seven samples of base oils, asphalt, and lubricating oils. Five laboratories participated with the manual apparatus and eight laboratories participated with the automated equipment. Information on the type of samples and their average flash point are in the research report available at ASTM Headquarters.[7]

15. Keywords

15.1 automated Cleveland open cup; Cleveland open cup; fire point open cup for flash point; flammability; flash point; petroleum products

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1007.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: S15-1009.

ANNEXES

(Mandatory Information)

A1. CLEVELAND OPEN CUP TESTER

A1.1 *Test Cup*, conforming to Fig. 3 with dimensions as shown with the figure. The cup shall be made of brass or other non-rusting metal of equivalent heat conductivity. The cup may be equipped with a handle.

A1.2 *Heating Plate*, shall be of sufficient dimension and materials to ensure that thermal heat to the test cup is only applied to the bottom of the test cup and that extraneous heat to the test cup other than to the bottom is minimized. An example for manual apparatus that utilize Bunsen burners or exposed electric heating elements is shown in Fig. 2.

A1.3 *Ignition Source Applicator*—The device for applying the test flame may be of any suitable type. When using a test flame, it is suggested that the tip be 1.6 ± 0.05 mm ($\frac{1}{16}$ in.) in diameter at the end, and that the orifice be approximately 0.8 ± 0.05 mm ($\frac{1}{32}$ in.) in diameter. The device for operating the test flame applicator may be mounted in such a manner as to permit automated duplication of the sweep of the test flame, the radius of swing being not less than 150 mm (6 in.). The center of the test flame should be supported so that it swings in a plane not greater than 2 mm ($\frac{5}{64}$ in.) above the plane of the rim of the cup. It is desired that a bead, having a diameter of 3.2 to 4.8 mm ($\frac{1}{8}$ to $\frac{3}{16}$ in.), be mounted in a convenient position on the apparatus so that the size of the test flame can be compared to it.

A1.4 *Heater*—Heat may be supplied from any convenient source. The use of a gas burner or alcohol lamp is permitted, but under no circumstances are products of combustion or free flame to be allowed to come up around the cup. An electric heater which can be controlled automatically or controlled by the user is preferred. The source of heat shall be centered under the opening of the heating plate with no local superheating. Flame-type heaters may be protected from drafts or excessive radiation by any suitable type of shield that does not project above the level of the upper surface of the heating plate.

A1.5 *Temperature Measuring Device Support*—Any convenient device that will hold the temperature measuring device in the specified position during a test and that will permit easy removal of the temperature measuring device from the test cup upon completion of a test may be used.

A1.6 *Heating Plate Support*—Any convenient support that will hold the heating plate level and steady may be employed.

A2. VERIFICATION OF APPARATUS PERFORMANCE

A2.1 *Certified Reference Material (CRM)*—CRM is a stable, pure (99 + mole % purity) hydrocarbon or other stable petroleum product with a method-specific flash point established by a method-specific interlaboratory study following RR:D02-1007[6] guidelines or ISO Guide 34 and 35.

TABLE A2.1 D 92 Typical Flash Point Values and Typical Limits for CRM

Hydrocarbon	Purity (mole %)	Flash Point (°C)	Limits (°C)
n–tetradecane	99 +	115.5	±8.0
n–hexadecane	99 +	138.8	±8.0

A2.1.1 Typical values of the flash point corrected for barometric pressure for some reference materials and their typical limits are given in Table A2.1 (see Note A2.2). Suppliers of CRM's will provide certificates stating the method-specific flash point for each material of the current production batch. Calculation of the limits for these other CRM's can be determined from the reproducibility values of this test method, reduced by interlaboratory effect and then multiplied by 0.7 (see Research Report RR:S15-1008[8]).

NOTE A2.1—Supporting data for the interlaboratory study to generate the flash point in Table A2.1 can be found in research report RR:S15-1010.[9]

NOTE A2.2—Materials, purities, flash point values, and limits stated in Table A2.1 were developed in an ASTM interlaboratory program to determine suitability of use for verification fluids in flash point test methods. Other materials, purities, flash point values, and limits can be suitable when produced according to the practices of RR:D02-1007 or ISO Guides 34 and 35. Certificates of performance of such materials should be consulted before use, as the flashpoint value will vary dependent on the composition of each CRM batch.

A2.2 *Secondary Working Standard (SWS)*—SWS is a stable, pure (99 + mole % purity) hydrocarbon, or other petroleum product whose composition is known to remain appreciably stable.

A2.2.1 Establish the mean flash point and the statistical control limits (3σ) for the SWS using standard statistical techniques.[10]

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[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: S15–1008.

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: S15–1010.

[10] *MNL7, Manual on Presentation of Data and Control Chart Analysis*, 6th ed., ASTM International, 1990.

APPENDIX

(Nonmandatory Information)

X1. TECHNIQUE TO PREVENT SURFACE SKIN FORMATION WHEN TESTING FLASH POINT OF ASPHALTS BY TEST METHOD D 92

X1.1 Introduction

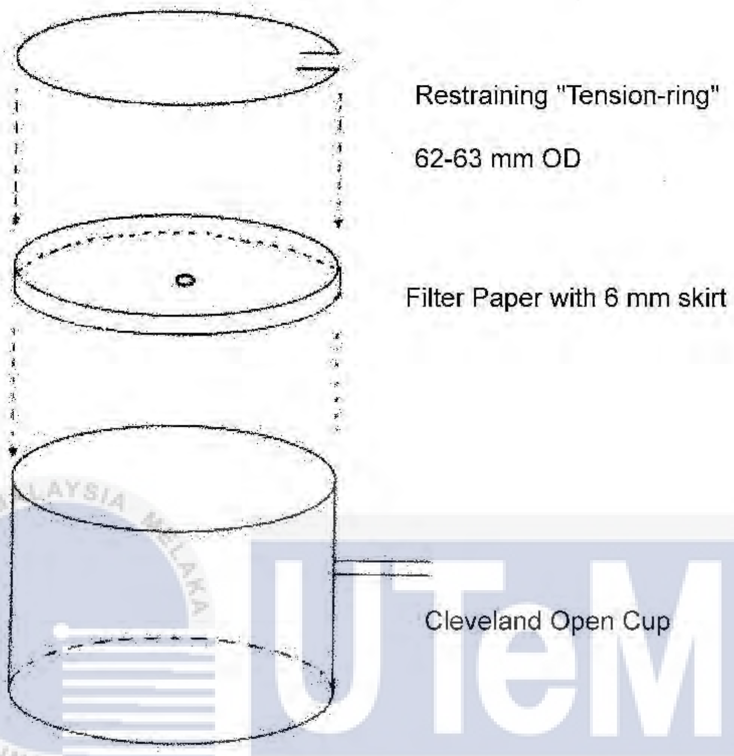
X1.1.1 This technique to prevent surface skin formation when testing flash point of asphalts by Test Method D 92 was developed by Imran Hussami of Frontier El Dorado Refining Company.

X1.2 Materials Required

X1.2.1 *Filter Paper*, qualitative, No. 417 (or equivalent), 7.5 cm diameter.

X1.2.2 *Restraining Tension-ring*, metal wire, circular, but with its 15 mm straight ends folded inwards, parallel to each other (see Fig. X1.1). Dimensions: wire about 2 mm thick, 62 to 63 outside diameter with bent ends 15 mm apart along the circumference of the circle. Total length of wire is about 210 mm. An ordinary metal coat hanger or similar material can be used to make the tension-ring.

X1.2.3 *Single-hole Punch*, (or equivalent) capable of making a 6 mm diameter hole in the center of the filter paper.



NOTE—Use of this alternate technique may cause bubbling in some samples. Bubbling could interfere with automatic flash detection devices, and it also may cause a slower heating rate in some samples.

FIG. X1.1 Technique to Prevent Surface Skin Formation When Testing Flash Point of Asphalts by Test Method D 92

X1.3 Procedure

X1.3.1 Determine the center of the filter paper by means of a ruler. Using the single-hole punch, punch a 6-mm diameter hole *in the center* of the 7.5 cm diameter qualitative filter paper.

X1.3.2 Curl up the sides of the filter paper, about 6 mm all around, and place it in the base of the Cleveland open cup flash point test cup, with the 6-mm skirt facing upward (see Fig. X1.1).

X1.3.3 Place the restraining tension-ring snugly over the curved portion of the filter paper in the base of the cup. (The tension-ring prevents the filter paper from moving upward during the test.)

X1.3.4 Fill the cup with the sample *4 to 5 mm below the filling mark* (this is to compensate for the sample that is absorbed by the filter paper which will be released during the test). (**Warning—**Filling all the way to the filling mark could produce premature flash point results.)

X1.3.5 Start the test either using a manual tester or an automatic unit (following manufacturer's instructions) and determine the flash point.

X1.3.6 Report the flash point corrected for bariometric pressure to the nearest 1°C.

SUMMARY OF CHANGES

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D 92–02b) that may impact the use of this standard.

(*1*) Deleted Note 3 and replace Note 6 with a generic description of a draft shield.

(*2*) Revise 11.1.1 and 11.2.2.

(*3*) Revise 11.1.4 and add Note 11.

(*4*) Revise A1.2.

APPENDIX I: ASTM D2864

Designation: D2864 – 10$^{\varepsilon 1}$

Standard Terminology Relating to Electrical Insulating Liquids and Gases[1]

This standard is issued under the fixed designation D2864; 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.

ε^1 NOTE—The part of speech was added editorially to the terms in January 2012.

INTRODUCTION

The definitions contained in this terminology pertain to terms as they are used in conjunction with fluid insulating materials. Insofar as possible, the definitions are consistent with accepted general usage, and may also contain additional information deemed to be of value in testing of fluid insulating materials.

1. Referenced Documents

1.1 *ASTM Standards:*[2]

D611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents

D2007 Test Method for Characteristic Groups in Rubber Extender and Processing Oils and Other Petroleum-Derived Oils by the Clay-Gel Absorption Chromatographic Method

D2140 Practice for Calculating Carbon-Type Composition of Insulating Oils of Petroleum Origin

D2300 Test Method for Gassing of Electrical Insulating Liquids Under Electrical Stress and Ionization (Modified Pirelli Method)

D2500 Test Method for Cloud Point of Petroleum Products

D3117 Test Method for Wax Appearance Point of Distillate Fuels (Withdrawn 2010)[3]

E355 Practice for Gas Chromatography Terms and Relationships

2. Terminology

ac, *n*—symbol used to designate an electric voltage or current whose amplitude varies periodically as a function of time, its average value over one complete period being zero. One complete repetition of the wave pattern is referred to as a CYCLE, and the number of cycles occurring in one second is called the FREQUENCY, measured in hertz (Hz). For example, the electricity supplied by commercial utility companies in the United States is, in most localities, 60 Hz, although other frequencies may be encountered.

acid treating, *n*—a refining process in which an unfinished petroleum insulating oil is contacted with sulfuric acid to improve its color, odor, stability, and other properties.

ac loss characteristics, *n*—those properties of a dielectric or insulation system (such as dissipation factor, power factor, and loss index) that may be used as a measure of the power or energy losses that would result from the use of such material in an ac electric field.

additive, *n*—a chemical compound or compounds added to an insulating fluid for the purpose of imparting new properties or altering those properties which the fluid already has.

ambient temperature, *n*—the temperature of the surrounding atmosphere as determined by an instrument shielded from direct or reflected rays of the sun.

aniline point, *n*—the minimum temperature for complete miscibility of equal volumes of aniline and the sample under test. See Test Methods D611. In comparing two samples of similar molecular weight, the aniline point can be used as a means of comparing aromatic content of the two samples. A product of high aniline point will be low in aromatics and naphthenes, and therefore high in paraffins.

API gravity, *n*—an arbitrary scale developed by the American Petroleum Institute and frequently used in reference to petroleum insulating oil. The relationship between API gravity and specific gravity 60/60°F is defined by the following:

$$\text{Deg API Gravity at } 60°\text{F} = 141.5/(\text{sp gr } 60/60°\text{F}) - 131.5$$

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[1] This terminology is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gasesand is the direct responsibility of Subcommittee D27.15 on Planning Resource and Development.

Current edition approved May 15, 2010. Published June 2010. Originally approved in 1970. Last previous edition approved in 2010 as D2864 – 10. DOI: 10.1520/D2864-10E01.

[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] The last approved version of this historical standard is referenced on www.astm.org.

aromatics, *n*—that class of organic compounds which behave chemically like benzene. They are cyclic unsaturated organic compounds that can sustain an induced electronic ring current due to delocalization of electrons around the ring.

Discussion—Empirically, the aromatic portion of a mineral insulating oil can be estimated by correlation with physical properties (See Test Method D2140), or by selective adsorption on clay-gel (See Test Method D2007).

askarel, *n*—a generic term for a group of synthetic, fire-resistant, chlorinated aromatic hydrocarbons used as electrical insulating liquids. They have a property under arcing conditions such that any gases produced will consist predominantly of noncombustible hydrogen chloride with lesser amounts of combustible gases.

atomic absorption, *n*—the absorption of radiant energy by ground state atoms. Substances when dispersed as an atomic vapor will absorb characteristic radiations identical to those which the same substances can emit. This property is the basis for analysis by atomic absorption spectroscopy.

capacitivity, *n*—the same as **permittivity, relative.**

color, *n*—a quality of visible phenomena of insulating fluids, the numerical value for which is derived by comparing this quality using transmitted light with that of a series of numbered reference standards.

combustible gases, *n*—flammable gases formed from breakdown (partial or complete) of some insulating materials subjected to electrical or thermal stress, or both.

conductance, *n*—the ratio of the current carried through a material to the difference in potential applied across the material. It is the reciprocal of *resistance* . The unit is: (ohm) $^{-1}$ or siemens.

Discussion—1—**Conductance** is a general term. Specific reference may be made to **conductance dc** and **conductance ac.**

Discussion—2—For dielectrics the conductance may be dependent on the **electrification time.**

conductance, apparent dc, *n*—the dc conductance measured at the end of a specific electrification time. The "apparent dc conductance" is the reciprocal of the "apparent dc resistance." The unit is: (ohm) $^{-1}$ or siemens.

Discussion—The term "apparent dc conductance" is used to distinguish the current-voltage relationship found in electrical insulating materials, where the current (leakage plus absorption) usually decreases with time, from the relationship found in metallic conductors where the steady-state current is reached in a fraction of a second.

conductance, dc, *n*—the ratio of the total current (in amperes) passing through a material to the dc voltage (in volts) applied between two electrodes that are in contact with, or immersed in a specimen. The "dc conductance" is the reciprocal of the "dc resistance." The unit is: (ohm) $^{-1}$ or siemens.

conductivity, *n*—the ratio of the current density carried through a specimen to the potential gradient paralleling the current. This is numerically equal to the conductance between opposite faces of a unit cube of liquid. It is the reciprocal of **resistivity.**

Discussion—1—**Conductivity** is a general term. Specific reference may be made to **conductivity, dc.**

Discussion—2—For dielectrics the conductivity may be dependent on the **electrification time.** (See also **conductivity, apparent dc volume** and **conductivity, dc volume.**)

conductivity, apparent dc volume, *n*—the "dc volume conductivity" measured at the end of a specified electrification time. It is the reciprocal of the apparent dc volume resistivity. The unit most commonly used is: (ohm-centimetre) $^{-1}$ or siemens per centimetre. The SI unit is (ohm-metre) $^{-1}$.

conductivity, dc, *n*—the ratio of the current density passing through a specimen at a given instant of time and under prescribed conditions, to the dc potential gradient paralleling the current. It is the reciprocal of the dc resistivity. In common practice the "dc conductivity" is numerically equal to the "dc conductance" between opposite faces of a centimetre cube of liquid. The unit is: (ohm-centimetre) $^{-1}$ or siemens per centimetre. The SI unit is: (ohm-metre) $^{-1}$.

Discussion—The "dc conductivity" may contain components of both surface conductance and volume conductance, but, in general, surface effects are not common in measurements on fluid dielectrics. The property most commonly measured is either the "dc volume conductivity" or the "apparent dc volume conductivity."

conductivity, dc volume, *n*—the property of a material that permits the flow of electricity through its volume. It is numerically equal to the ratio of the steady-state current density to the steady direct voltage gradient parallel with the current in the material. The dc volume conductivity is the reciprocal of the dc volume resistivity. The unit commonly used is: (ohm-centimetre) $^{-1}$ or siemens per centimetre. The SI unit is (ohm-metre) $^{-1}$.

Discussion—For electrical insulating materials the time required for the steady-state current to be reached may be very long; from several minutes to several months may be required.

corona, *n*—a luminous discharge due to ionization of the air surrounding an electrode, caused by the high electric field strength in the vicinity of the electrode, exceeding a certain critical (that is, threshold) value.

corona effect, *n*—light emitted in the UV range of the electromagnetic spectrum by electronically excited molecules that have reached a singlet state and have not consumed the absorbed energy by other physical process.

corona (partial discharge) inception voltage, *CIV*, *n*—the lowest voltage at which continuous partial discharge (or corona) exceeding a specified intensity is observed as the applied voltage is gradually increased. Where the applied voltage is alternating, the *CIV* is expressed as $1/\sqrt{2}$ of the peak voltage.

corona (partial discharge) extinction voltage, *CEV*, *n*—the highest voltage at which partial discharge (or corona) no longer exceeds a specified intensity as the applied voltage is gradually decreased from a value above the corona inception voltage. Where the applied voltage is alternating the CEV is expressed as $1/\sqrt{2}$ of the peak voltage.

corrosive sulfur, *n*—elemental sulfur and thermally unstable sulfur compounds in electrical insulating oil that can cause corrosion of certain transformer metals such as copper and silver.

dc, *n*—symbol used to designate an electric voltage or current whose amplitude does not vary periodically with respect to time, as for example the output of a chemical cell or that of a thermocouple. The term is also applied to the output of such devices as dynamos and rectifiers, whose amplitude is not strictly time-invariant.

dew-point temperature, *n*—the temperature (above 0°C) to which a gas or vapor must be cooled at constant pressure and constant water-vapor composition in order for saturation to occur.

Discussion—At temperatures below 0°C, vapor may first be noticed in the form of frost. It is common to refer to the temperature at which this occurs as the frost-point temperature. Dew-point temperatures also exist for other gas or vapor systems in which saturation with respect to a substance other than water vapor can occur.

dielectric, *n*—a medium in which it is possible to maintain an electric field with little supply of energy from outside sources. The energy required to produce the electric field is recoverable, in whole or in part. A vacuum, as well as any insulating material is a dielectric.

dielectric breakdown voltage, *n*—the potential difference at which electrical failure occurs in an electrical insulating material or insulation structure, under prescribed test conditions.

dielectric constant, *n*— See **dielectric constant, relative** (especially Note 2). (See also **permittivity** (especially Note 2).)

dielectric constant, absolute, *n*—the same as **permittivity.**

dielectric constant, relative, *n*—the same as **permittivity, relative.**

Discussion—1—Current practice including international usage is to prefer the term *relative permittivity*.

Discussion—2—Common usage has been to drop the term "relative" and simply use dielectric constant when the dimensionless ratio is the quantity being referred to.

dielectric failure, *n*—the failure of an element in a dielectric circuit that exists when the insulating element becomes conducting. This event may take the form of a gradual increase in current exceeding a specified value, but it usually takes the form of an almost instantaneous charge transfer accompanied by collapse of the insulating properties and partial of complete localized destruction of the dielectric medium. In the case of liquids and gases the failure may be self-healing.

dielectric strength, *n*—a property of an insulating material described by the average voltage gradient at which electric breakdown occurs under specific conditions of test.

dissipation factor, *D*, *n*—the ratio of the loss index to its relative permittivity or

$$D = \kappa''/\kappa'$$

It is also the tangent of its loss angle, δ, or the cotangent of its phase angle, θ. The dissipation factor is related to the power factor, *PF*, by the following equation:

$$D = PF/\sqrt{1-(PF)^2}$$

Discussion—It may be expressed as $D = \tan\delta = \text{cotan}\,\theta =$

$$G/\omega C_p = 1/\omega C_p R_p = \omega R_s C_s$$

where G is the equivalent parallel ac conductance, C_p is the parallel capacitance, R_p is the equivalent parallel ac resistance, C_s is the series capacitance, and R_s is the equivalent series resistance.

dissipation factor, dielectric, *n*—same as **dissipation factor.**

dissolved water, *n*—water that is in solution interspersed between molecules of insulating liquid.

electric constant, *n*— the same as **permittivity of free space.**

electric creepage strength, *n*—the average voltage gradient under specific conditions of test and for a specific electrode configuration, at which dielectric failure occurs along the interface between a solid insulating material and the fluid in which it is immersed, or at the interface between two solids that are in close physical contact with each other but are not bonded chemically. Dielectric creepage failure may result in tracking.

electrical discharge, *n*—a discontinuous movement of electrical charges through an insulating medium, initiated by electron avalanches and supplemented by secondary processes.

electrification time, *n*—the time during which a steady direct potential is applied to electrical insulating materials before the current is measured.

emulsified water, *n*—water that is suspended in insulating liquid as clusters of water molecules. It usually gives insulating liquid a milky appearance.

ester-based insulating fluid, *n*—an insulating fluid that may be either synthetic or natural-based. Synthetic ester fluids are produced by the reaction of an organic acid with an alcohol, usually a diol, triol, or tetraol. Natural ester fluids are derived from an animal or agricultural (vegetable or seed) source. They are generically referred to as triglycerides, being a combination of glycerol and various organic acids of varying molecular weights.

fire point, *n*—the lowest temperature at which a specimen will sustain burning for 5 s under specified conditions of test.

flash point, *n*—the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of a test flame causes the vapor of a specimen to ignite under specified conditions of test.

fluorescence, *n*—photoluminescence in which the emitted optical radiation results from direct transitions from a photo–excited singlet energy level to a lower singlet level, these transitions taking place generally within 10 nanoseconds after excitation.

free electrons, *n*—an electron, not directly associated with the structure of an atom or molecule, free to move under the influence of an applied electric or magnetic field.

free radical, *n*—an atom or a diatomic or polyatomic molecule which possesses one unpaired electron. Free radicals act a initiators or intermediates in such basic phenomena as oxidation, combustion, photolysis, and polymerization.

free water, *n*—water that is high enough in concentration to form water droplets and separate from the insulating liquid.

furanic compounds, *n*—a class of chemical compounds characterized by the presence of heterocyclic structures consisting of a five-membered ring containing four carbon atoms and one oxygen atom. These compounds may be found dissolved in electrical insulating fluids, either as residual contaminants of refinery extraction processes in which furfural is used, or from the degradation of cellulose insulation.

gas chromatography, GC, *n*—all chromatographic methods in which the moving phase is gaseous. The stationary phase may be either a dry granular solid or a liquid supported by the granules or by the wall of the column, or both. Separation is achieved by differences in the partition-distribution of the components of a sample between the mobile and stationary phases, causing them to move through the column at different rates and from it at different times. (E355)

gas–liquid chromatography, GLC, *n*—gas chromatographic method utilizing a liquid as the stationary phase, which acts as a solvent for the sample components.

gas–solid chromatography, GSC, *n*—gas chromatographic method utilizing an active (absorbant) solid as the stationary phase.

gassing tendency, *n*—the capability of an insulating liquid either to absorb or generate gases when exposed to voltage stress. The measure of the gassing tendency is the volume of gas evolved or absorbed per unit time by an insulating liquid subjected to electrical stress under prescribed conditions (by Test Method D2300). It is commonly expressed in units of microlitres per minute (μL/min) with a positive value indicating gas is evolved and a negative value indicating gas is absorbed. The SI unit is cubic millimetres per minute (mm^3/min).

DISCUSSION—The term *gassing* is sometimes used synonymously with either *gassing tendency* or *average gassing coefficient*, AGC.

guard electrode, *n*—one or more electrically conducting elements, arranged and connected in an electrical instrument or measuring circuit so as to divert unwanted conduction or displacement currents from, or confine wanted currents to, the measuring device.

hank, *n*—specifically, a coiled or looped bundle (as of yarn, rope or wire) ususally containing a definite aggregate measure of the material.

hydrogen treating, *n*—a refining process in which an unfinished petroleum insulating oil is contacted with hydrogen gas at elevated temperatures and pressures in the presence of a catalyst, to improve its color, odor, stability, and other properties.

inhibitor, *n*—any substance which when added to an electrical insulating fluid retards or prevents undesirable reactions.

insulating liquid, fluid or gas, *n*—a fluid (liquid or gaseous) which does not readily conduct electricity. Electrical insulating fluids typically provide both electrical insulation and heat transfer in electrical equipment.

insulating material, *n*—a material of relatively low electrical conductivity and high dielectric strength, usually used to support or provide electrical separation for conductors, in which a voltage applied between two points on or within the material produces a small and sometimes negligible current.

interfacial tension, *n*—the force existing in a liquid-liquid phase interface that tends to diminish the area of the interface. This force, which is analogous to the surface tension of liquid-vapor interfaces, acts at each point on the interface in the plane tangent at that point. (*Compilation of ASTM Standard Definitions*, 7th Edition.)

loss angle, δ, *n*— the angle whose tangent is the dissipation factor or arctan κ"/κ'. It is also the difference between 90 deg and the phase angle.

loss angle, dielectric, *n*— same as **loss angle.**

loss index, *n*—the same as **loss index, dielectric.**

loss index, dielectric, $\kappa''(\varepsilon_r'')$, *n*—the product of the "relative permittivity" and the dissipation factor and is a measure of the ac dielectric loss. It is also the magnitude of the imaginary part of the "relative complex permittivity."

DISCUSSION—It may be expressed as:

$$\kappa'' = \kappa' D = \text{power loss}/(E^2 \times f \times \text{volume} \times \text{constant})$$

When SI units of watts, volts per metre, hertz, and cubic metres are used the constant has the value: 5.556×10^{-15}. More commonly, when units of watts, volts per centimetre, hertz, and cubic centimetres are used the constant has the value: 5.556×10^{-13}.

loss tangent, *n*—same as **dissipation factor.**

mineral insulating oil, *n*—an oil of mineral origin, refined from petroleum crude oil, possessing electrical insulating properties.

DISCUSSION—Mineral insulating oils are differentiated from oils derived by synthesis, or from animal or vegetable sources.

naphthenic oil, *n*—a term applied to mineral insulating oil derived from special crudes having very low, naturally occurring *n*-paraffin (wax) contents. Such an oil has a low natural pour point and does not need to be dewaxed nor does it usually require the use of a pour depressant.

neutralization value, *n*—a number used as a measure of the acidic or basic constituents present in an insulating liquid, usually expressed in terms of equivalent milligrams of potassium hydroxide per gram of sample.

oxidation inhibitor, *n*—any substance added to an insulating fluid to improve its resistance to deleterious attack in an oxidizing environment. For example, 2,6-ditertiary-butyl paracresol is sometimes added to petroleum insulating oil to improve its oxidation stability.

oxidation life, *n*—a measure of the ability of an insulating liquid to resist oxidation under a prescribed set of conditions. Often the changes in color, neutralization number, interfacial tension, initial appearance of sludge, or rate of sludge formation are the criteria used to measure this quality.

oxidation stability, *n*— see **oxidation life.**

paraffinic oil, *n*—a term applied to mineral insulating oil derived from crudes having substantial contents of naturally occurring *n*-paraffins (wax). Such an oil must be dewaxed and may need the addition of a pour depressant in order to exhibit a low pour point.

partial discharge, *n*—an electrical discharge that only partially bridges the insulation between conductors. It may or may not occur adjacent to a conductor.

permittivity, $\kappa(\varepsilon)$, *n*—a factor giving the influence of an extensive, isotropic dielectric medium on the forces of attraction or repulsion between two electrified bodies (see Discussion 1 of this definition). It is the product of the "relative permittivity," $\kappa'(\varepsilon_r)$, and the "permittivity of free space (vacuum)," $\kappa_o(\varepsilon_o)$.

$$\kappa = \kappa' \cdot \kappa_o$$

The SI unit is: farad per metre (F/m);

where:

farad (F) = s^4A^2/m^2kg,
s = seconds,
A = amperes,
m = metres, and
kg = kilograms.

Discussion—1—The permittivity appears in the equation for Coulomb's law on the forces of attraction of repulsion of point charges:

$$\text{Force} = Q_1 Q_2 / 4\pi\kappa r^2$$

In SI units: Force is in newtons, Q is in coulombs, r is in metres, and κ is in farads per metre.

Discussion—2—In older sources the incorrect term, dielectric constant, and the correct term, absolute dielectric constant, can be found applied to the factor in Coulomb's equation. Modern practice encourages the use of "permittivity."

permittivity, absolute, *n*— the same as **permittivity.**

permittivity, relative, $\kappa'(\varepsilon_r)$, *n*—a dimensionless number, dependent upon the nature and condition of the dielectric medium and upon the frequency of the applied electromagnetic field. For a vacuum $\kappa' \equiv 1.0$. For other dielectrics it is the quotient obtained by dividing the equivalent parallel capacitance, C_p, of a given configuration of electrodes with a material as a dielectric by the capacitance, C_v, of the same configuration of electrodes with vacuum (or air for most practical purposes) as the dielectric:

$$\kappa' = C_p / C_v$$

Discussion—1—Experimentally, vacuum must be replaced by the material at all points where it makes a significant change in capacitance. The equivalent circuit of the dielectric is assumed to consist of C_p, a capacitance in parallel with resistance.

Discussion—The "relative permittivity" is also the real part of the "relative complex permittivity."

Discussion—In common usage the term "relative" is often dropped and the dimensionless ratio is called simply the permittivity. In order to avoid confusion the full term "relative permittivity" should be used.

permittivity, relative complex, $\kappa^*(\varepsilon_r^*)$, *n*—the ratio of the admittance, Y, of a given set of electrodes with a material as dielectric to the admittance, Y_v, of the same configuration with vacuum as dielectric:

$$\kappa^* = Y/Y_v = Y/j\omega C_v = \kappa' - j\kappa''$$

where:

κ' = relative permittivity,
κ'' = dielectric loss index, and
C_v = capacitance with vacuum as the dielectric.

permittivity of free space (vacuum), $\kappa_0(\varepsilon_0)$, *n*—is defined by the following equation derived from wave theory:

$$\kappa_0 = 1/\mu_0 c^2 = 8.854 \times 10^{-12} \text{ farad per metre}$$

where: the magnetic permeability of free space,

$$\mu_0 = 4\pi \times 10^{-7} \text{ henry per metre; 1 henry} = m^2kg/s^2A^2$$

and the speed of electromagnetic waves in free space,

$$c = 2.998 \times 10^{8} \text{ me tres per second.}$$

Discussion—The units and values given above are consistent with current National Institute of Standards and Technology practice and the usage of SI units. Other systems of units exist and are in use: in one, $\kappa_0 = 1.0$ and is dimensionless (electrostatic system) and in another $\mu_0 = 1.0$ and is dimensionless (electromagnetic system). No universal agreement has been reached on this issue.

phase angle, θ, *n*—*(1)* the angle whose cotangent is the dissipation factor, arcot κ''/κ'. (2) The angular difference in the phase between the sinusoidal alternating voltage applied to a dielectric and the component of the resulting current have the same frequency as the voltage.

phase angle, dielectric, *n*— same as **phase angle.**

phase defect angle, *n*— same as **loss angle.**

polybutene oil, *n*—a family of branched-chain polyolefins made by the polymerization of a mixture of iso- and normal butenes. The products are colorless, viscous liquids and are used as cable oils and in capacitors because they are good dielectrics, impermeable to water vapor and gas, and resistant to oxidation. Other synonyms for polybutene are polybutylene, polyisobutylene, and polyisobutene.

polychlorinated biphenyl (PCB), *n*—PCB's belong to a broad family of organic chemicals known as chlorinated hydrocarbons and are produced by attaching one or more chlorine atoms to a biphenyl molecule.

Discussion—For purposes of simplicity, mono-chlorine atoms are included in this definition.

pour point, *n*—the lowest temperature at which a liquid can be observed to flow under specified conditions.

power factor, *PF*, *n*— the ratio of the power in watts, W, dissipated in a material, to the product of the effective values of voltage, V, and current, I, in volt-amperes:

$$PF = W/VI$$

The power factor is related to the **dissipation factor,***D*, as follows:

$$PF = D/\sqrt{1 + D^2}$$

power factor, dielectric, *n*— same as **power factor.**

programmed temperature gas chromatography, PTGC, *n*—utilized changes in column temperature with time.

quality factor, *Q*, *n*— the reciprocal of the dissipation factor (when applied to insulating materials).

reclaiming, *n*—the removal of contaminants and products of degradation such as polar, acidic, or colloidal materials from used electrical insulating liquids by chemical or adsorbent means.

Discussion—Methods listed under *reconditioning* are usually performed in conjunction with reclaiming. Reclaiming typically includes treatment with clay or other absorbents.

reconditioning, *n*—the removal of insoluble contaminants, moisture, and dissolved gases from used electrical insulating liquids by mechanical means.

Discussion—The typical means employed are settling, filtration, centrifugation, and vacuum dehydration or degassing.

relative density (specific gravity), *n*—the ratio of a mass of a given volume of liquid at a given temperature to the mass of an equal volume of pure water at the same temperature. When reporting results, explicitly state the standard reference temperature, for example, relative density 15/15°C.

rerefining, *n*—the use of primary refining processes on used electrical insulating liquids to produce liquids that are suitable for further use as electrical insulating liquids.

Discussion—Techniques may include a combination of distillation and acid, caustic, solvent, clay, or hydrogen treating and other physical and chemical means.

resistance, *n*—the ratio of the potential difference applied to a specimen to the current passed through by the applied potential. It is the reciprocal of **conductance**. The unit is ohm.

Discussion—1—**Resistance** is a general term; specific reference may be made to "dc resistance" and "ac resistance."

Discussion—2—For dielectrics the resistance may be dependent on the **electrification time**.

resistance, apparent dc, *n*—the dc resistance measured at the end of a specified electrification time. The "apparent dc resistance" is the reciprocal of the "apparent dc conductance." The unit is ohm.

Discussion—The term "apparent dc resistance" is used to distinguish the current-voltage relationship found in electrical insulating materials, where the current (leakage plus absorption) usually decreases with time, from the relationship found in metallic conductors where the steady-state current is reached in a fraction of a second.

resistance, dc, *n*—the ratio of the dc voltage (in volts) to the total current (in amperes) carried through the material between two electrodes that are in contact with, or immersed in a specimen. The "dc resistance" is the reciprocal of the "dc conductance." The unit is ohm.

resistivity, *n*—the ratio of the potential gradient paralleling the current passing through the specimen, to the current density. This is numerically equal to the resistance between opposite faces of a unit cube. It is the reciprocal of **conductivity**. The unit commonly used is: ohm-centimetre. The SI unit is ohm-metre.

Discussion—1—**Resistivity** is a general term and could refer to either "dc resistivity or "ac resistivity."

Discussion—2—For dielectrics the resistivity may be dependent on the electrification time. (See also **resistivity, dc volume** and **resistivity, apparent dc volume**.

resistivity, apparent dc volume, *n*—the "dc volume resistivity" measured at the end of a specified electrification time. It is the reciprocal of the "apparent dc volume conductivity." The unit most commonly used is ohm-centimetre. The SI unit is ohm-metre.

resistivity, dc, *n*—the ratio of the dc potential gradient paralleling the current to the current density at a given instant in time and under prescribed conditions. It is the reciprocal of the" dc conductivity." In common practice the "dc resistivity" is numerically equal to the dc resistance between opposite faces of a centimeter cube of liquid. The unit is ohm-centimetre. The SI unit is ohm-metre.

Discussion—The "dc resistivity" may contain components of both surface resistance and volume resistance, but, in general, surface effects are not common in measurements on fluid dielectrics. The property most commonly measured is the "dc volume resistivity" or the" apparent dc volume resistivity."

resistivity, dc volume, *n*—the property of a material that impedes the flow of electricity through its volume. It is numerically equal to the ratio of: the steady direct voltage gradient parallel to the current; to the steady-state current density within the material. It is the reciprocal of the *dc volume conductivity*. In common usage where the voltage gradient is in volts per centimetre and the current density is in amperes per square centimetre, the unit of dc volume resistivity is (ohm-centimetre). The SI unit is ohm-metre.

Discussion—For electrical insulating materials the time required for the steady-state current to be reached may be very long: from several minutes to several months may be required.

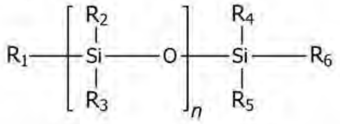
room temperature, *n*—a temperature in the range from 20 to 30°C (68 to 86°F).

Discussion—The term "room temperature" is usually applied to an atmosphere of unspecified relative humidity.

scavenger, *n*—a chemically active substance which consumes or deactivates an undesirable substance in a system or mixture.

shield, *n*—a conductive protective member, partly or wholly enclosing one or more elements of electric equipment or test specimen, for the purpose of reducing or eliminating the electric or magnetic flux, or both, within or beyond that element or elements.

silicone fluid, *n*—a generic term for a family of relatively inert liquid organosiloxane polymers used as electrical insulation. They have the generic formula:



$$R_1-\left[\overset{\displaystyle R_2}{\underset{\displaystyle R_3}{\mathrm{Si}}}-\mathrm{O}\right]_n-\overset{\displaystyle R_4}{\underset{\displaystyle R_5}{\mathrm{Si}}}-R_6$$

where the attached R groups may be H, methyl, vinyl, phenyl, alkyl, or substituted phenyl or alkyl radicals.

solvency, *n*—the ability of a liquid or gas to dissolve a solid, liquid, or gaseous solute.

solvent extraction, *n*—a process used in refining some mineral insulating oils in which an unrefined or a partially refined petroleum distillate is contacted countercurrently with an immiscible solvent so as to selectively remove undesirable materials from the mineral oil.

Discussion—Solvents typically used for mineral insulating oils include phenol, furfural, and liquid sulfur dioxide. Solvent extraction is most frequently used in combination with other refining processes such as hydrotreating, acid-treating, or clay-treating.

specific gravity, *n*—see **relative density**.

specific inductive capacity, *n*—the same as **permittivity, relative**.

storage factor, *n*—a name formerly used at times for the *quality factor*.

synthetic insulating fluid, *n*—an insulating fluid derived from manufactured materials, whose basestock does not occur in nature, or whose basestock is derived through significant processing of petroleum basestocks beyond fractionation, distillation, cracking, and hydrotreating. Examples of synthetic fluids are sulfur hexafluoride, silicone fluids, polybutene oils, synthetic isoparaffin fluids, and synthetic ester-based products.

tan δ (tangent delta), *n*— same as **dissipation factor**.

transformer oil, oxidation inhibited, *n*—a suitably refined mineral insulating oil to which an oxidation inhibitor has been added.

Discussion—It is Canadian practice to consider transformer oils containing 2,6-di-*tert*-butyl-para cresol in concentrations of no more than 0.08 %, by weight, as being uninhibited.

transformer oil, uninhibited, *n*—a suitable refined mineral insulating oil containing no additives and only such as remain in the oil.

Discussion—It is Canadian practice to consider transformer oils containing 2,6-di-*tert*-butyl-para cresol in concentrations of no more than 0.08 %, by weight, as being uninhibited.

viscosity, *n*—same as **viscosity, absolute**.

viscosity, absolute, η, *n*—the ratio of shear stress to shear rate. It is the property of internal resistance of a fluid that opposes the relative motion of adjacent layers. The unit most commonly used for insulating fluids is the centipoise.

viscosity, coefficient of, *n*— same as **viscosity, absolute**.

viscosity, dynamic, *n*— same as **viscosity, absolute**.

viscosity, kinematic, *n*—the quotient of the absolute (dynamic) viscosity divided by the density, η/ρ, both at the same temperature. For insulating liquids the unit most commonly used is the centistokes (100 cSt = 1 St).

viscosity, Saybolt Universal, *n*—the efflux time in seconds of 60 mL of sample flowing through a calibrated Saybolt Universal orifice under specified conditions.

water content,, *n*—the water content of a substance, as measured under specified conditions. (*Compilation of ASTM Standard Definitions*, 7th Edition.)

wax appearance point, *n*—the temperature at which wax or other solid substances first begin to separate from the liquid oil when it is cooled under prescribed conditions (Refer to D3117).

Discussion—Wax appearance is closely related to the *cloud point*observed in Method D2500.

wax content of insulating oil, *n*—the amount of solid material that separates from a mixture of liquid oil and a suitable wax antisolvent (methyl ethyl ketone, sec-butyl acetate, etc.) under prescribed test conditions.

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