WearCheck
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TECHNICAL BULLETIN
ISSUE 85

ESTER-GAZERS: TESTING GUIDELINES FOR NATURAL ESTER OILS IN TRANSFORMERS - part 1

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ABSTRACT

Lubricants can be categorised in many different ways. One of the most common classifications is by the constituent base oil: mineral, synthetic or vegetable. Mineral oil, which is derived from crude oil, can be produced to provide a range of qualities associated with the oil's refining process.

Natural esters, derived from 100% renewable vegetable oils, are superior to mineral oil for use in distribution and power-generation transformers of all voltage classes, both new and retrofilled.

In this Technical Bulletin, Corné Dames discusses the different condition monitoring options for the analysis of transformer component performance where natural esters are used as lubricants in transformers, to determine the degree of contamination by mineral oils and other substances.

1. SOME BACKGROUND INFORMATION ON MIXTURES OF NATURAL ESTER OILS AND OTHER DIELECTRIC FLUIDS

Natural esters and mineral oils are miscible and mostly compatible; they are also compatible with halogenated hydrocarbon insulating fluids. Mixing mineral oil and natural esters may or may not significantly impact the typical properties of impact performance. If the property values change, it may or may not be proportional to the ratio of the content of the fluids.

NOTE: If the purpose of using the natural ester oil is to comply with the National Electrical Code, that would require that less-flammable fluids have an ASTM D92 fire point of not less than 300°C and that the installation complies with all restrictions provided for in the product listing of the fluid.

Too much mineral oil contamination of the natural ester might fail to meet the requirements of the Safety Code. The natural ester manufacturer should be contacted to determine the maximum mineral oil content range, that is allowed, to ensure that flammability parameters are met. Typically, a maximum of 7% mineral oil is acceptable.

As a rule, it is not advisable to mix synthetic esters, synthetic hydrocarbons, and high molecular weight hydrocarbons, although they are miscible. Silicone fluid is not miscible with natural ester oils, so cross contamination should be avoided. Typically, natural esters are miscible with non-flammable halogenated hydrocarbons, like PCBs. This might occur when retro filling older transformers that are filled with this insulating fluid. It would be advisable to consult the manufacturers in such a case

2. LABORATORY SCREENING RECOMMENDATION FOR CLASSIFYING IN-SERVICE NATURAL ESTER OILS

- Visual condition (ASTM D1524)
- Colour (ASTM D1500)
- Dielectric breakdown voltage (ASTM 1816)
- Water content (ASTM D1533)
- AC loss characteristics (dissipation factor) (ASTM D924)
- Fire point (ASTM D92)
- Viscosity (ASTM D445)
- DGA – we will look at the interpretation of these values in a follow-up article

Functional tests, although not required:
- Interfacial tension (ASTM D971)
- Relative density (ASTM D1298)
- Pour point (ASTM D97)
- Volume resistivity (ASTM D1169)
- Neutralisation number (ASTM D664 and ASTM D974)

3. TYPES OF OIL TESTS AND THE SIGNIFICANCE OF EACH TEST

This article is based on reviews of the most commonly applied property tests as per available standards and guides. We will look at how to apply the values of each test to natural ester insulating fluid. The typical values and value limits for new and used ester oils differ primarily from those established for mineral oil.

Due to the inherent differences in chemical, electrical, and physical properties between natural esters and mineral insulating fluids, some standards still need to be updated. For that, some clarification of in-service data is required.

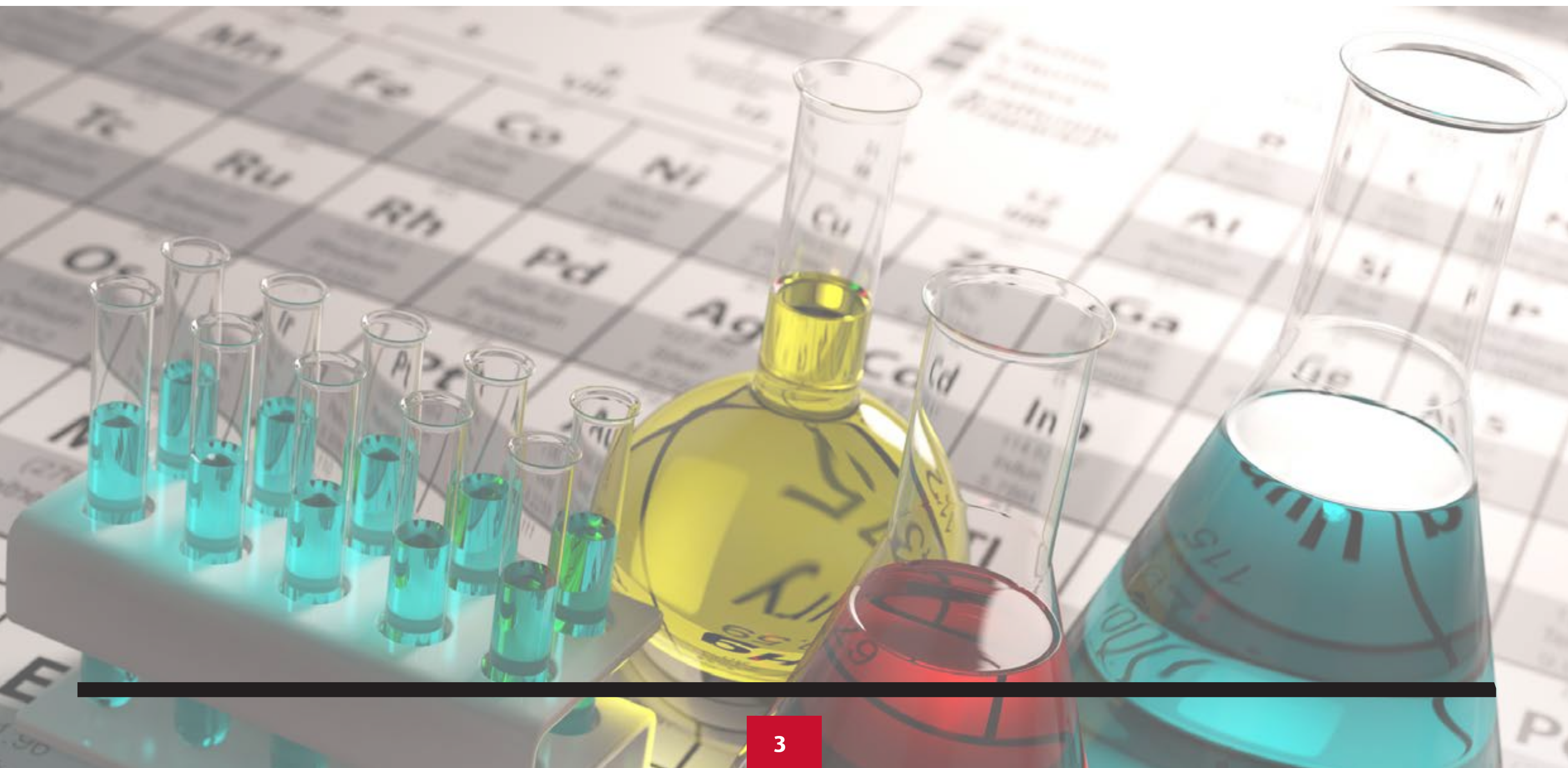
Table 1: Insulating fluid tests that are suitable for natural ester-based dielectric fluids

TEST	ASTM or IEC method number
Practices for sampling	D923
Acid number (Neutralisation)	D664, D974
Dielectric breakdown	D1816
Dielectric breakdown voltage – impulse conditions	D3300
AC loss characteristics – dissipation factor and relative permittivity	D924
Interfacial tension	D971
Colour [a]	D1500
Kinematic viscosity	D445
Flashpoint and Fire point – Cleveland Open Cup Method	D92
Relative Density (specific gravity)	D1298
Pour point	D97, D5949, D5950
Volume resistivity	D1169
Gas analysis	D3284, D3612
Oxidation stability	IEC 61125, Method C[b]
Water content – Karl Fischer Method[c]	D1533
Visual examination of used fluids	D1524
Gassing under electrical stress and ionisation	D2300
Corrosive sulphur test	D1275
Polychlorinated biphenyls (PCBs)	D4059
Furanic compounds	D5837

[a] NE insulating fluids tinted with dye by the manufacturer should not impact the test beyond the 1.0 limit

[b] As modified in IEC 62770, Annex A (reducing the test duration from 164h to 48h)

[c] Alternative reagents as listed in ASTM D1533 for natural esters as modified in IEC 62770, Annex A (reducing test duration from 164h to 48h).



3.1 PRACTICES FOR SAMPLING (ASTM D923)

As with any sample, accuracy is critical to empower the diagnostic services to evaluate the product quality in the sample. Carelessness during the sampling process or contamination of the sample may lead to a misleading result that does not represent the body of fluid being analysed. This can lead to erroneous conclusions and recommendations, which may lead to unmerited expenditure or an incorrect reliability rating of units.

3.2 ACID NUMBER (ASTM 664 AND ASTM 974)

The formation of acidic components in natural esters follows different paths than in mineral oils. These components are mainly produced by hydrolysis, pyrolysis, and oxidation of the natural ester. Before a decision is made to take action due to high acid levels in natural ester oils, it is essential to consider the chemical reactions producing these components and their relative concentrations.

During the hydrolysis of bonds in natural esters, fatty acids are released. These fatty acids formed during the process of hydrolysis are long-chained organic acids. These acids primarily consist of a backbone of 18 carbon molecules, and some 16 carbon chains might also be present. The strength of an organic acid is inversely related to the chain length. Long-chain acids such as stearic acid, C18, are weak, and are not associated with detrimental effects in the transformer system.

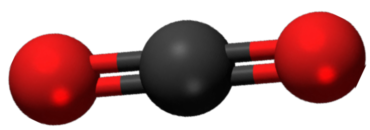
On the other hand, acetic acid (a short-chain acid) in sufficient amounts, can have adverse effects on the condition of other materials that come into contact with it. It has been noted that the acid number increases significantly during the first months of transformer operation. This can be attributed to the hydrolysis reaction due to the insulation paper's moisture. If any dissolved water is present in the fluid, it will also facilitate the hydrolysis reaction.

Breaking of the ester bonds due to pyrolysis will also form fatty acids. The heat in the system that aids in this reaction causes the fatty acids that are formed, to break down even further. As a result, there is an increase in the acid number from this process. The increased acid number during this process corresponds directly to the increased dissolved hydrocarbon and carbon oxide gases. The fatty acids formed during pyrolysis are not detrimental, because they consist of long-chain organic acids.

During the oxidation of ester oils, shorter chain acids are formed. Sites that are easily oxidised produce acids with carbon chains ranging from seven to eleven lengths carbons per chain. It might even be possible that shorter carbon-chain acids are formed. These types of acids formed are of most concern.

It is also possible that acids are introduced into the system due to contamination or other reactions, not including the oil. If significant changes in the acid number are noticed that cannot be linked to hydrolysis, pyrolysis, or oxidation, the cause should be investigated.

ASTM D664 is the preferred method when testing dielectric fluids that have become discoloured, because it uses a potentiometric endpoint rather than a colorimetric endpoint, as in ASTM D974.



3.3 DIELECTRIC BREAKDOWN VOLTAGE (ASTM 1816)

The breakdown voltage of an insulating fluid is essential to measure its ability to withstand electrical stress. The breakdown voltage is the voltage at which breakdown occurs between two electrodes under electrical stress under prescribed test conditions. This test indicates the presence of any contamination (e.g., water, dirt, and other conductive particles in the oil). One or more may be present, leading to low dielectric values.

Particular caution should be taken when filling the test cell with natural ester oil. Guard against trapping air bubbles that can cause misleading low breakdown voltage readings.

An extended sample rest time is required for natural esters because of their higher viscosity, allowing any air bubbles present to escape.

NOTE: ASTM dielectric breakdown voltage test D877 is being eliminated in the ASTM insulating fluid standards and has been eliminated in other IEEE standards and guides.

3.4 AC LOSS CHARACTERISTICS – DISSIPATION FACTOR AND RELATIVE PERMITTIVITY (ASTM D924)

This test describes determining the dissipation factor and relative permittivity of new electrical insulating fluids and oils in service or after service in transformers. The dissipation factor (power factor) measures the dielectric losses in an electrical insulating fluid in an alternating electric field and the energy dissipated as heat. A low dissipation factor indicates low dielectric losses. These losses due to the dissipation factor should not be confused with transformer load and excitation losses.

Transformer load and excitation losses are indicative of the transformer's energy efficiency. The losses associated with the dissipation factor are several orders of magnitude lower than the load and excitation losses.

New natural ester oils have inherently higher dissipation factors than mineral oils. Field data indicates that the rate at which the dissipation factor increases under normal operating conditions is much higher relative to mineral oils. However, additional analysis is required before setting limits for continued service.

Relative permittivity - often referred to as dielectric constant and occasionally as specific inductive capacity (SIC) - is the ratio of the capacitance of a capacitor using the material to be measured as the dielectric to the capacitance of a capacitor with vacuum as the dielectric, both having identical electrodes.

The local voltage stress distribution can be affected by the relative permittivity of materials in contact with each other. The relative permittivity of a new natural ester is inherently higher than those of new mineral oils. The relative permittivity describes the ability to polarise a material subjected to an electrical field. This improves the electrical stress distribution.

3.5 INTERFACIAL TENSION (ASTM D971)

The method covers the measurement, under non-equilibrium conditions, of the interfacial tension of insulating fluids against water.

The surfactant content of an insulating fluids migrates under charge attraction into the water at the interface. This can be measured by the interfacial tension between electrical insulating fluids and water. Surfactants are ionic or polar, soluble contamination, or oils-deterioration products that decrease the interfacial tension value. The polar charges on water molecules attract surfactant species. Water molecules are strongly attracted to one another and require a specific force to break their interfacial tension.

As the surfactants present in the oil are attracted across the oil-water interface, they obstruct some of the water-to-water attractions that weaken the tensile forces of the water interface. The amount of weakening in these forces is determined by the surfactant type. The weakening of the water-to-water attractions is measured as a decrease in interfacial tension value. The interfacial tension is measured in millinewtons per meter (mN/m).

ASTM has not published an acceptance value limit for the interfacial tension of new natural ester oils. Natural ester oils have inherently lower interfacial tension than used mineral oils. We cannot display a value until ASTM has published a limit for the interfacial tension of new natural ester oils.

Additional field data is required before limits for field-aged oils can be established for use as a guide. However, further investigation should be done when there is more than a 40% decrease in the interfacial tension value from the initial transformer sample before energisation.

3.6 COLOUR (ASTM D1500)

A low colour number, of mineral insulating oil, is desirable to permit inspection of assembled apparatus in a tank. New natural ester oils may initially be slightly darker in colour (typically a slight amber appearance) than highly refined new mineral oil. While an increase in colour number during service indicates the oil's deterioration or contamination in mineral oil, this may not be the case for natural ester oils. Other tests (such as dissipation factor and neutralisation number) are better oil deterioration or contamination measures.

NOTE: that natural oil manufacturers may add clear colourants for identification purposes. Such tints should not impact the ASTM colour and visual examinations beyond the 1.0 limit

3.7 KINEMATIC VISCOSITY (ASTM D445)

The viscosity of dielectric coolants within the range of normal operating temperatures is essential because it can impact the cooling and performance of some other transformer components, such as load tap changers (LTCs), which are immersed with the same insulating fluids.

The viscosity of mineral insulating oil and natural ester oils is usually measured by the time of flow of a given quantity of oil under controlled conditions of temperature and pressure. Viscosity is the measure of the resistance of a fluid to flow. Kinematic viscosity is the ratio of the dynamic viscosity of a fluid to its density. Dynamic viscosity is its internal resistance to flow due to some shearing force. It is the resistance to the movement of one layer of fluid over another.

The viscosity at the operating temperatures of electrical insulating fluids influences their heat-transfer properties in natural and forced (pumped) convective flow and, consequently, the temperature rise of operating transformers containing them. Natural esters typically have a higher viscosity than mineral oil. An increase in viscosity over time can indicate excessive polymerisation of natural esters from oxidation, typically due to abnormal exposure to air and heat.

3.8 FLASHPOINT AND FIRE POINT – CLEVELAND OPEN CUP METHOD (ASTM D92)

The lowest temperature at which the vapour pressure is sufficient to form an explosive mixture with air near the surface of the fluid, is called the flashpoint of a flammable fluid. The lowest temperature at which a fluid is heated in an open container and attains sufficient combustible vapours to ignite and sustain a fire for five seconds is called the fire point of a fluid. Contamination with lower flash- and fire-point materials like mineral oil might cause low flash- or fire-point values in natural ester oils.

Results of this test can be used as an element of fire risk assessment that considers other factors pertinent to assessing the fire hazard of a particular end use. This method should only be used to measure and describe the properties of fluids in response to heat and flame under controlled laboratory conditions. It should not describe or appraise fluids' fire hazards or risks under actual fire conditions.

Natural esters have significantly higher flash and fire points than conventional mineral oil. ASTM D6871 requires a minimum 275°C flash point and 300° C fire point per method D92 for new natural ester insulating fluids. We will discuss this in a follow-up article.

3.9 RELATIVE DENSITY (ASTM D1298)

An insulating fluid's relative density (specific gravity) is the ratio of the weights of equal volumes of fluid and water at 15°C (60°F). Relative density is not significant in determining the quality of a fluid. It may be pertinent in determining suitability for use in specific applications. In particularly cold climates, ice may form in equipment exposed to subzero temperatures and may float on fluids that have densities higher than 0.91. Although density measurements are too insensitive to help detect many contamination issues when results are significantly different from the typical value listed by the manufacturer, the cause should be investigated.

3.10 POUR POINT (ASTM D97, ASTM D5949, ASTM D5950)

The pour point is the lowest temperature at which an insulating fluid flows under the prescribed conditions.

The pour point indicates the temperature below which fluid circulation might be difficult, if not impossible. Fluid circulation can be limited by viscosity even above the pour-point temperature. Natural ester oils have a higher viscosity than conventional mineral oil and lower viscosities than HMWH and silicone dielectric fluids at average operating temperatures. For transformers using natural esters with operational (particularly mechanical) internal accessories, a higher minimum temperature may be required before operation than that required for mineral oil.

In addition, natural esters can cease to flow if left standing for long periods at low temperatures, even above their pour-point temperatures established by testing.

The pour point has little significance as far as contamination or deterioration is concerned. Still, it can be helpful for fluid type identification and determining the type of equipment in which it can be used. The pour point and viscosity, along with the range of possible ambient temperatures, should be factored in for the processing equipment (e.g., pumps) selection for reconditioning or reclamation.

3.11 VOLUME RESISTIVITY (ASTM D1169)

A fluid's volume resistivity (specific resistance) is a direct-current measurement of its electrical insulating capability at opposite faces of a centimetre cube. The resistivity in ohms/centimetre (Ω/cm) of a fluid is the ratio of the direct potential gradient in volts/centimetre (V/cm) paralleling the current flow within the sample to the current density in amperes/square centimetre (A/cm^2), at a given instant in time and under prescribed conditions. A lower resistivity typically indicates the presence of conductive contaminants, but the test has not been widely applied to service-aged fluids. New natural esters have inherently lower volume resistivity than mineral oils.

3.12 GAS ANALYSIS (ASTM D3284, D3612)

Abnormal thermal and electrical stresses are the principal cause of excessive gas formation within a transformer. Dissolved gas analysis (DGA) can identify the types and quantities of gases formed in the insulating fluid. The gas-in-oil data characterise the type of faults. It is also possible to determine the severity and progress of a fault as well as the current condition of the transformer. The gases found in the Bucholz relay and gas blankets can also be used for analysis.

While the gases produced in natural ester fluid and cellulose insulated systems are the same as those produced in mineral oil/cellulose systems, the circumstances and quantities in which they are produced are sometimes different. There are three principal differences caused by the chemical structure of these fluids. These are:

- Linolenic acid is a significant component in some natural esters. During the oxidation of linolenic acid, ethane is produced. For example, soybean- and rapeseed-based ester oils have sufficient linolenic acid content to produce measurable amounts of ethane routinely. After the initial operation of a transformer under normal conditions, the ethane levels can rise to a few hundred ppm. This generation is considered "stray gassing" and not a fault condition. Depending on the operating temperatures, ethane generation tends to level off after a few weeks to a few months. Also, exposure to sunlight or fluorescent light can increase the amount of ethane in the insulating fluid. UV exposure should be avoided, particularly for containers of samples before testing. Watch for increases in the other hydrocarbon gases to follow the onset or development of fault conditions.
- Pyrolysis of natural ester oils can produce carbon oxides in proportions that can mask or confuse the carbon oxide production with the production of these gases from the pyrolysis of cellulose.
- Pyrolysis of natural esters produces a different hydrocarbon gas profile than in mineral oil. Heating will produce the same types of hydrocarbon gases, but the proportions will differ from those produced by mineral oil. Watch for concurrent increases in the carbon oxide gases.

The testing methods for natural ester oils are the same methods used for mineral oil. The key ASTM tests related to gases generated in fluid-immersed transformers are as follows:

- Test method for analysing gases dissolved in electrical insulating oil by gas chromatography (ASTM D3612): All dissolved gas methods of ASTM 3612 are suitable. The "head space" DGA technique (ASTM D3612, Method C) results in less column maintenance compared to direct injection (ASTM D3612, Method B) when testing DGA in natural ester oils.
- Practice for combustible gases in the gas space of electrical apparatus using portable meters (ASTM D3284): Testing and field experience indicate that under the same magnitude of electrical overstress, natural esters will typically produce a smaller volume of the gases compared to mineral oil. However, for the same thermal overstress, some natural esters produce significantly larger volumes of the gases than others, depending on the type of ester base used.

There are differences between mineral oil and natural ester gassing tendency, as indicated by ASTM D2300. There are differences in gas solubility coefficients between the various natural esters and mineral oils, and their respective values should be used for data interpretation.

WATCH OUT FOR ESTER-GAZERS PART TWO IN AUGUST 2023!

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About the writer...

Corné Dames is a Transformer consultant. She has 20+ years' experience in the industry, having previously worked as laboratory manager for a major industrial laboratory group, focusing on transformer health. She has been intrigued by transformer chemistry right from the start of her career, particularly in the analysis of test data. Corné has vast practical and theoretical knowledge of reliability maintenance programmes.

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