IEC 60296 (ed. 4) From a Transformer Oil Manufacturer's Perspective

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ABSTRACT

This paper highlights some of the major updates in the latest IEC 60296 (ed. 4, published in 2012), "Fluids for electrotechnical applications – Unused mineral transformer oils for transformers and switchgear" [1], and some of the considerations a transformer oil manufacturer needs to take in order to fully meet the IEC 60296 standard.

1. INTRODUCTION

The IEC 60296 (ed. 4) published in February 2012 replaces the IEC 60296 (ed.3) from 2003 [2]. Since 2003, various findings made an update of the IEC 60296 necessary. Few of them mentioned below:

In 2005, Doble conducted testing for presence of corrosive sulphur in some 500 un-used transformer oils. The tests showed that more than 40 percent of the oils were corrosive as per ASTM 1275A [3] which was the existing test method for corrosive sulphur.

In 2006, the laboratory of Sea Marconi Technologies and the Italian utility Terna, together with Italian and US Universities, identified a single compound, suspected to been added to the oil as an antioxidant, in declared un-inhibited transformer oils [4].

In 2009 Cigre published a report on the copper corrosion problem "Copper sulphide in transformer insulation" [5] and IEC published the corrosive test method IEC 62535 [6], complimenting the already existing corrosive tests DIN 51353 [7] and ASTM 1275B [8].

In 2010 Cigre started the working group A2-40, "Copper sulphide long-term mitigation and risk assessment" [9], and IEC initiated work to establish an official IEC method of detecting Dibenzyl Disulfide [10]. Professor Kapila from the University of Missouri – Rolla presented at the My Transfo 2010 the detection of an undeclared metal deactivator in transformer oils in his presentation *"Rapid and Specific Determination of Additives, Contaminants and By-products in Transformer Mineral Oils with Electrospray – Mass Spectrometry and Tandem Mass Spectrometry"* [11]

In 2011, the Belgian laboratory Laborelec [15] presented a paper on its discovery of an undeclared metal deactivator in transformer oils.

2. Major change of IEC 60296 (ed. 4) versus IEC 60296 (ed. 3)

- interpretation of "un-inhibited" and "inhibited" transformer oil
- Metal passivators / deactivators
- Dibenzyl disulfide
- Other additives
- Corrosive sulfur test

2.1 interpretation of "un-inhibited" and "inhibited" transformer oil

In the previous IEC 60296 (ed. 3) inhibitors improving the oxidation stability of the transformer oil were strictly limited to those described in the IEC 60666 "Detection and determination of specified additives in mineral transformer oils" [13]. No other additives improving the result the oxidation stability were allowed as per the IEC 60296 (ed. 3). If any of the antioxidant of the IEC 60666 were added, the oil should have been declared as either "trace inhibited" or "inhibited" transformer oil depending on the amount of inhibitor used.

In the IEC 60296 (ed.4), the definition of "un-inhibited" and "inhibited" transformer oils is only linked to the antioxidants of the IEC 60666. As before, if any of the antioxidant described in the IEC 60666 are added - the transformer oil should be declared as either "trace inhibited" or "inhibited". Furthermore, in the IEC 60296 (ed. 4) any additives may be used, including those which as per IEC 60296 definition are defined as an antioxidants. The choice of antioxidants is no longer limited to those described in the IEC 60666.

As a consequence, the transformer oil manufacture can add any antioxidant additive that is not described in the IEC 60666 and declare the oil as "un-inhibited", even though the transformer oil contains a synthetic antioxidant.

2.2 Metal Deactivators

The discovery of metal deactivators in some transformer oils on the market [11 and 12] raised concerns. These types of additives could hide potentially corrosive oil and give so called "false negative" results on corrosion tests. These additives additionally sabotage the oxidation test and do not give a fully representative result of the oxidation test, though the known metal deactivators are consumed quickly in service. The use of such additives without declaration and agreement with the buyer of the transformer oil – is a violation of both the previous IEC 60296 (ed. 3) and the IEC 60296 (ed.4).

Chemical literature classifies metal deactivators into two major groups as per their functioning mechanism, which either can be of chelant or of passivating type [19]. The IEC 60296 (ed. 4) describes both *"metal deactivators"* and *"metal passivators"* as examples of *"antioxidant additive"*, but it is important to note that the IEC 60666 can only detects metal passivators, i.e. not metal deactivator of the chelant type.

As per the IEC 60296 (ed.4) the oil shall be tested for "metal passivator additives of IEC 60666". Some of the other metal deactivators found declared or un-declared in transformer oils and that is not described in the IEC 60666, can be detected by independent laboratories such as: Sea Marconi [14] and Laborelec [15].

2.3 Dibenzyl Disulfide Detection

Method for detection of dibenzyl disulfide as per IEC 62535 [6] is added as a compulsory requirement. For more information regarding this additive, see: [4], [16] and [17].

2.4 Other Additives

Known by few and used by even less, in the previous IEC 60296 (ed.3), article 5.4C gave the buyer an option to request the supplier to declare all additives in the oil. In the IEC 60296 (ed.4) all additives shall be declared.

An transformer oil that fully complies with the IEC 60296 (ed.4) shall include the following information on additives in the Product Data Sheet (PDS):

- Antioxidant additives, as per the IEC 60666
- Metal Passivators, as per the IEC 60666
- Dibenzyl disulfide as per the IEC 62535
- Other Additives – shall be declared

It is important to note that as per the IEC 60296 (ed.4), if any additives are added that improve the oxidation stability, i.e. not only those described in the IEC 60666, the transformer oil shall be subject to the 500 hours oxidation test as per IEC 61125 C [18]. Example: any oil containing a metal deactivator, passivating or chelant type.

2.5 Corrosive Sulfur Test

Additionally to the corrosive sulphur test DIN 51353, the IEC 62535 [6] test on "potentially corrosive sulphur" is included in the IEC 60296 (ed. 4). An informative annex has also been added with information on "potentially corrosive sulfur". The annex includes useful information on how to "detect corrosive sulphur compounds in oil containing a metal passivator additive (declared or suspected)," [1]

3. Manufacturing of Transformer Oil Meeting the IEC 60296 (ed.4)

For a mineral transformer oil to meet the IEC 60296 (ed.4) standard: crude oil selection and correct oil fractionation by distillation are important to meet the requested viscosity at 40 and -30 degree Celsius (ISO 3104), sufficient amount of polycyclic aromatic compounds must be removed in order to pass the health requirements (IP 346), corrosive sulphur has to be removed in order to pass the "not corrosive" requirements tested as per IEC 62535 and

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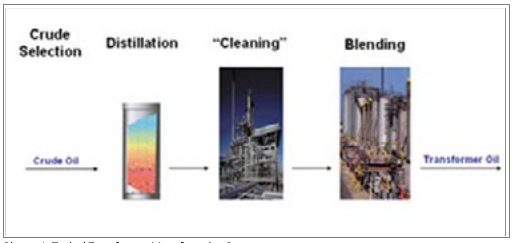
DIN 51353. As per the IEC 60296 (ed. 4), the transformer oil shall meet more than 20 requirements.

To manufacture mineral naphthenic transformer oil that meets the IEC 60296 (ed.4), there are basically four steps as illustrated in picture 1: Crude oil selection, Distillation, Cleaning and Blending. Paraffinic transformer oil additionally needs to undergo a dewaxing process.

3.1 Crude Oil Selection

A typical misunderstanding is that the corrosive behavior of the oil is dependent on the origin of the crude oil. There are several sources of good quality naphthenic crude oil, appropriate for premium transformer oil production, with very different total sulphur content, such a North Sea crude with approximately 0,5 weight percent sulphur and Venezuelan crude with approximately 2,7 weight percent sulphur. But there is no link between transformer oils that are corrosive, i.e. do not pass the corrosive tests of IEC 62535 and the DIN 51353, and the origin of the crude oil. Instead oil found to be corrosive have originated from both low sulphur containing crude to high sulphur level crude oils. So the total sulphur content of a crude oil is not an indicator if the finished transformer oil will be corrosive or not.



Picture 1: Typical Transformer Manufacturing Process.

3.2 Distillation

The distillation of the crude oil is a process where the oil is being separated into different fractions depending on the boiling temperatures. The light fractions, i.e. the gases, distills at a temperature of less than 40 degrees Celsius (<105°F), while the heaviest fractions, i.e. asphalt, would distill at approximately 700 degrees Celsius (1290°F) and above. A typical cut for transformer oil is similar to light gasoil or diesel, i.e. typical 300–370 degrees Celsius (572–698°F).

Table 1 contains the boiling points of certain sulphur compounds. The distillation process generally excludes the lower molecular weight compounds from the transformer oil distillate and it is only the higher molecular weight molecules, such as the substituted dibenzothiophene compounds, that are present in the oil after the distillation process. In the case of benzothiophene or dibenzothiophene compounds, there are substituted hydrocarbon groups attached to the carbons on the base ring structure. As the molecular weight of the molecule increases the boiling point will increase above what is listed in the table. However, the base benzothiophene chemistry is retained.

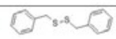
Compound		Boiling Point	Boiling Point
Thiophene		83 °C	183°F
Diethylsulfide	$(C_2H_5)_2S$	92 °C	198°F
Thiophenol		169°C	336°F
Benzothiophene		221°C	430°F
Dibenzyl-disulfide		270°C	518°F
Dibenzothiophene		332°C	630°F

Table 1: Boiling Point for certain sulphur compound. Source: Ergon and Albermarle

3.3 Cleaning by means of Hydrotreatment

The transformer oil distillate is not acceptable as transformer oil as it will not pass the IEC 60296 (ed.4) requirements. The distillate stock still contains sulphur compounds that are corrosive at typical conditions found in a transformer in service and the oxidation stability of the distillate is not sufficient and would not pass the oxidation test IEC 61125 C [18] required as per the IEC 60296 (ed.4).

There are several methods used for cleaning the transformer oil distillate but most commonly, used by the major transformer oil manufacturers, is hydrotreatment (also sometimes called hydrofinishing or hydroprocessing). Hydrotreatment (see picture 2) is a process where unstable molecules such as those containing oxygen, nitrogen, sulphur, and metals are removed and olefins and aromatics are saturated. By removing these unstable molecules and saturating the carbon-carbon double bonds, we are eliminating the risk of getting an oil that is dangerous for health (by removing the polycyclic aromatic compounds) and eliminating the possibility of adverse reactions that could happen under the typical operating conditions found in a transformer. During the hydro-treatment process the

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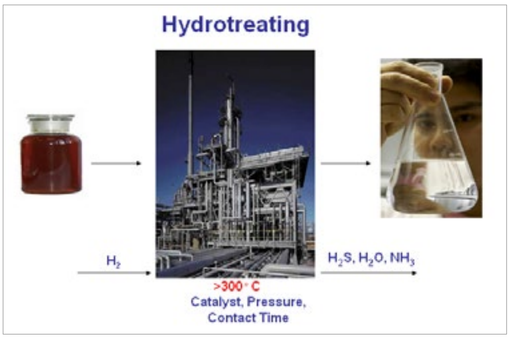
oil is exposed to high pressure, approximately 2000 PSIG (~140 bar), high temperature up to 385 degrees Celsius (725°F), plenty of hydrogen to feed chemical reactions and a nickel-molybdenum or cobalt-molybdenum catalyst tailor made for removal of reactive compounds.



Picture 2: Cleaning the Transformer Oil Distillate by Hydrotreating

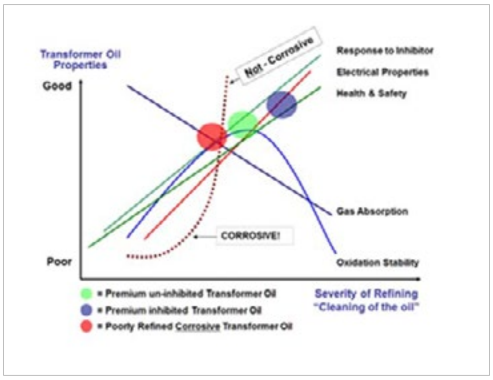
The severe conditions found in the hydro treatment process, you will never find in a transformer even under the most severe conditions, see table 2, and the sulphur containing compounds that would survive the hydrotreatment process and end up in the finished transformer oil are very few (typically 25-150 ppm), stable and non-corrosive, i.e. they easily pass the DIN 51353 and the IEC 62535 required by the IEC 60296 (ed.4).

Parameter	Hydrotreating Process	Power Transformer
Pressure	261 KPA 1800 PSIG	10 - 100 KPA 1,5 - 15 PSIG
Temperature	316° - 385°C 600°-725°F	Typical ~75°C / 167°F
Hydrogen	~95% by volume	ppm by volume
Catalyst	Ni-MO or Co-MO	Copper

Table 2: Typical values for hydrotreating process versus a power transformer

Picture 3 illustrates the balance of various parameters the transformer manufacturer needs to take into consideration when producing transformer oil meeting the IEC 60296 (ed.4). As seen from the picture 3: all parameters, with the exception of gassing tendency and oxidation stability, are improved by a more severe refining, i.e. more severe hydro treatment / cleaning of the oil. With more severe cleaning, the ability of the oil to absorb gasses decreases due to the reduction of aromatic carbons. There is an optimum point where maximum oxidation stability is achieved by keeping enough stable sulfur and nitrogen compounds in the oil.

The optimum point of maximum oxidation stability is of crucial importance when manufacturing an un-inhibited transformer oil meeting the IEC 60296 (ed.4), i.e. an oil that will pass the 164 hours oxidation stability test as per IEC 61125 C [18] without the use of additives such as peroxide decomposers, metal passivators and deactivators that all have an impact on the oxidation test.



Picture 3: Cleaning the Transformer Oil Distillate by Hydrotreating

3.4 Blending

The blending can be either adding different streams of the refining process or adding additives such as the ones described by IEC 60666 for a trace or inhibited type transformer oil. As described in 3.4 "Other Additives", the IEC 60296 (ed.4) open up for the use of any additives, including additives not described in the IEC 60666, that improves the oxidation stability - as long as they are declared in the product data sheet and certificates of compliance.

4. Conclusions and Recommendations

There are major changes in the IEC 60296 (ed. 4) from 2012 compared to previous IEC 60296 (ed. 3) from 2003. The interpretation of un-inhibited and inhibited transformer oils puts more focus on the use of additives in transformer liquids.

To secure premium quality, it is important that users of transformer liquids request that the supplier fully meet the IEC 60296 (ed.4) – including the full declaration of additives in the Product Data Sheet (PDS).

This paper highlights some of the major changes in the IEC 60296 (ed.4) but it is not a substitute for the IEC 60296 (ed.4). It is therefore highly recommended that the user downloads the latest IEC 60296 from the IEC.

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