

## eraspec oil – Applications and methods for determining the condition of insulating liquids with FTIR spectroscopy



### Introduction – Electrical grids

Electrical grids serve as the backbone of modern society, facilitating the transmission and distribution of electricity to homes, businesses, and industries. They play a crucial role in ensuring the reliable and continuous supply of power, essential for sustaining daily activities and driving economic growth.



Preventive maintenance of transformers within these grids is paramount for several reasons. Transformers are critical components that facilitate the efficient transmission of electricity, stepping up or down voltage levels as needed. Regular maintenance helps identify and address potential issues before they escalate, ensuring optimal performance and minimizing the risk of costly downtime or failures.

Insulation liquids, such as transformer oil, are vital for preserving the integrity and functionality of transformers. These liquids serve as both insulators and coolants, helping dissipate heat and prevent electrical breakdown. Monitoring the condition of insulation liquids is essential to detect any degradation or contamination, which could compromise transformer performance and safety. Regular testing and analysis enable timely replacement or remediation, safeguarding the reliability and longevity of transformers within the electrical grid. Depending on the specific application, crucial transformers contain between 2,000 and 100,000 L of insulation liquid.

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### Classification of insulating liquids



Classic insulating liquids, which are also often referred to as transformer oils or insulating oils, are based on mineral oil and are produced from crude oil in various refining steps. Basically, these are relatively low-viscosity, highly refined products in which an attempt is made to remove as many impurities as possible and to saturate the chemical structures as much as possible. The chemical composition of the hydrocarbons plays a major role and is influenced by the type of crude oil and the production process. This has an impact on ageing resistance, dirt holding capacity, low-

temperature properties and dielectric properties. For this reason, the industry is very interested in the

chemical composition of the hydrocarbons, which is mainly the relationship between aromatic, naphthenic and paraffinic structures.

Insulating oils are essentially divided into the groups of inhibited and uninhibited products. Approx. 0.3 % to 0.4 % phenolic antioxidant is added to the inhibited products. Non-inhibited products usually have a slightly higher natural sulfur content, which ensures intrinsic oxidation resistance. The product classification of new and recycled transformer oils is based on IEC 60296 or ASTM D3487.

With ester-based insulating fluids, there is a new group of products entering the market. A distinction is made between "natural esters", which have similar base materials to biodiesel, and "synthetic esters", which are produced using a petrochemical process. In comparison to classic insulating oils, poorer properties have to be accepted in many areas, but with a significantly higher flash point of over 300°C there is a clear advantage in the area of fire protection and its risk assessment. The properties of synthetic esters are regulated in IEC 61099 and the definitions of natural esters can be found in IEC 62770 and ASTM D6871.

## Measurement of insulation liquids during maintenance and other events

The standard IEC 60422 provides monitoring guidance and procedures that are required for the use and maintenance of mineral insulating oils and other hydrocarbon-based liquids in transformers and other electrical equipment.

When regularly testing mineral insulating oils in service, it is important to examine not only the dielectric properties but also the chemical properties such as the acid content or the concentration of antioxidant "inhibitors". The presence of passivating agents must also be monitored if this applies to the transformer in question.

The decrease of these substances over time is a very important aspect for the evaluation of the electrical system and often leads to corrective actions.



### Stages of interest

Getting robust information about the insulation liquids is of interest at different stages during the lifetime of an insulation liquid:

1. Quality and process control carried out by the manufacturer
2. Incoming quality control undertaken by the producer or consumer
3. Initial testing after filling and/or energizing of the transformer
4. Preventive maintenance during the lifetime of the transformer
5. Special occasions like reclaiming, refilling or post-mortem projects
6. For the stage of recycling or upcycling

During trading, shipment and storage the aspect of fingerprinting is of high importance to trace potential contamination and to identify accidental mix-ups.

Typically, measurements are performed during production of the equipment, during the process of the factory acceptance and after initially energizing the unit. Further events like the drying or the reclaiming of insulation liquids often trigger the need for checking the quality of these liquids.

For the most important stage of preventive maintenance, depending on the category and rating of electric equipment, typical sampling frequencies are between once per year or every second year but might be more often in case of a higher risk rating.

### Who is measuring?

Although samples are usually taken by the operator of the electrical system during maintenance, the properties are often determined in specialized and independent test laboratories. In addition to this established setup, there are good reasons for different industries to build up the most important measurement technology in-house. A potential reduction in costs, the acceleration of processes or more flexibility are important reasons for the decision to have measurement capabilities in-house. Modern instruments like the ERASPEC OIL are easy to use and offer a clear advantage for all user groups.

Possible user groups or industries are the following:

- Utilities operating the transmission or distribution grid
- Electric power utilities producing the electric power
- Producers of electrical equipment such as transformers, switch gears, reactors, measurement transformers or tap-changers
- Service providers for drying, reclaiming or filtering of insulation liquids
- Producers or traders of insulation liquids
- Manufacturing and industrial facilities using transformers and electrical equipment

### Maintenance guideline IEC 60422

The maintenance guide IEC 60422 provides monitoring guidance and procedures that are required for the use and maintenance of mineral insulating oils and other hydrocarbon-based liquids in transformers and other electrical equipment. The frequency of testing as well as recommended parameters are mentioned according to the category of equipment and the voltage rating. There are also maintenance guidelines for natural and synthetic esters, but these are not the subject of this document.

Very relevant for the discussion of results for mineral transformer oils is the information shown in Table 1 which defines the conditions and limits for properties which have been discussed in this document.

Property	Unit	Condition and Limits		
		Good	Fair	Poor
Water content	mg/kg	< 15	15 to 20	> 20
Acidity	mg KOH/g	< 0.10	0.10 to 0.15	> 0.15
Inhibitor content	% original value	> 60 %	40 % to 60 %	< 40 %
Passivator content	mg/kg	> 70	50 to 70	< 50

Table 1: Conditions and limits according to IEC 60422 for Category A, power transformers with a rating above 170 kV

Based on these conditions, corrective measures such as refilling inhibitors or passivators or even cleaning or changing the insulating oil are recommended. Therefore, quick and reliable information on the condition of the transformer oil is important and time-critical.

As the focus in these industries is generally not on the operation of laboratory measuring devices, the solution from eralytics can be a great asset to such companies in setting up a monitoring solution, allowing them to benefit from the strengths of eralytics analyzers. The rugged design of the devices in combination with their easy operation and accurate results make them an ideal partner. One solution to cover important parameters according to IEC 60422 is the FTIR spectrometer ERASPEC OIL.

## ERASPEC OIL – The latest trend for transformer oil monitoring

ERASPEC OIL is a portable stand-alone infrared analyzer for high-speed transformer oil analysis and condition monitoring in full compliance with IEC 60666 methods. Its patented interferometer design delivers laboratory-grade results directly on site. No special calibration or configuration is required as the analyzer delivers requested parameters out of the box:

- Inhibitor content
- Passivator content

Also the determination of unwanted contamination such as silicone oil or various types of ester can be measured down to very low limits of detection. The spectrum of applications is rounded off with the possibility to measure aromatic content according to IEC 60590 and water above a certain threshold.

As shown in Figure 1, around 10 mL to 20 mL of sample is automatically inserted from a beaker. Once the spectrum has been determined, it is automatically analyzed, and after only 120 seconds the numeric results are directly displayed on the large touchscreen. Typically, no solvent is required as the new sample displaces the previous sample.



Figure 1: ERASPEC Oil with the manual feeding of a sample

### Discussion of measurement results for typical parameters

Based on the specific spectrum, ERASPEC OIL provides requested parameters automatically on the screen and enables numerous types of export. Additionally, the analyzer allows the examination of the measured spectra to support the understanding of the processes. In Figure 2 a typical naphthenic inhibited and an uninhibited mineral oil insulation liquid are shown.

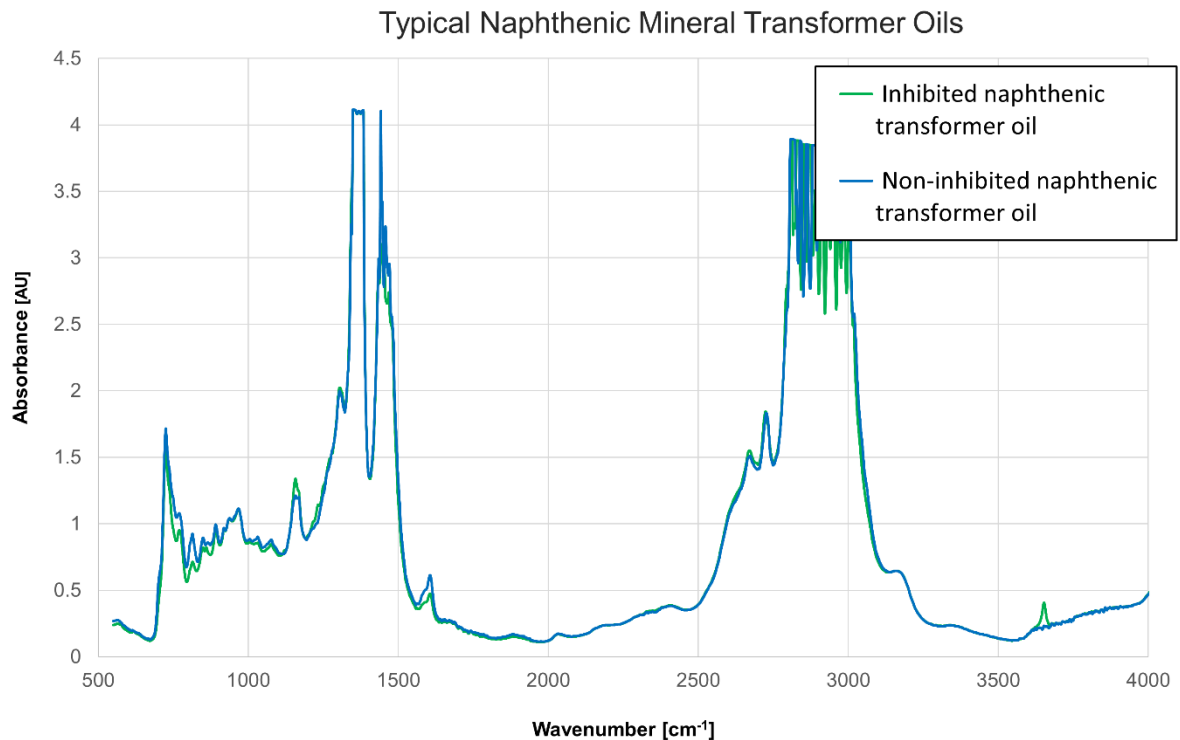


Figure 2: Spectrum of typical naphthenic insulation liquids

At first glance they might look very similar, but significant differences appear by looking at wavelength  $3650\text{ cm}^{-1}$  where the response of the inhibitor is located and at  $1650\text{ cm}^{-1}$ , which is the region for aromatic components. Some other regions appear different and this is used for fingerprinting but not used to determine any specific measure.

Another common insulation liquid has been added in Figure 3. Due to its isoparaffinic nature the missing aromatics show no response at the region sensitive for this chemical structure.



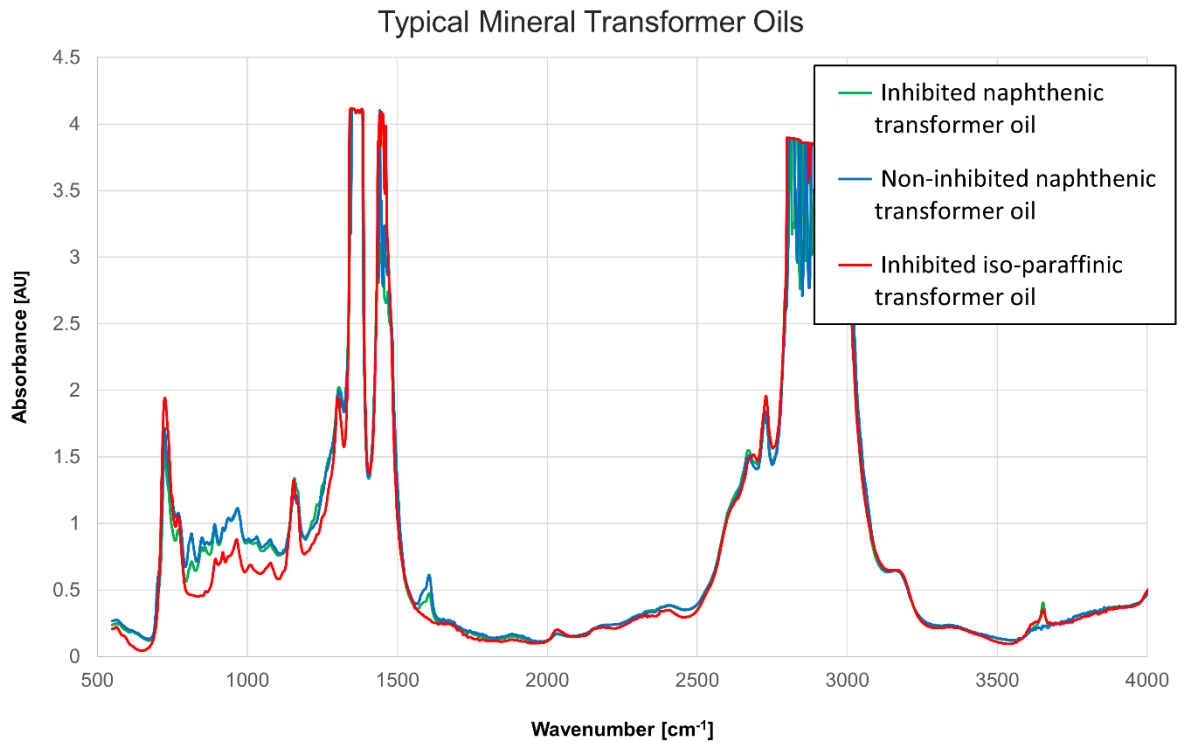


Figure 3: Naphthenic insulation oils compared to iso-paraffinic liquids

By zooming in, differences between the individual materials can be seen easier as shown in Figure 4. Based on specific calibrations the peak heights are transposed to specific values which have been summarized in Table 2.

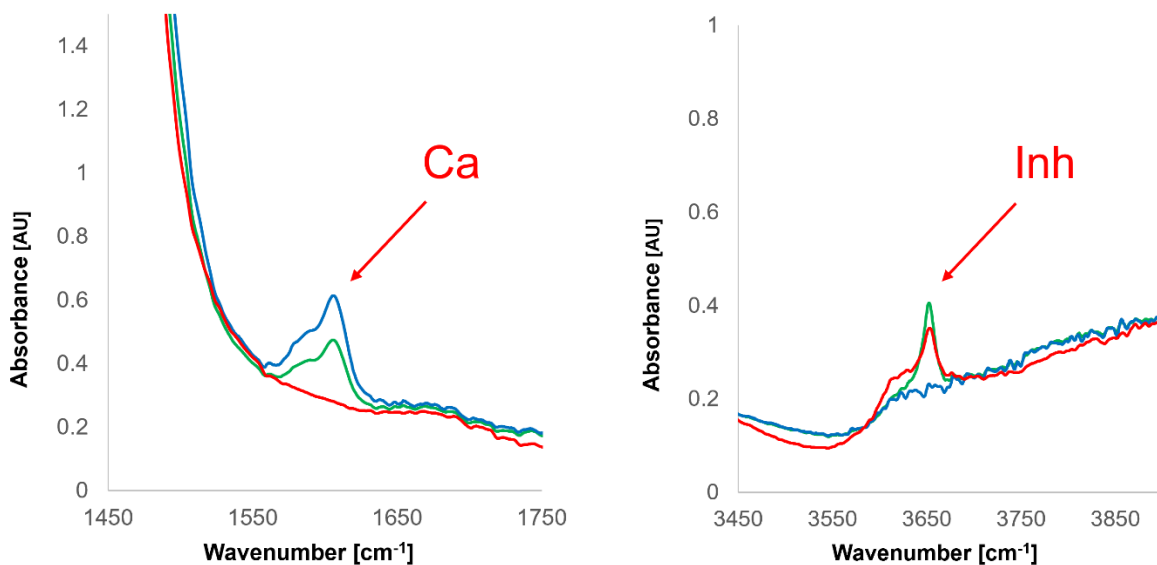


Figure 4: Details of spectra at regions showing aromatic content (Ca) and inhibitor content (Inh).

Type of insulation liquid	Inhibitor content [%]	Aromatic content [%]
Naphthenic inhibited	0.38	5.5
Naphthenic uninhibited	0.0	6.5
Isoparaffinic inhibited	0.35	0.0

Table 2: Summarizing the results based on stored calibration

In comparison to classic insulation liquids produced from crude oil or liquefied from natural gas, synthetic and natural ester liquids show a significantly different spectrum. The reason for this is a different chemical structure dominated by double oxygen bonds. In figure 5 an example is displayed which shows the differences

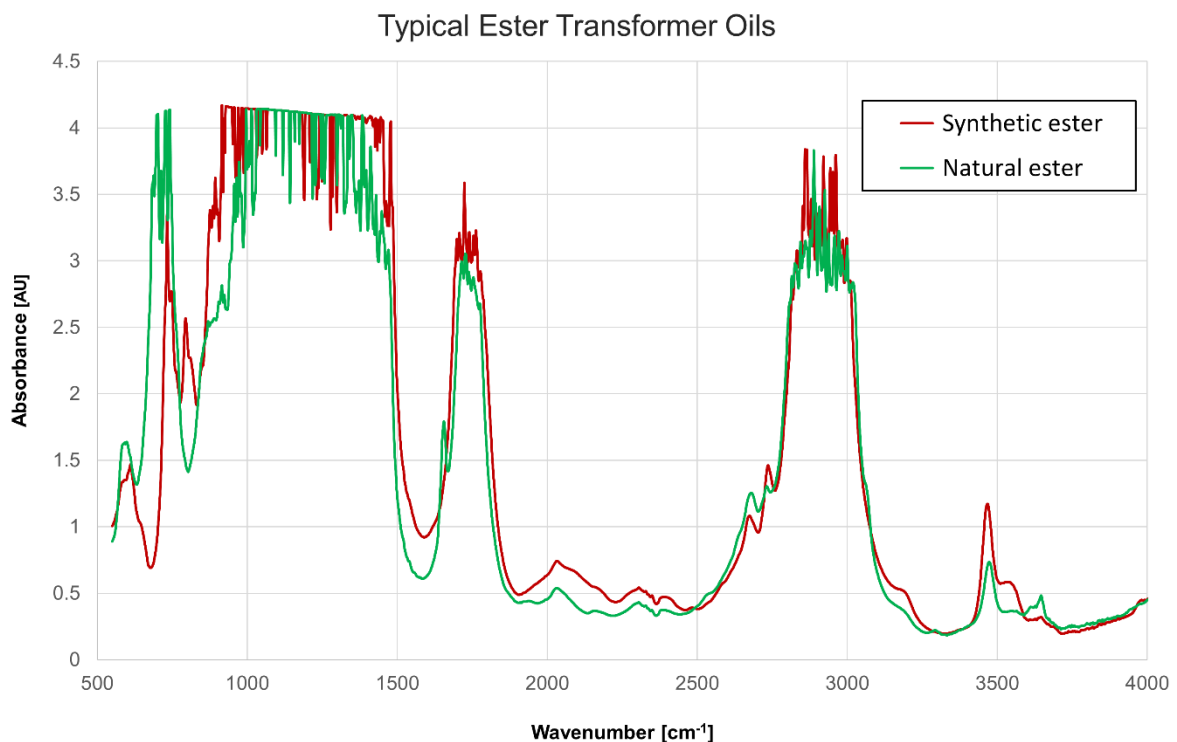


Figure 5: Spectra of synthetic ester & natural ester

Calibration of additives such as antioxidants for these ester liquids can certainly be added, but couldn't be done for the time being as information from the manufacturers of such products has not yet been collected. Industry is using increasing amounts of these rather new liquids due to the advantages in the field of potential fire safety. This fact is driven by the rather high fire point (> 300°C) of ester liquids in comparison with classic mineral oil insulation liquids. Downsides are limitations in the field of low-temperature behavior, oxidation stability and dielectric performance.

This is the reason why operators of electrical equipment do not want to have these alternative liquids mixed or contaminated within their units filled with mineral insulation oils. FTIR spectroscopy is also able to deliver answers in this domain. Unwanted contamination of ester liquids can be detected down to a limit of detection in the range of 0.05 %. In case of a transformer containing 10.000 liters this would be as little as 5 liters.

In Figure 6 the region of interest for the determination of ester contamination at 1750 cm<sup>-1</sup> is displayed. In addition to a fresh oil that is not contaminated, a clear peak can be seen for the "old" oil, which is around 0.05%. The peak of 0.10% used for calibration is also displayed as a reference and the respective significance can be easily recognized.

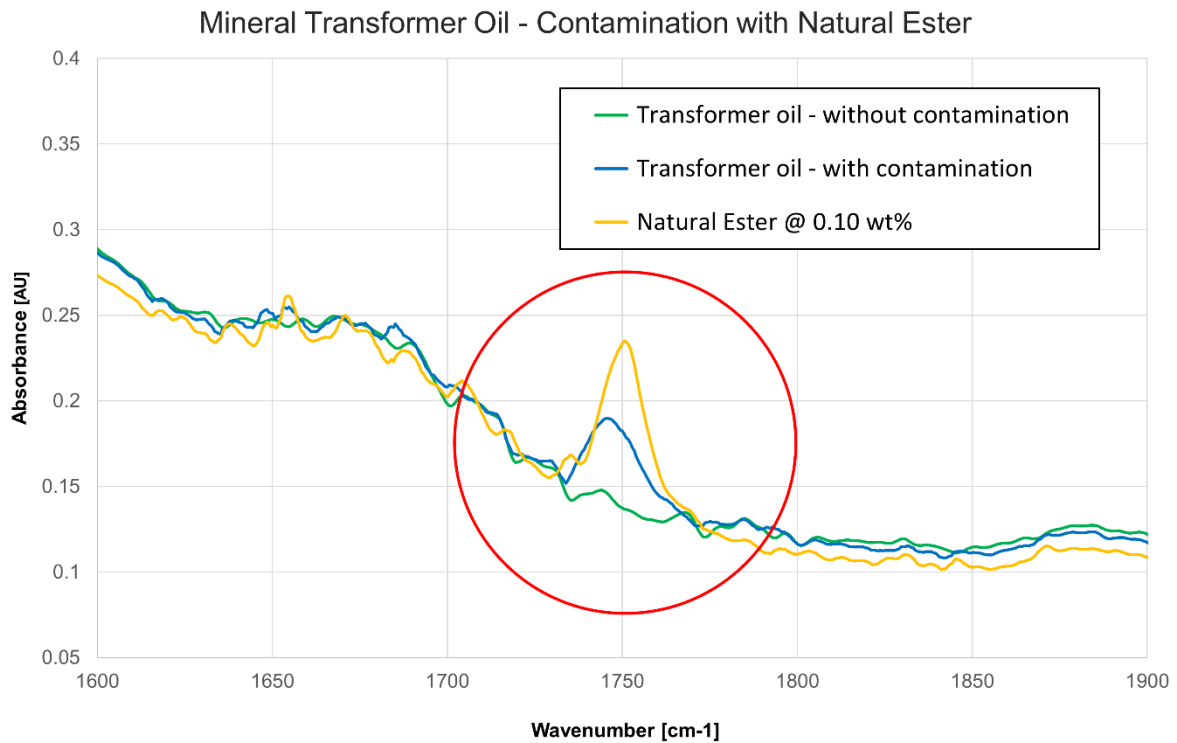


Figure 6: Detail of spectrum showing a contamination of mineral insulation oil by an ester liquid

#### Estimation of acidity based on chemometric models.

Previous evaluations were based on the interpretation of specific peaks, which is called 'direct trending'. Chemometric models play a crucial role in improving the analytical capabilities of infrared spectroscopy. By using statistical methods, chemometric models extract meaningful information from complex spectral data, which allows further properties of the sample to be predicted. ERASPEC OIL uses multi-linear regression, a method that is both very robust and easy to calibrate.

For mineral insulation liquids the parameter Acidity in [mg KOH/g] can be estimated by using FTIR combined with chemometric modeling. Following the standard method IEC 62021 Acidity is typically measured with titration methods. For very low Acidities ( $\leq 0.01$ ), which is the case for fresh and unused insulation oils, titration is out of the question. But for insulation oils in use, Acidity becomes higher and chemometric prediction is a strong alternative. Especially working as a screening tool, the results give robust feedback on whether further investigation might be necessary.

In Figure 7 a typical calibration cross-plot can be seen. The "standard error of calibration" (SEC) gives good feedback about the quality of the model in place. With a RMSEC of 0.01 mg KOH/g the model proves to be suitable.



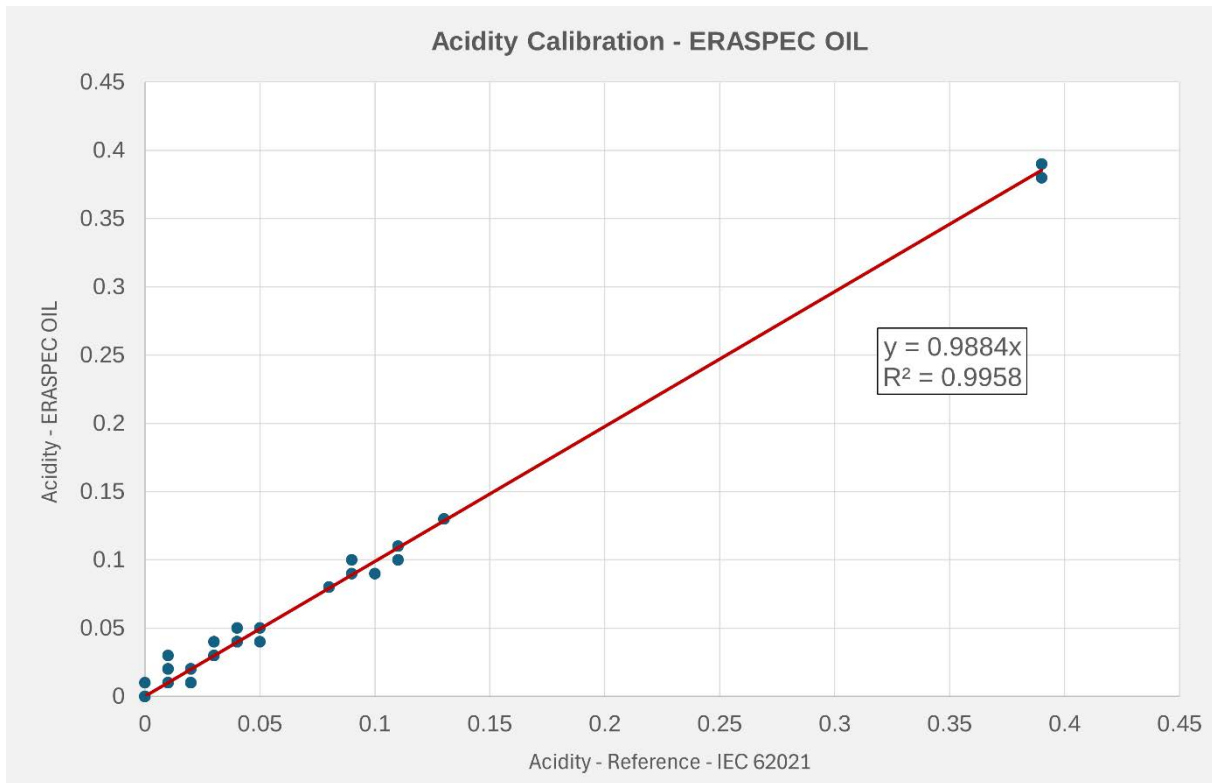


Figure 7: Cross-plot for chemometric calibration of acidity

## Summary

The electrical grid is fundamental to modern society, providing essential power transmission and distribution. Transformers in this grid must be checked regularly to ensure reliable performance. Monitoring insulation liquids, like transformer oil, is crucial to detect degradation or contamination, preserving transformer functionality. The standard IEC 60422 outlines monitoring procedures for these liquids, which are typically tested by specialized laboratories. ERASPEC OIL offers a portable infrared analyzer for on-site transformer oil analysis, measuring parameters like inhibitor and passivator content. Chemometric models, such as multi-linear regression, enhance infrared spectroscopy's analytical capabilities, predicting properties like acidity in insulation liquids. Chemometric modeling serves as a robust alternative to traditional titration methods, especially for higher acidity levels. Overall, these advancements in monitoring and analysis technologies contribute to maintaining the reliability and longevity of transformers within electrical grids.

## References

- IEC 60296      Fluids for electrotechnical applications – Mineral insulating oils for electrical equipment
- IEC 60422      Mineral insulating oils in electrical equipment - Supervision and maintenance guidance
- IEC 60666      Detection and determination of specified additives in mineral insulating oils
- IEC 61099      Insulating liquids - Specifications for unused synthetic organic esters for electrical purposes
- IEC 62770      Fluids for electrotechnical applications - Unused natural esters for transformers and similar electrical equipment
- ASTM D2668      Standard Test Method for 2,6-di-tert-Butyl- p-Cresol and 2,6-di-tert-Butyl Phenol in Electrical Insulating Oil by Infrared Absorption
- ASTM D3487      Standard Specification for Mineral Insulating Oil Used in Electrical Apparatus
- ASTM D6871      Standard Specification for Natural (Vegetable Oil) Ester Fluids Used in Electrical Apparatus