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# IMPROVING METHODS FOR SAMPLE PREPARATION OF TRANSFORMER OILS BY ICP-MS

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Abstract- Acids used in sample preparation of transformer oils can have a strong influence on matrix effects in inductively coupled plasma, leading to distorted analysis results. The article uses various acids for different methods of sample preparation (mineralization methods) and considers and eliminates matrix effects on the sample base and on the results of determining the elements of transformer oils. The work also provides a physical explanation of the effect of acids on the sample under study and selects the optimal sample preparation method to reduce matrix effects. It has been experimentally proven that as the viscosity of the sample increases, the sampling rate decreases, which can cause suppression of the signals of the elements being determined due to a decrease in the amount of analyte supplied to the device.

**Keywords**: Transformer Oil, Matrix Effects, Mineralization Methods, Influence of Acids, Sample Viscosity.

# **1. INTRODUCTION**

Currently, the predominant type of liquid dielectric for power oil-filled transformers remains to be mineral transformer oil. Frequent oil quality monitoring is performed, allowing for the identification of the oil's primary electrical and chemical properties. Simultaneously, it is still unclear how much the elemental makeup of transformer oil has changed or degraded. The transformer oil decays like most insulation and dielectric materials. This deterioration is due to resistance to electrical stresses and heat transfer from the core and coils to the oil. The condition of the dielectric oil is determined by contamination, type of dielectric oil, and the shape of the acid compounds, such as metal supplied particles. In addition to contamination, dielectric oil decomposes by exposure to partial discharge, arc, and temperature rise. The oil decomposes into low molecular weight gases, oil-soluble gases, and carbon particles. The behavior of each type of dielectric oil in converting to carbon particles is different.

It is feasible to forecast changes in the oil's operational characteristics, such as the rate of oxidation and the solubility of water in the oil, thanks to knowledge about the microelement basis of transformer oil. Furthermore, while interpreting the findings of chromatographic examination of gases dissolved in oil, information regarding the hydrocarbon content of oils in some situations needs to be taken into consideration. The mass spectrometric approach is one of the best ways to keep an eye on the chemical makeup of mineral transformer oils. The primary benefits of the mass spectrometric approach are its small sample volume analysis, quick analysis times, low detection limits, and multi-element analysis [1]. ICP-MS does, however, have certain limitations, including as spectral and non-spectral matrix effects, just like any analytical technique. These matrix effects make it more difficult to accurately distinguish the elements in complex samples and considerably raise the lower limit of the calculated contents when examining samples with complex compositions. The identification of matrix effects is primarily influenced by sample preparation, the acids used to dissolve the materials, the degree of dilution, etc.

Because it can remove complex organic matrices, oxidative mineralization is frequently utilized in elemental analysis of transformer oil. The relative complexity of the procedures, the noticeably higher time and material costs, and, of course, the elevated risk of further sample contamination and volatile element loss are all characteristics of this kind of sample preparation. Most frequently used as an oxidizing agent, concentrated nitric acid contributes the least to the appearance of spectrum interference as compared to HClO4 or H2SO4 [2]. Papers [2, 3] show that when various samples, including transformer oils, are stored with 2 ml of concentrated nitric acid in closed jars at a pressure of up to 25 bar and a temperature of 180 °C for 3 hours, practically the whole organic matrix is mineralized. In certain instances, hydrogen peroxide is added to the reaction mixture [4] to decrease the production of nitrogen dioxide vapor and hasten sample mineralization by raising the temperature [5].

Over the past 20-30 years, extensive experience has been gained in the quantitative determination of elements in samples using ICP-MS, and special methods for dealing with spectral overlap have been developed that consider only non-spectral interference. Labor-intensive and labor-intensive universal techniques (seven selections of additional dilutions, addition methods, and calibration solution matrices) have been proposed and are widely used. Instrumental methods for their removal remain insufficiently evaluated in analytical practice. The application of the internal standard method is complicated by the lack of clear criteria for selecting elements for this role. This situation is exacerbated by the fact that each type of mass spectrometer (various in configuration and design) has unique characteristics and requires the development of unique strategies to obtain correct results. Against this background, we consider it especially important and promising to develop a universal approach to eliminating non-spectral interference in the simplest and most economical devices with quadrupole mass filters. In fact, these are the most commonly used mass spectrometers.

Acids used in transformer oil sample preparation might thereby significantly affect matrix effects in inductively coupled plasma, distorting the findings of analysis. This article focuses on the use of different acids in different sample preparation (mineralization) techniques, as well as considering and removing matrix effects on the sample base and on the elements of transformer oil outcomes. In addition, the paper chooses the best sample preparation technique to minimize matrix effects and offers a physical explanation of how acids affect the sample being studied.

#### 2. EXPERIMENTAL PART

#### 2.1. Mineralization Method

Microwave (MW) radiation is typically utilized to enhance the process, even though open mineralization (on a basic heating plate or in a customized heating block) is relatively common [6, 7]. Due to the ability to program temperature and power, this not only allows for a reduction in sample preparation time, which is typically reduced from several hours to thirty minutes, but it also lowers the risk of additional sample contamination when using closed glasses. [8] The inability to mineralize more than 10-12 samples at once and the comparatively large volume of containers are the drawbacks of commercially available conventional microwave ovens [9]. To boost process productivity and sample preparation, however, contemporary microwave systems are outfitted with either dedicated vessels (Speedwave Xpert) or microinserts in the main beaker (Ethos ovens). Because this breakdown approach uses modest amounts of oxidizing chemicals, relatively low detection limits can be achieved

Samples that have been digested are further diluted prior to ICP-MS analysis because concentrated acid is typically utilized for sample preparation. The content of the identified elements and salts in the sample is taken into consideration when choosing the dilution ratio, in addition to the requirement to lower the solution's overall acidity. It is recommended to add nitric acid solutions to the mass spectrometer at a concentration of less than 2 M (15 Vol. %), according to the authors of [9]. Concentrated acids are most often used for sample preparation, so digested samples are further diluted before ICP-MS analysis.

The degree of dilution is selected based on the need to reduce the overall acidity of the solution, as well as the content of specific elements and salts in the sample. Increased concentration of nitric acid still plays an important role in dilution and can lead to rapid corrosion of the nickel interfacial cone as well as suppression of the analyte signal. The authors of [10] claim that it is advisable to introduce a nitric acid solution with a concentration of less than 2 M (15 vol. %) into the mass spectrometer. However, in some cases this recommendation may not be followed [9]. Obviously, in this case, either a platinum sampler and skimmer [11] or a flow-through sample injection system [12] should be used. In general, the degree of dilution varies depending on the method, but a 10-fold dilution remains the norm. A more radical method of combating the acidity of the analytical solution is to evaporate the mineralized sample and dissolve it in a diluent of the required composition [11]. However, it is no longer used in modern practice due to the high risk of further sample contamination and long analysis times.

The studies were carried out on an inductively coupled plasma mass spectrometer from Agilent Technologies 7700 (USA) to measure the concentration of metals in transformer oil. To mineralize the samples, a microwave system "Speedwave Xpert" (Germany) was used with the ability to control temperature, equipped with small-volume vessels for working with microsamples. Dispensers with a volume of 100-1000 µl and 1-10 ml manufactured by pipet4u and Eppendorf (Germany), disposable tips and polypropylene tubes with a volume of 15 and 50 ml were used [10-13]. For mineralization of samples and preparation of calibration solutions, 65% nitric (HNO<sub>3</sub>) and 37% hydrochloric acids (HCl) were used, and also for rapid dissolution of the sample during mineralization, a 30% solution of hydrogen peroxide (H<sub>2</sub>O). Suprapuz (Merck Germany) was used. All solutions were diluted with deionized water (18.2 M $\Omega$ ·cm). The correct calibration of the device was controlled by analyzing a standard sample of drinking water.

Table 1. Microwave decomposition of transformer oil (method 1)

Descenta	Acid			Volume			
Reagents	HNO <sub>3</sub> (65%)			8.0 ml			
	100 mg	100 mg (0.1 ml) of the sample was placed in the vessel, then					
	8.0 ml of HNO <sub>3</sub> was added to mineralize it. The mixture was						
Process	thorough	nly shake	n or stirre	d with a clea	n Teflon or glass rod.		
	Before closing the vessel, wait at least 10 minutes, then heat it						
	in a microwave oven according to the following program						
	Step	$T[^{\circ}C]$	P [bar]	Time [min]	Power [%]		
Program	1	145	80	10	80		
	2	170	80	10	80		
	3	190	80	20	90		
	4	50	60	10	0		

### **3. RESULTS AND DISCUSSIONS**

The procedure for microwave decomposition of transformer oil was carried out using three methods (Tables 1-3) [14].

Table 2. N	<b>Microwave</b>	decomposition	of transformer	oil	(method 2	2)
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	Acid			Volume			
Reagents	HNO <sub>3</sub> (65%)			5.0 ml			
	HCl (37%)			2.0 ml			
	To fi	To fill the digesting vessel, weigh out 100 mg of the					
	sample. Add 2.0 ml of HCl and 5.0 ml of HNO <sub>3</sub> . Using a						
Drocess	glass or Teflon rod that has been cleaned, gently shake or						
1100055	swirl the contents. Prior to sealing the container, give it a						
	minimum of ten minutes. Reheat in the microwave using						
	the following program.						
Program	Step	$T[^{\circ}C]$	P [bar]	Time [min]	Power [%]		
	1	140	35	3	70		
	2	160	35	2	80		
	3	200	35	2	90		
	4	50	25	1	0		

Table 3. Microwave decomposition of transformer oil (method 3)

	Acid			Volume		
Reagents	HNO <sub>3</sub> (65%)			9.5 ml		
	H <sub>2</sub> O <sub>2</sub> (35%)			2.5 ml		
Process	Put 300 mg of the material into the digestive vessel by weight. Add 2.5 ml of $H_2O_2$ and 9.5 ml of HNO <sub>3</sub> . Using a clean glass or Teflon rod, gently shake or swirl the mixture. Prior to sealing the container, give it a minimum of 20 minutes. Reheat in the microwave using the following program.					
Program	Step	<i>T</i> [°C]	P [bar]	Time [min]	Power [%]	
	1	130	70	3	50	
	2	190	80	3	60	
	3	230	85	5	70	
	4	50	25	1	0	

Transformer oils were mineralized using all three methods in a microwave oven. As can be seen from the figure, when using the second sample preparation method, the concentrations of elements in the sample decreased. In other words, those samples that we dissolved in hydrochloric acid showed a decrease in the concentration of elements. Similarly, the figure shows that when using the third sample preparation method, the concentrations of elements became close to the concentrations obtained by the first method, even in some elements such as Fe, Zn and Ag, excess concentrations were observed.



Figure 1. Change in the concentration of transformer oil elements after microwave mineralization [13]

This is because more argon kinetic energy falls per unit volume of liquid when the sample's viscosity increases and, as a result, the atomizer's load decreases at a constant gas flow rate. Furthermore, the difference in the average droplet sizes in diluted and more concentrated solutions is not as noticeable for acids with lower viscosities (like nitric acid). Of course, acid solutions' physical characteristics might vary greatly from waters. Viscosity, surface tension, density, and volatility are the ones that should be emphasized the most from an analytical perspective. The values of certain physical quantities for solutions of hydrochloric and nitric acids at various concentrations are displayed in table 4 (these acids were employed in the work). The viscosity and density of acid solutions play a major role in determining how they behave.

Table 4. Physical properties of acid solutions at 25 °C [15]

Acid	Content mass %	Viscosity (rel. H <sub>2</sub> O)	Surface tension mN/m	Density q/sm <sup>3</sup>
INO	4	1.014	72.15	1.022
$\Pi NO_3$	30	1.397	68.75	1.180
HC1	4	1.057	72.45	1.017
	30	1.896	65.75	1.149

The influence of the sample collecting rate on the acid content, from which it follows that as the viscosity of the sample increases, the sampling rate drops. As a result of less analyte entering the device, the latter may suppress the signals of the identified elements. Table 4 shows that solutions with higher acidity simply have higher viscosity values. As for the processes occurring directly in the plasma, the influence of the acid in this situation largely depends on the values of the operating parameters of the device, notably, the flow rate of argon via the atomizer and/or the power of the generator [16]. Signal suppression in samples with high acidity is a consistent result of utilizing the "standard" mode on a mass spectrometer that has been set to maximum sensitivity of determination using a particular solution of elements of different atomic masses in 2 vol. % HNO3. This phenomenon could be explained by the fact that elements ionize less efficiently in these conditions, particularly those with a high IP, since the plasma is more heavily loaded with nitric acid, which lowers the local temperature of the plasma. The fact is that the amount of plasma energy needed to evaporate and atomize HNO3 molecules is significantly higher than that of H2O molecules, and the amount of energy transferred to the central channel by the breakdown products of nitric acid, which have a thermal conductivity higher than that of Ar is significantly lower than it is in the vicinity of water [16].

The effect of acids on the average Sauter diameter (one of the most important properties of primary aerosols) when using pneumatic nebulizers can be roughly evaluated using the Nukiyama Tanazawa equation, which relates the physical properties of the solution being atomized.

However, as with impactor sprays, they only work within certain ranges of viscosity, surface tension, and density, so they often produce inaccurate results that are purely theoretical. It has been experimentally shown that the effect of acid on the size distribution of primary aerosol droplets is different for different sample delivery modes. In self-priming mode, as the concentration of nitric or hydrochloric acid in the solution increases, a more highly dispersed aerosol is formed. This is because as the sample viscosity increases and the nebulizer load decreases, more kinetic energy of argon is lost per unit volume of liquid at a constant gas flow rate. At the same time, the increased acidity during pumping results in the formation of an aerosol enriched with droplets of larger average diameter. Furthermore, the difference in the average diameter of droplets in dilute and more concentrated solutions is less pronounced for acids with lower viscosity (nitric and hydrochloric acids).

The effect of acids on the formation of tertiary aerosols emerging from the spray chamber is more universal: the higher the acid content in the solution, the less coarse the aerosol is. However, this effect is more pronounced in self-priming mode. One possible explanation for the observed pattern is that larger droplets are less likely to leave the spray chamber with the higher density of the solution composing the droplet due to their greater inertia. Another important characteristic is the fraction of the aerosol volume occupied by droplets, which is a kind of indicator of the influence of the acid on the transport of the solution through the spray chamber but does not change at all with a slight increase in the acid concentration, or rather, a slight increase is observed. Note that the aerosol volume fraction value also depends on the design of the spray chamber used.

Furthermore, the transmission capacity of charged particles with reduced average kinetic energy through ion optics reduces under low plasma temperature circumstances. Signal suppression is eliminated when one approaches the regime of so-called "stable" parameters, which is defined as reducing the argon flow rate relative to the "standard" characteristics while maintaining or increasing the used generator power. This is because the processes of desolation, evaporation, atomization, and ionization become more efficient. This is caused by two factors: first, the aerosol travels more slowly from the burner injector tip to the plasma as the argon flow rate decreases; second, the region with a sharp temperature difference (ranging from one hundred to several thousand degrees) shifts towards the injector tip. Together, these factors lengthen the residence time of the sprayed solution in the high-temperature zone [16]. Additionally, it was observed that optimizing the device's operational parameters results in lower argon flow rates and greater power values at sample depths of less than 15 mm, which further enhances the effectiveness of neutralizing the acidic effect.

It is important to note that the magnitude of the change in signal intensity, as well as the relaxation time, depend on the nature and concentration of the acid. The greater the difference in the acidity of the solutions, the more dramatic the change in signals will be. Conducted studies show that during microwave decomposition it is necessary to use those acids that give low spectral interference. At high acids, the mass data obtained by the spectrometric device are distorted and incorrect results are obtained at the output. Using this data, problems in transformers can be diagnosed in advance. For example, as shown in the figure, an increase in the concentration of iron and copper in the oil warns of problems with the core and tank of the transformer, and a large amount of aluminum indicates problems in the windings and ceramic bushings of the transformer.

#### 4. CONCLUSION

Oxidative mineralization is widely used in elemental analysis of transformer oil due to its ability to remove complex organic matrices. There are many methods for oxidative mineralization of transformer oil, which use various acids to dissolve it. An optimal sample preparation method was selected that reduces the influence of matrix effects that distort the analysis results. The physical processes occurring directly in the plasma have been studied. It was revealed that the effect of acid in this case strongly depends on the values of the operating parameters of the device, primarily, the flow rate of argon through the atomizer and/or the power of the generator. As the viscosity of the sample increases, the sampling rate decreases, which can cause suppression of the signals of the elements being determined due to a decrease in the amount of analyte entering the device.

#### NOMENCLATURES

#### Acronyms

- ICP-MS Inductively Coupled Plasma Mass Spectrometry
- HClO<sub>4</sub> Perchloric Acid
- H<sub>2</sub>SO<sub>4</sub> Sulfuric Acid
- HNO<sub>3</sub> Nitric Acid
- H<sub>2</sub>O<sub>2</sub> Hydrogen peroxide
- IP Ionization Potential
- Ar Argon gas
- HCl Hydrochloric Acid

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