SAMPLE CONTAINER SELECTION FOR INSULATING LIQUIDS

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Any commercial laboratory performing insulating liquid analysis receives a myriad of sample containers through the course of a year. Not all of these containers are compatible with the liquid they are storing, nor do they maintain the properties to be tested intact.

The selection of a sample container is a specific science with the ultimate goal of not impacting the sample in any way. Thus, the insulating liquid in the sample container must reflect the insulating liquid in the electrical apparatus whether it be a transformer, load tap changer (LTC), oil circuit breaker (OCB), or some other device.

The requirements for a sample container are:

- Large enough to hold the volume of liquid necessary for analysis.
- Does not impart any contamination (chemical or particles) to the sample.
- Seals the sample from external contamination.
- Shields the sample from direct sunlight to prevent photo-degradation either by having a dark container or by using a covering for the container after the sample is taken.
- Prevents the loss or gain of gases or water when testing for these properties.

Changes in an insulating liquid's properties while in the sample container can yield lab results that do not reflect the bulk oil in the apparatus and can yield a faulty diagnostic assessment of the apparatus or insulating liquid. Tests such as dissolved gas-in-oil analysis (DGA), water content, and methanol/ethanol analysis are critical tests that can easily be impacted in a negative way and can lead to a faulty condition assessment of the apparatus.

DISOLVED GAS ANALYSIS

DGA is used to determine the operational condition of an electric apparatus and is considered the most important insulating liquid test. If the sample container retaining the DGA sample is not completely sealed, gases such as hydrogen and carbon monoxide are easily lost. Because of their poor solubility in insulating liquids, these gases easily diffuse to the atmosphere, typically in a matter of minutes to hours.

Hydrogen is usually reflective of partial discharge, and carbon monoxide is most often due to paper degradation in the transformer. The loss of these gases would indicate that a faulty condition does not exist when, in fact, it may. In addition, additional concentrations of oxygen, nitrogen, and even carbon dioxide can be dissolved into the oil from the external atmosphere. The presence or absence of oxygen and nitrogen helps one understand the condition of the transformer's preservation system and whether the gaskets, O-rings, and/or conservator bladder are performing their intended functions. Elevated oxygen and nitrogen concentrations may indicate there is a leak, where the apparatus may be perfectly sealed resulting in unneeded costly maintenance.

For free-breathing LTCs and OCBs, a high amount of oxygen that is commensurate to the amount of oxygen in the air is expected. When oxygen levels fall low, it usually indicates that advanced aging of the oil is occurring or that the apparatus breathing mechanism is plugged. If the incorrect sample container is used or the sample is not taken properly, atmospheric oxygen will be present, and these types of conditions cannot be determined.

WATER CONTENT

Accurate water content values help determine the wetness or dryness of the entire insulation system including the paper or solid insulation. It must be

SOURCE: LEWAND & KOEHLER

remembered that most transformers are dry and are intended to stay that way to minimize aging of the solid insulation. Aging of solid insulation is directly proportional to water content. As paper ages, it produces water as one of the byproducts, so the amount of water in the transformer will gradually increase over time. Samples must properly reflect those conditions.

Selecting the correct sample container is imperative for keeping the properties of the samples intact and reflective of the properties of the bulk insulating liquid in the electrical apparatus.

The best time to take samples in the northern hemisphere is usually the summer months when most transformers are operating at highest load and warmest environmental temperatures. This condition will provide the best estimate of the water condition existing in the solid insulation of the apparatus. However, the summer months are also the most humid months, and contact of the insulating liquid with the humid atmosphere can increase the water content of the sample both dramatically and quickly, so this must be guarded against.

Thus, samples for water content must only be taken in enclosed systems such as DGA glass syringes. Even taking samples in metal bottles that can be filled to overflowing can be impacted, as the humid air in the bottle during filling will increase the water content by several ppm(mg/kg). A difference in a few ppm(mg/kg) can make a dramatic difference in calculating the water in paper content.

Environmental humidity will also negatively impact dielectric strength, especially in plastic bottles, as plastic is not a solid barrier against the ingress of water into the sample. Dielectric strength is reduced by increasing water content and particles. Plastic bottles allow water to diffuse across the plastic and can increase the water content substantially in just a few hours due to the natural physical process of the humidity outside the sample container trying to reach equilibrium with the humidity of the insulating liquid inside the sample container. This may result in lower dielectric strength of a sample when tested in a laboratory as compared to the insulating liquid that exists in the actual apparatus. Thus, plastic containers are not advisable for use when water content or dielectric strength measurements are going to be conducted.

METHANOL/ETHANOL

Methanol and ethanol concentrations are chemical aging markers that are produced from degradation of cellulose inside the transformer. They are better suited than furanic compounds in transformers where thermally upgraded paper is present and are commonly found in 65°C rise-rated transformers. However, furanic compounds are light alcohols and very volatile, so sampling for them must be treated in the same way as DGA samples and taken in glass syringes so they are not lost to the atmosphere.

These are but a few examples of how sample analysis can be negatively impacted by choosing the incorrect sample container. A variety of plastic, glass, and metal bottles have been used over the years along with glass syringes and steel cylinders. ASTM D923, *Standard Practice for Sampling Electrical Insulating Liquids* provides a list of sample containers and the advantages and disadvantages of each and should be consulted before drawing a sample. Lewand & Koehler provide a very detailed commentary on proper sampling technique and sample containers.

SAMPLE CONTAINERS FOR WATER CONTENT

To illustrate some of this information, research was performed to determine which types of containers are best at maintaining the quality of the sample for water content analysis. Four types of sample containers were evaluated in this nine-week testing: high-density polyethylene (HDPE), glass bottles, glass syringes, and aluminum bottles.

Table 1: *Water Ingress into Different Containers*

Average of Three Determinations per Measurement, ASTM D1533, mg/kg (ppm)				
Sample	**HDPE**	**Glass Bottle**	**Glass Syringe**	**Aluminum Bottle**
Initial Bottle 1	3.8	3.4	3.4	4.8
Initial Bottle 10	5.0	5.7	5.9	9.8
Week 1	14.4	7.6	5.5	5.7
Week 2	20.9	5.5	4.4	4.6
Week 3	30.4	9.5	9.7	7.1
Week 4	29.2	8.7	7.5	5.2
Week 5	37.8	21.4	5.9	5.4
Week 6	40.9	29.6	7.7	6.3
Week 7	44.7	27.3	7.2	7.3
Week 8	45.0	17.1	8.0	7.3

Transformer oil (22 liters) was dried with nitrogen to a water content of less than 5 mg/kg. This dried oil was transferred to 40 containers representing 10 each of four different container types: 1-liter HDPE bottle, 1-quart glass bottle with a hard plastic cap and paper liner, 50-ml glass syringe, and 1-liter aluminum bottle with sure-lock cap fitted with an aluminum liner. The containers were uncapped and aspirated with compressed air to remove unwanted particles and then transferred to a glove bag previously filled with nitrogen. All containers were filled with oil inside the glove bag.

Each container was filled to overflowing with oil and capped. Syringes were filled to the 40-ml mark, and all gas bubbles were expelled. The stopcocks and caps were then checked for tightness on each container. After filling, the first and last samples of each category were tested for water content within 1 hour. The rest of the samples were maintained in a glove bag under conditions of room temperature (approximately 22.5°C) and high humidity (83% to 88%). One sample for each category was tested weekly and the results recorded. The results are presented in Table 1.

The graph in Figure 1 summarizes the results from Table 1.

Results over the eight-week period indicate that HDPE bottles are not suitable for long- term storage

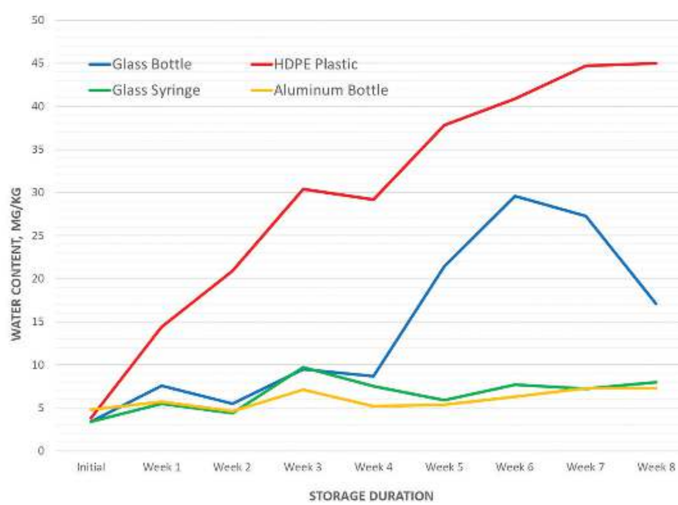

Figure 1: *Water Ingress into Various Containers under Controlled Conditions*

when a water content test is to be performed. As Figure 1 indicates, the maximum water content at the end of the experiment was 45 ppm for the HDPE bottle; the other containers were much lower. In addition, the caps worked loose on both the HDPE and glass bottles over time. If sample integrity is to be maintained for a long time, then periodic resealing of the caps is necessary.

Figure 1 depicted how the oil in each container fluctuated in water content in relation to the maximum

Table 2: *Suggested Sampling Container Applications*

Tests	Container Type
DGA, water, methanol/ethanol	Glass syringes, aluminum bottles for water only
Furanic compounds, oil quality	Particle-free glass bottles, aluminum bottles

water content that could be dissolved in oil using 88% relative humidity and a laboratory temperature of 22.5°C. At 22.5°C, 100% saturation of water in oil is about 61.2 ppm. Eighty-eight percent of 61.2 ppm is 53.9 mg/kg. Those were the concentrations used to calculate to what percentage the oil in each container rose in relative saturation over time. As shown in Figure 1, the HDPE plastic container was almost 84% (45 ppm/53.8 ppm) of that value by experiment's end, whereas the glass bottle rose to 32%, and the syringe was only about 15%. The aluminum bottle stayed fairly constant at 10 to 12%.

It is obvious that external relative humidity impacts the water content of the sample in certain containers, specifically HDPE plastic bottles. Testing the sample quickly and keeping the relative humidity in the laboratory low would negate some of these effects. However, keeping the relative humidity in the laboratory too low may also have the opposite effect. Equilibrium with the relative humidity of the ambient environment would also tend to dry the sample if the concentration of water on the outside of the sample container was less than the water in the oil in the sample container.

Figure 2: Bottles and Syringes for Water Ingress Test

These effects do not seem to manifest themselves in either syringes or aluminum bottles. Therefore, syringes and aluminum bottles are the best containers to store oil for determining water content in transformer insulating liquid samples (Figure 2).

Because glass bottles cannot be filled to the top because of possible breakage due to expansion and contraction, some atmosphere exists inside the bottle. The external atmosphere can definitely affect the final water content of the sample prior to analysis, especially if that atmosphere is really humid.

CONCLUSION

Selecting the correct sample container is imperative to keeping the properties of the samples intact and reflective of the properties of the bulk insulating liquid in the electrical apparatus. Evaluating the laboratory test data of such samples may yield incorrect or incomplete diagnostics. Remember that the analysis is only as good as the sample taken.

It has been our experience that the only compatible plastic bottle is high-density polyethylene (HDPE) plastic. However, we have moved away from plastic bottles almost completely because of the particles present in plastic bottles from the manufacturing process and the speed in which water can move through the plastic into the sample. Table 2 provides suggestions on which containers to use for particular applications.

Lastly, the caps for glass bottles must also be selected carefully. The only acceptable types are those caps with foil, polyethylene, polypropylene, or PTFE liners.

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