A DAY IN THE LIFE OF AN OIL LABORATORY

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INTRODUCTION

I work in a laboratory testing oil samples taken from electrical assets such as transformers, cables and bushings used by electricity utilities, generators, and heavy industry. When clients drop off samples, I often see them peeking into the lab as if it's an exotic new world and for most people, it is. One of my favorite things to do as a laboratory professional is introduce people to the exciting world of laboratory operations, specifically the testing and diagnostics of insulating materials in electrical apparatus.

Working in an oil lab is a unique environment in which no two days are alike. I'd like to offer a glimpse into what really happens behind the scenes in an oil testing laboratory. In some ways it's much more complicated than it may seem on the surface but in other regards, it's quite simple with a process driven by quality and the desire to offer exceptional customer service and support.

Every sample will embark on a unique journey, starting with sampling on site and ending with reporting and sample disposal. It sounds simple on paper but there are many steps involved, all of which require a high level of technical aptitude and attention to detail. A multitude of people with a wide range of skills are needed to keep a lab running like a well-oiled machine.

But first, what is an oil laboratory and what exactly does it do?

There are oil testing laboratories located around the globe that serve the power industry and have the capability to perform analytical tests on insulating materials found in electrical equipment. Performing oil testing on samples collected from electrical assets such as power transformers, is often an annual procedure carried out by utilities, service companies, and large industrial asset owners. The results of these tests are a crucial part of monitoring the health of these assets as an oil sample can provide a lot of insight into the condition of the insulating materials and more importantly detect and identify incipient fault conditions. Therefore, when a problem with a transformer is expected, one of the first things the asset owner will do is commission an oil sample to be sent to a lab for testing.

Here's just a few insights to be gained by performing routine oil analysis:

- Detecting incipient fault conditions through changes in gassing behavior
- Determining if the oil is of sufficient quality for in-service use
- Measuring moisture content to detect leaks or paper degradation
- Measuring indicators related to the condition of the cellulose insulation
- Uncovering contamination in oil

Oil laboratories can offer a variety of services from routine test packages to fully customized testing solutions and consultation services. Some labs may offer well over 100 tests including forensic/failure analysis, new oil specification packages, and high voltage testing. A customer's testing needs are dependent on several factors and with the variety of testing options available today, there's guaranteed to be a testing solution that fits the customer's individual needs. Oil lab technical personnel are well versed in the options available and will work directly with clients to recommend testing packages based on the customer's specific circumstances.

Oil testing can be divided into two basic categories:

1. Routine tests are performed on oil samples taken on a regular basis (typically every to 1 to 5 years depending on the asset type). These provide a high-level indication of the asset's health.
2. Specialty tests are performed for specific purposes often to elucidate unusual findings from routine tests or following concerns about the health of an asset.

Routine tests include:

- Dissolved Gas Analysis (DGA)
- Oil quality
  - Water content
  - Interfacial Tension (IFT)
  - Neutralization number
  - Power Factor
  - Dielectric Breakdown Voltage
- Tests to assess the condition of paper insulation
  - Furanic compounds
  - Degree of Polymerization (DP)
  - Methanol and Ethanol
- Additives
  - Oxidation inhibitor content
  - Passivator content
- Corrosive sulfur

Specialty tests are varied and include:

- Forensic/failure mode investigations
- Material compatibility
- Oil contamination identification
- Pilot clay treatment
- Elemental analysis
- Oxidation stability testing

Figure 1: Journey of a Sample

SAMPLING

The testing journey really starts at the time the sample is taken (Figure 1). The goal is to pull a sample that is truly representative of the bulk liquid inside of the apparatus being tested. Improper sampling techniques and containers can lead to contamination of the sample that could adversely impact various test results. This can potentially lead the laboratory to flag the data as an apparatus or oil quality issue when in reality, the bad data is a consequence of poor sampling technique or inappropriate containers.

The cost of having to go back to the site to pull a new sample can be quite costly; and between labor, materials, and shipping, the cost of a poorly drawn sample can easily reach upwards of $500USD [1].

There are many guides and papers available that detail how to take a good sample such as ASTM D923*, Standard Practice for Sampling Electrical Insulating Liquids* [2]. Steps include cleaning/flushing oil lines, and valves, and using appropriate bottles and syringes: improper containers can easily contaminate a sample. Using a particle free, light block, glass or metal container (Figure 2) is preferred for oil quality testing; this is because:

1. Plastic containers are not compatible with transformer oil: with an exception being high density polyethylene (HDPE). However, it's important to keep in mind that plastic, even HDPE, "breathes" and moisture can diffuse through the container wall into the sample leading to erroneously high water content results and low dielectric breakdown values.
2. Clear jars or bottles increase the likelihood of photodegradation of the oil, especially if the sample is stored in direct sunlight [3]. Effects on oil quality tests can include increases in acidity and power factor, and a rapid decrease in IFT. DGA results can also be negatively impacted with notable increases in hydrogen, carbon oxides, and select hydrocarbon gases.

An air-tight glass syringe with stopcock is appropriate for DGA testing as gases will not travel in or out of the syringe if properly sealed. Most laboratories can provide test kits that include most of the materials you'd need to take a sample such as a syringe with stopcock, glass or metal bottle, tubing, and sample identification paperwork.

So what happens to your carefully collected and packaged oil sample between sending it to your laboratory and receiving the final report?

Figure 2: Syringes and Bottles for Testing Insulating Liquids

RECEIVING

On any given day, an oil lab can receive dozens or even hundreds of samples. Personnel trained to receive laboratory orders are the first in line to handle the samples upon arrival and this process requires more attention to detail than one may think. There is a lot of critical information that needs to make its way to the testing and reporting team to ensure a smooth testing process and accurate diagnostics. The key information includes:

- Asset nameplate information
    - Equipment type (transformer, load tap changer (LTC), bushing, voltage regulator)
    - Serial number
    - kV and MVA ratings
    - Manufacturer and year of manufacture
    - Design type (core vs. shell form)
    - Preservation System (free breathing, nitrogen blanketed, conservator with bladder)
    - Cooling (ONAN, ONAF, KNAN)
- Sampling information
    - Sample date/time
    - Top oil temperature
    - Sample point (bottom main tank, gas relay, etc.)
    - Ambient conditions at time of sampling (temperature, humidity)
- Testing required
- Priority (same day or next day turnaround, routine)

The more information the laboratory knows about the equipment being tested, the more precise the technical team can be when providing testing recommendations and diagnostic comments on the final report. We'll cover this in more detail when we discuss reporting.

Figure 3: Sample Receiving

After unboxing, determining the priority level of a sample is the next step in the receiving process. Most samples received at a lab are for routine testing as part of the asset's "annual physical" and will be assigned a routine priority. However, there will be times when test data is urgently needed to make important decisions or prevent a failure. Those samples are assigned a rush priority and select tests can be turned around by a lab in just a few hours.

Once the priority has been determined, it's now time to officially "check in" the samples by logging them into a software program/database. Most laboratories utilize a Laboratory Information Management System (LIMS) to track samples, manage test data, and issue reports. The receiving technician will assign a priority level, due date, and assign testing all within the LIMS system. Key equipment and sampling information is also entered into the software at this time. Once the order has been double checked for accuracy, the samples are then cleared for testing and placed in storage (Figure 3) until it's their turn to be tested.

STORAGE

How the samples are stored prior to testing is also a critical but sometimes overlooked part of the sample testing process. The storage area and the lab space (Figure 4) in which the samples are tested is very important as environmental conditions such as direct sunlight, temperature, and humidity can all have a negative impact on not only the samples but the test equipment as well. Samples should be stored in a temperature-controlled environment, ideally between 20°-25°C. You would be hard pressed to find a lab that's not temperature controlled these days but there are other storage considerations to take into consideration. As previously mentioned, keeping samples out of direct sunlight is desirable and controlling humidity is also critical since moisture can negatively impact both instrumentation and test results.

Figure 4: Sample Storage and Testing Floor

QUALITY

Before discussing the details of the testing process, it's important to understand the quality control measures that a lab must take to guarantee accurate and precise results. For any laboratory, quality should be of the utmost importance. Second only to safety, data quality is what differentiates a great laboratory from an average one and a robust quality program should be a primary factor when choosing a lab.

Critical safety and financial decisions are made based on the test results issued by your oil testing provider, so wouldn't you want to be certain that your results are as accurate and precise as possible? It's normal to see a certain amount of variability in test data between laboratories as the test and sample conditions will vary but these variations should be kept to a minimum. In most cases, sub-par data and larger than expected variations in results are caused by:

- Poor calibration and maintenance of test equipment
- Inadequate staff training
- Lack of adherence to test procedures

A robust Quality Management System (QMS) should address these potential issues by having clear procedures that must be adhered to by all laboratory staff.

There are different quality accreditations a laboratory can obtain, ISO 17025 arguably being one of the most respected and desired of them all. Internationally recognized, ISO 17025 is a quality standard for testing and calibration laboratories that enables the lab to demonstrate that they operate competently and generate valid results, thereby promoting confidence in their work [4].
So, your lab of choice holds a quality accreditation, but what does that really mean in terms of day-to-day quality as it relates to your data? There are many requirements that a lab must continuously meet to maintain an accreditation and ensure confidence in results. To start, a laboratory must have sufficient resources to carry out testing, including facilities, personnel, equipment, and data systems.

Let's break down some of the key components of a successful quality program.

Equipment: Laboratories utilize many types of instrumentation, glassware, and chemicals to perform testing so it's important that all equipment and materials are fit for use. It's mandatory that all equipment used to produce customer data be calibrated or standardized and qualified daily using standard reference materials and other quality control checks. Therefore, the first thing a lab will do each day is run quality control standards to determine if the equipment is operating properly and capable of generating accurate data (Figure 5). This qualification step must be performed *before* the instrument or apparatus is given the greenlight to run customer samples by a trained technician. If an instrument does not pass quality control checks, the instrument must be taken out of service until the problem has been assessed and remedied. This should be a non-negotiable step in every laboratory. If you can't trust the equipment, you cannot trust the data.

An experienced analytical chemist is a particularly valued member of any lab team as they will be uniquely qualified to spot potential or current issues with analytical instrumentation. The daily QC process is similar for all test methods with each test apparatus undergoing its own series of daily checks. Subjective tests such as color, neutralization number, and corrosive sulfur, may require that each operator run their own set of quality control samples, so any individual biases are addressed in the daily qualification process.

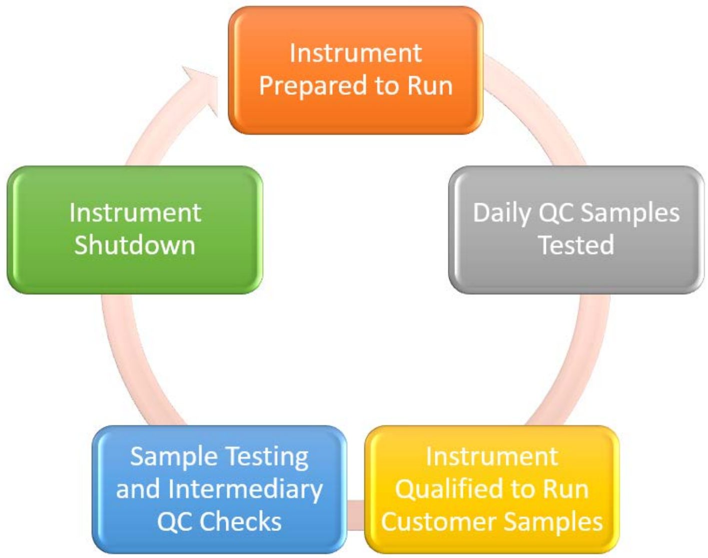

Figure 5: Instrument Quality Control Cycle

A lab may run dozens or even hundreds of samples per day so it may be necessary to employ some intermediary checks throughout the day by mixing additional quality control samples in with the regular customer samples. This is recommended since a lot can happen to an instrument or process over the course of an 8+ hour run and is an important factor when analyzing the repeatability and precision of quality control samples.

Instrument calibration records including uncertainty budgets and statistical quality control (SQC) charts (Figure 6) should be reviewed daily by the lab's quality assurance/quality control (QA/QC) team to ensure that the instruments/processes are working correctly and not trending in an undesirable manner. Traceability to an International System of Units (SI) through organizations such as the National Institute of Standard and Technology (NIST) is also part of this process.

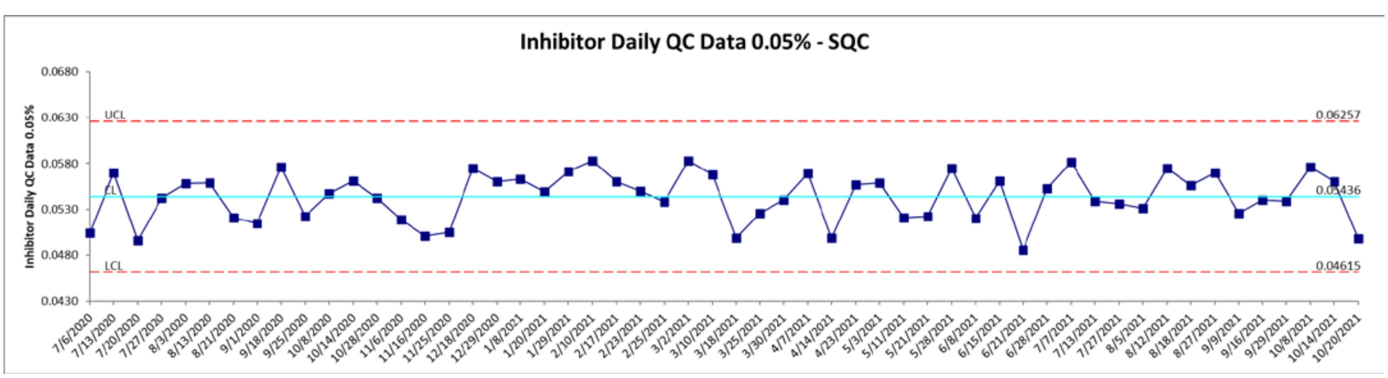

Figure 6: Statistical Quality Control (SQC) Chart

Training: Having a highly trained and competent staff is just as critical to the quality of data. Every employee in a lab should be thoroughly trained and monitored before they are authorized to perform testing independently. This ensures that methods and procedures are being followed to the letter and thus reduces the risk of human error due to lack of training. A commitment to continuously monitor the competency of the testing team is also a requirement of many quality standards. A lab can meet this requirement by participating in Inter-Laboratory Studies (ILS), Proficiency Testing Programs (PTP), testing of blind samples, and periodic test method assessments.

Advanced technical and data analysis training will also be provided by top-tier laboratories. Having a deeper understanding of the data is a key element in being able to produce the highest quality results possible. Teaching technicians and chemists about electrical equipment diagnostics and what measurands are critical will lead to a heightened sense of awareness when it comes to spotting potential fault conditions and anomalies in data. Having diagnostic-savvy testing technicians and analysts will allow the lab to flag suspicious data earlier in the testing process, allowing the asset owner to be informed of a potential problem as soon as possible. This is important since some faults can develop rapidly and being alerted just a few hours sooner can make all the difference.

Facilities and environmental conditions: As with all precise scientific measurements the environmental conditions in the laboratory need to be controlled in a manner that does not jeopardize the integrity of, or invalidate, the test results. If the environment cannot be controlled, the laboratory should stop operations until the problem is fixed. Some ASTM and IEC test methods may provide environmental guidelines such as an appropriate temperature range in which the test should be conducted.

Temperature, humidity, and barometric pressure are three environmental parameters that need continuous monitoring in the laboratory as they can all have an impact on certain tests. Excess humidity in particular is known to have a marked negative impact on test results; especially impacting water content, dielectric breakdown voltage, and power factor at 25°C. Even short duration exposures to humid air can cause the oil to absorb the water and lead to depressed dielectric breakdown voltage results. This is one of the reasons why we see seasonal fluctuations in water content and dielectric breakdown voltages, especially during the summer and in more humid parts of the world.

Weather stations should be installed in the lab so the environmental conditions can be easily monitored (Figure 7). Laboratories with a larger footprint may require multiple weather stations as environmental conditions may vary throughout the lab and they should be deployed in the areas that contain the tests that are most sensitive to extreme environmental conditions.

Figure 7: Dielectric Breakdown Voltage Test Set and Calibrated Digital Barometer

Documentation and Technical Record Storage: If it's not documented, did it really happen? In the eyes of most quality management professionals, the short answer is no. Laboratories produce *a lot* of test data along with other quality and technical records. Thoroughly documenting and storing test data and reports, instrument calibration records, Non-Conformance Reports (NCR) and Corrective and Preventive Actions (CAPA) reports, and staff records is a critical part of any laboratory quality program. Luckily, we live in a modern society where we can easily store data and records on networks and in databases. All records must be stored in a manner that is secure, protected against network/database failures and allows for data to remain confidential.

Documentation can also make or break a lab when it comes to earning a recognized accreditation. Maintaining records of what you do, when you do it, and who did it is imperative. If the test conditions, original observations, calibration records, and test data/reports are not formally recorded, the data is not considered to be valid or defensible [5]. Any result reported to a customer should have a long paper trail associated with it including the date tested, equipment used for testing, who performed the test, who analyzed the data, and other observations.

Non-conforming Work and Corrective Actions: Even the best laboratories in the world make mistakes from time to time. How a lab handles and responds to non-conforming work is a key process that should be looked at carefully when choosing a lab. When a mistake occurs, the laboratory has a responsibility to report the mistake to the customer and put measures in place to prevent recurrence. At this point, the CAPA process is initiated to investigate and solve the problem.

First, the issue must be thoroughly documented and investigated. A root cause analysis will be conducted to identify the cause(s); utilizing a Five Whys analysis is a popular approach to root cause investigations. Once the root cause(s) is identified, the next step is to address the issue by putting together a corrective action plan. What good is it to identify an issue if nothing is going to be done to prevent it from happening again? A laboratory with a good CAPA process will implement and monitor the effectiveness of corrective actions to reduce or eliminate the likelihood of the same problem (or similar) from occurring again.

A continuous improvement mindset is paramount for any lab. Laboratories should continually seek and implement new approaches to improve the quality of data and services.

TESTING

It’s now clear that an extraordinary amount of time and effort is focused on quality related activities such as calibration and documentation, yet for most of us in the lab, performing the tests is the fun part. On an average day, dozens, if not hundreds, of samples make their way through the testing process requiring the use of many different test methods and equipment. Samples are tested according to internationally recognized test methods from organizations such as ASTM, IEC, and ISO. Some specialty and custom testing may be run according to proprietary methods developed by the laboratory performing the tests. Officially sanctioned methods are considered “tried and true” having been thoroughly researched, tested, and approved by a slew of industry professionals. These methods are reviewed on a periodic basis and updated as needed based on the results of new research or technological advancements.

Determining the daily testing schedule in a high-volume environment can be a challenge in and of itself. Laboratory chemists and technicians often rely on LIMS software to assist with workload planning as it will allow them to view the testing required, priority, and due date for the samples which are the first factors considered when scheduling the production for the day. It’s common for labs to plan out their routine sample workflow in advance, often adopting a “first come, first serve” approach and adding in high-priority rush/emergency samples as needed.

There are dozens of different analytical instruments (Table 1) and test apparatuses used in an oil laboratory (Figure 8). Today, many tests can be automated and once the sample is introduced to an instrument, the instrument handles the rest. Some instruments can even provide diagnostic reports. However, there are still many tests commonly used in the industry that do not currently have the ability to be fully automated. Using automated test equipment reduces human error and enables a lab to increase throughput, but machines still have limitations and require maintenance. Oil samples can be tough on sensitive instrumentation and performing routine and preventative maintenance is critical part of day-to-day operations. Having trained staff on-site to perform this work will result in reduced downtime which allows the lab to continue to meet critical customer deadlines.

Instrument Type	Testing
Gas Chromatography (GC)	• DGA • Methanol/Ethanol • Dibenzyl Disulfide (DBDS) • Polychlorinated Biphenyls (PCBs)
High-performance liquid chromatography (HPLC)	• Furanic Compounds • Passivator Content
Fourier-transform infrared spectroscopy (FTIR)	• Inhibitor Content • Chemical fingerprinting/identification • Contamination Identification
Inductively coupled plasma atomic emission spectrometer (ICP-AES)	• Dissolved Metals • Halogens • Silicone
Atomic absorption spectrometer	• Particulate metals
Scanning electron microscope and energy dispersive x-ray spectroscopy (SEM/EDS)	• Elemental Analysis • Material Identification
Dielectric breakdown and power factor test sets	• Dielectric Breakdown Voltage • Power/Dissipation Factor • Permittivity/Dielectric Constant • Volume/DC Resistivity

Table 1: Common Instrumentation and Uses

Figure 8: Oil Laboratory Testing Floor

Most tests do not require a lot of upfront preparation of the samples. Many tests are run on the oil “as is” but there may be times when an aliquot of sample may need to be removed from the original testing container. When this is necessary, it's essential to take care during this process so the sample isn't contaminated during the transfer. Mineral transformer oil is highly hygroscopic, meaning it will absorb water from the atmosphere very easily, even during short duration exposures. Therefore, it's recommended that tests such as water content, dielectric breakdown voltage, and power factor be completed prior to removing a sub-sample since those tests are at a higher risk of being adversely impacted by exposure to the environment.

Routine testing is typically started and finished on the same day. It's a good practice to first run the tests that are easily impacted by environmental conditions, such as water content, dielectric breakdown voltage, power factor, and particle count. Once those tests have been finished, the remaining tests are run in parallel until all requested tests have been completed. Tests such as DGA, are partially automated. Personnel prep the samples by hand and once the instrument is loaded, the GC does the rest. This is an ideal test to set up during the day so the instrument runs the batch overnight. Routine turnaround times for most laboratories is in the seven to ten business day range.

After the test data is generated, it is recorded, reviewed, validated, and stored; most probably in a LIMS. The individual results are cross checked against corresponding data points and compared to previous sample history to look for inconsistencies.

Transformers and other equipment are tested many times over the course of their service life and it's important to track the trend of that data. How the results change from sample to sample helps determine the severity of an incipient fault condition or determine if the oil is aging more rapidly than expected. Therefore, comparing the current data to previous history is one of the most important steps of the data review process and this takes special training and attention to detail. Certain test results are not expected to change much in-service, especially in the six months to a year between samples. However, if there is an issue with the electrical equipment or oil there may be large variations in data in a short period of time. When a lab sees notable changes in the current data as compared to the previous history, the sample should automatically be flagged for verification testing to confirm the original result.

After the data has been double checked by the testing technician, the result is validated and released to a quality control analyst or technical expert for secondary review and reporting.

REPORTING

An oil lab can generate hundreds or even thousands of test results on any given day. Every single one of these results is verified and analyzed by trained personnel before the report is issued. Electric equipment diagnostics can be a complex science, especially in the case of DGA, with so many factors to consider. Although IEEE and IEC guides, and software tools can help with the data interpretation (Figure 9) it's important to get human eyes on the data as even the most sophisticated software tools can miss subtle trends and other details important to the final diagnosis. This is due to the many variables that are considered during the diagnostic review process including meaningful nameplate information (manufacturer, year of manufacture), application (wind/solar, traction), fluid type, historic data, and maintenance and loading history.

The report issued by the lab will contain all the test data along with diagnostic comments and expert opinions regarding the condition of the asset and next steps to be taken, if any. A good report analyst will look at the data through different lenses and use all available information to make sound diagnostic comments and recommendations. There are lots of different diagnostic guides and criteria that can be applied to the data to produce a diagnostic comment for mineral oil filled apparatus such as:

- IEEE C57.104-2019 - IEEE Guide for the Interpretation of Gases Generated in Mineral Oil-Immersed Transformers
- IEEE C57.106-2015 - IEEE Guide for Acceptance and Maintenance of Insulating Mineral Oil in Electrical Equipment
- IEC 60422:2013 Mineral insulating oils in electrical equipment - Supervision and maintenance guidance
- IEC 60599:2015 Mineral oil-filled electrical equipment in service - Guidance on the interpretation of dissolved and free gases analysis
- Limits proprietary to the laboratory, utility, manufacturer, etc.

If test data look particularly concerning and suggests a severe fault condition, the analyst may flag the asset as being in critical condition and request that the customer investigate immediately or pull a confirmation sample. Some oil laboratories employ world renowned experts that can jump in and assist at this point, offering guidance and support for next steps. A report ought to include enough information for the end user to make informed decisions about their assets or oil. Analytical reports should include the following:

- Asset and sample information: Nameplate information, sample data (date of sampling, top oil temperature)
- Criticality: Color coded condition level (green, yellow, red) or IEEE Condition Code
- Suggested resampling schedule
- Diagnostic comments: Detailed remarks regarding the overall condition of the asset and/or oil
- Recommendations for further investigative testing: Dissolved metals, degree of polymerization, corrosive sulfur, contamination investigations, etc.
- Recommendations for follow-up/next steps: reclaiming/replacing the oil, electrical testing, partial discharge (PD) detection, etc.

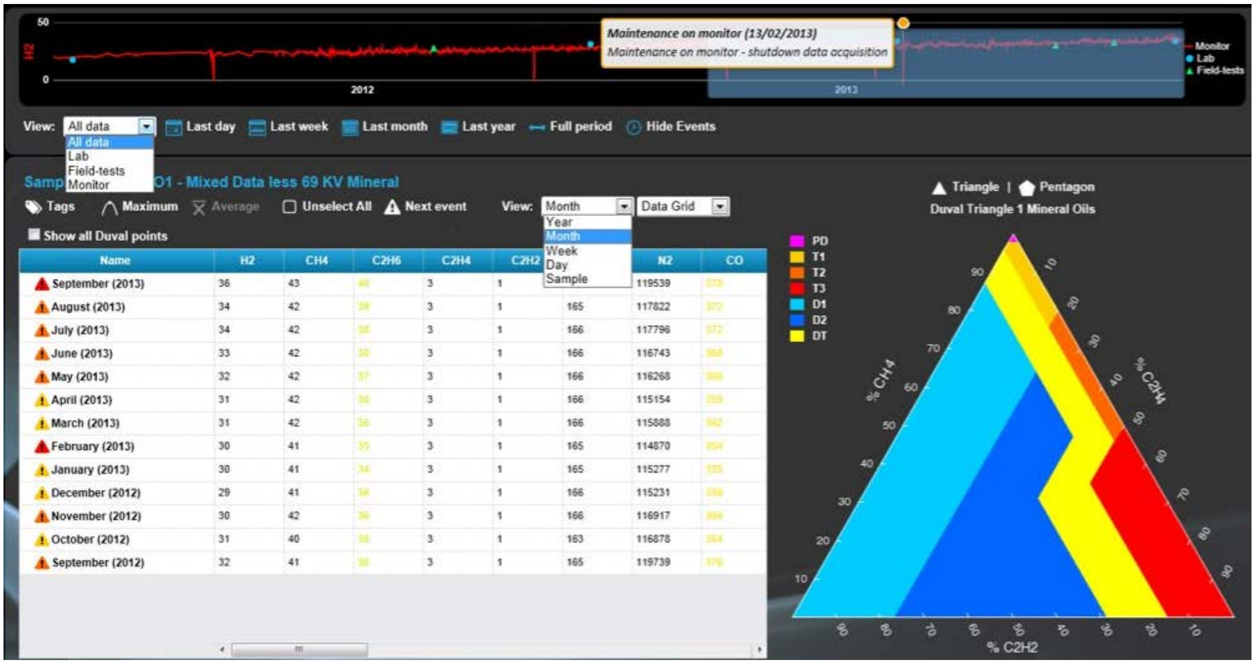

Figure 9: Duval Triangle Diagnostic Tool within Asset Management Software

DISPOSAL

The final chapter in the journey of a sample is disposal (Figure 1). After the report is issued, samples are typically retained in the lab for about thirty to sixty days, just in case testing needs to be repeated or added in the future. However, retaining samples for DGA, moisture content, and dielectric breakdown voltage makes less sense as the samples become compromised after the sample is opened and retest data make be skewed.

Hundreds of gallons of used oil and chemicals are disposed of each month. There are many precautions to take throughout the waste accumulation and disposal process as these operations are regulated by various federal and local government agencies. Before an environmental and industrial services provider picks up the waste for final disposal, the containers housing the waste are stored in a manner that complies with federal, state, and local regulations. Laboratories will have trained personnel on staff to manage waste and ensure compliance with all regulations.

Government regulatory agencies will have detailed instructions on how to store and dispose of chemicals. It's the lab's responsibility to ensure compliance and make sure that all chemicals involved in the process are tracked and disposed of in clearly labeled containers that are compatible with the waste stream. This is usually a straightforward process as most oil labs don't use many different types of chemicals. However, samples containing polychlorinated biphenyls (PCBs) are handled separately and often require special paperwork and handling considerations.

Chemicals, especially those used as reference standards, should be used, and disposed of prior to their expiration date. Most chemical and oil products do not wear out, they just get dirty, so recycling saves valuable resources and has environmental benefits [6]. Used oils and chemicals can be blended into fuel or re-refined into base stock or other products. Fuel blending is a common practice as BTU values can be quite high and therefore can be burned as fuel in several industrial applications.

MORE THAN MEETS THE EYE

Now that you've been given a peek behind the curtain, you can see there is a lot that goes on behind the scenes in an oil lab and day-to-day operations are considerably more complex than they may seem on the surface. The entire process from sampling to reporting requires many administrative, technical, management, and field personnel who utilize advanced analytical equipment and testing techniques, software, and data analysis skills. Laboratory technicians and chemists are responsible for more than just pouring oil into beakers. A career in an oil laboratory can be very rewarding and challenging. There is no limit to what you can learn in this industry.

The data oil labs issue is of critical value and many important safety and financial decisions are riding on the outcome of the oil testing. Therefore, choosing a laboratory should not be a surface level decision based on solely on factors such as location and price. Making an informed decision driven by data quality, testing needs, and technical expertise will only add to the value of the asset owner's asset management program.

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BIOGRAPHY

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