TESTS

The machine and the lubricant can telegraph hints and signals to us in a variety of ways, but only if we are both tuned in and literate to their message. Tuned in means being vigilant and ubiquitous, like a detective, always looking for clues even when camouflaged from view. Literate means not only recognizing the presence of the clue but also being wise to the meaning of its message and the corrective response.

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http://digitaleditions.walsworthprintgroup.com/publication/?i=284578&ver=html5&p=34#{%22page%22:38,%22issue_id%22:284578}

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Joseph FOTUE, CLS

I met a strange situation with the measure of TBN in one Laboratory. I noticed that out of three different gas engine oils that have almost the same TBN (around 5.5), the lab always has difficulties to measure the TBN of one. The method used is **ASTM D 2896**; this method recommends to measure TBN with an oil sample mass m = 10/TBN expected. The lab used to take 1.82 g as mass of the sample that is analysed. The value obtained for this oil is generally 1 mg KOH/g (Different from 5.5). The lab told me that when they change the mass of the sample of this oil (from 1.82g to 0.9 g), they obtain a value near to 5. The problem is that they don't know exactly what is the optimal mass in order to get the right answer.

Sarma: Please use grams of sample = 28 / Expected TBN

http://www.asiajuleh.com/astm/ASTM%20D%202896.pdf

Fotue: Thank you for inputs. The three oils have the viscosity grade SAE 40. Please Sarma, I read that the formula 28 / Expected TBN is used for procedure A whereas the lab follows the procedure B.

Boris Zhmud The lower TBN, the greater sample is required for accurate determination. If the sample is too small, the measurement error may exceed the actual TBN value, and the result you get may look really weird. The majority of GEO products have TBN in the range 3 to 7.

Robin McNabb If you look at the appendixes of the ASTM D2896 method, it lists that the ranges of base number used for the precision and bias calculations of used oils were lubricants with base numbers from 5-27. ASTM D2896 may not be the most applicable base number method to use for low ash oils

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What are the reasons, TBN goes high in used engine oils. Though it is surprising but it do happen sometimes. Typical TBN value is 20 mgKOH/g but we got 25.24 mgKOH/g. Sampled from sump drain. **Mahesh Poojary**

Anthony Gomes

Possibly due to top up with a another grade having higher TBN value. The elemental analysis by ICP can help in detecting such occurrences.

Asad Perwaiz

Yes. It often happen. When you test your sample using ASTM d2896 instead of ASTM d4739 which is recommended for used oil. The reason behind is very simple, fresh oil contain only additives those are base in nature but used oil contain both additives those are bases and by products of combustion, oxidation, those are too bases. Some time concentration of such by products become so high and if we use ASTM d2896 which uses strong acid in test give exaggerated value because strong acid neutralize both , real. Additive and by product (undesirable} during testing. This is why ASTM d4739 is recommended for used oil.

V.S.S. Sarma

ASTM D2896 employs perchloric acid as the reagent, versus ASTM D4739, which employs hydrochloric acid, a relatively weaker acid than perchloric acid, when applied to used oil analysis.

D2896 method may be better because the strong acid neutralizes strong and weak alkaline components in the oil, providing a more realistic measurement of the true base number, while D4739 only measures the strong alkaline components.

Some chemists believe that the strong acid used in D2896 reacts with weak alkaline wear metals in the oil, producing a falsely exaggerated base number. D4739 measures only the additive package, which is there to neutralize acids, making it a more accurate test for used oil analysis.

Jean-Michel Demaret

It is important that first you enquire with the lab what method they are using and if they have changed. The 3 methods I am aware are ASTM D2896, D4739 and IR. This last method which is preferred by the labs (cheap and fast) is neither precise nor accurate (my point of view only) .
ASTM D 4739 and ASTM D2896 have a similar repeatability (same person, same lab, same method, same equipment) of 1 mg/ KOH for sample in the 20 TBN mark. (95% of the results for the same sample will be within +- 1 mg/KOH). These values are certainly for relatively new oil, when the oil is worn out, the precision will be less.
It is possible that the TBN additives (Calcium sulphonate) becomes more active at high operating temperature and boost the TBN, however the rise won't last.

Peter Young

I had an instance of a TBN rising by 2 above new oil after 500 km when measured by FTIR. Retesting by ASTM D2896 showed the expected result of near new oil. Checking further I found that there was a coolant leak which interfered with the FTIR.
FTIR measurement works by measuring the COO functional group of the salt produced when Trifluroacetic Acid (TFA) is reacted with the basic ingredients in an oil.
Quantification is based on the difference in spectra before and after adding TFA.
A recent example of a contaminated 5W-30 diesel engine oil showed an FTIR TBN of 10 after 1 week. This had a new oil value of 7 when measured by FTIR. When measured by ASTM D2896, a more logical figure of 7.0 was obtained.
As Sodium and Glycol are both present in the oil when a coolant leak occurs, this can lead to falsely high TBN results.
In these circumstances, reversion to the wet method ASTM D2896 is advised.

In October 2016:

Joseph FOTUE, CLS Lubricants Engineer At TOTAL

TBN measure - ASTM D 2896

Dear All, I met a strange situation with the measure of TBN in one Laboratory. I noticed that out of three different gas engine oils that have almost the same TBN (around 5.5), the lab always has difficulties to measure the TBN of one. The method used is ASTM D 2896; this method recommends to measure TBN with an oil sample mass m = 10/TBN expected. The lab used to take

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Raymond Abraham Had a similar experience where one oil was below the condemning 50 % of fresh oil TBN, while the other used gas engine oils were well above. With low TBN levels there is an increased risk of acidic corrosion, engine wear and reduced component life. If other data like wear metals and oxidation levels are fine, then monitor the TBN at the next sampling event.
Gas engines require low ash GEO, with a TBN of 4 – 7 for fresh oils. Typically the TBN drops faster in the beginning but gradually levels out towards an equilibrium level. This assuming that the new oil top up levels are in keeping with the oil consumption (which is significant for gas engines). Close monitoring of new oil top up helps TBN reach equilibrium levels sooner.

Mark Minges, CLS, OMA I, OMA II Just a comment! Are the oils you had tested new oils or used? If they are new than D2896 is a good method however, the lab should use a minimum of 2g of sample for TBN values around 5. The less mass of sample the wider the repeatability. If it is a used oil you should have the lab run D4739 using the heated method to take the interference from ZDDP and possible water levels above 300PPM. D2896 is a designation for test TBN on new oils, where as D4739 is for used oils. Many labs will run lower mass samples to speed the test up to improve production. I agree with Robin McNabb! TBN is a tricky test and requires using a proper mass of sample as well as using appropriate testing methods to reduce error factors from possible interference from water or ZDDP (Anti-Wear Additive).

Dirk Beukelaers Chevron study : It is important to note there are several test methods for Total Base Number. The one used in product data sheets is generally ASTM D 2896. This method uses perchloric acid to neutralize the alkalinity in the oil and yields a slightly higher number than the test method used by the oil analysis labs. They generally use ASTM D 4739 and the acid used here is hydrochloric acid. This produces a number approximately 2 mg KOH/g LOWER than ASTM D 2896 for the same oil. Due to chemical interferences, this test method does not recognize all of the alkalinity that ASTM D 2796 sees.

Muhammad Asim Dear 1st check which procedure you are using are there are two procedure one is 120 ml TBN solvent and other is 60 ml TBN solvent is used

Denisa Ivana Hello Joseph .Generally measuring TBN in used oils is difficult. Sometimes you need to get the used sample filtered too and you have to use more used sample .ASTM 2896 is not precise for values near to TBN- 1. With new samples usually there is no problem.You can use 1 g of sample if you do not suspect a high TBN value.The most important is to get the right curve with the clear inflection point.If you suspect in the new oil (some marine for example) a high TBN then try with less mass (0,4 g).You surely will measure it.

Elena Yolova ASTM D 2896 is most suitable for fresh engine oils.

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3 Things that Should be Customized for Every Oil Analysis Program

July 30, 2015

1) Flagging Limits

The limits established by the equipment and/or lubricant manufacturers are a great place to start in establishing warning limits, but shouldn’t be the “be-all and end-all”. No one knows (or shouldn’t know!) more about your equipment and operating environment than you do. Therefore it’s only logical that you have the final say in what results require immediate action and which ones may only require monitoring. The pre-set limits may need to be stricter on sensitive or extra-critical equipment or less-stringent to prevent false alarms.

Repeatedly flagging results that should be considered normal in your specific situation is an unnecessary strain on your already limited time and resources. These limits should also be dynamic and have the ability to be adjusted overtime as your cleanliness standards increase.

2) Testing Packages

Most laboratories have standard test slates available based on the application and/or component being tested. These are great for convenience or those just beginning with their lubricant analysis program, but those with a more mature program require more customization with their routine and exception test packages.

If you know exactly which parameters are important for you to track and which ones are less important, then you are in the best position to optimize your testing for efficiency and cost-effectiveness.

3) Reporting Format & Layout

Your ideal report layout allows you to gain the following insight as easily and efficiently as possible:

- Readily identify the condition of the component and product being used.
- Know exactly what action, if any, should be taken.
- Review raw data for the performed tests – blank tables for tests that weren't performed make this step more difficult.
- Trace the trending for this particular unit in a visual format – line chart, pie chart, etc.

Similarly, the report should be available in a format that works best for you. PDF's are standard in the industry, but what if you don't have internet access? What if you want the report on your smartphone? What if you want to integrate your oil analysis reports with your vibration data (without manually typing the raw data)?

What do these all have in common?

Communication.

Customizing your oil analysis program to fit your needs can only result from a healthy and on-going dialogue between the end-users and the laboratory. Make sure the lab is aware of your operating conditions and any important changes that could affect results. Email is an excellent tool but will probably not suffice for these steps. Site visits are ideal when possible but other technologies such as Skype or FaceTime can be just as good (or at least a close second).

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Get Answers To These and All Your Questions About Oil Analysis!

- How often should I use oil analysis?
- Where is the best place to get an oil sample?
- What are the benefits and drawbacks of screening oil samples before sending them to my lab?
- How clean should I keep my oil and what type of filter should I use?
- What are all these numbers I see on my oil analysis report?
- How do I know which oil analysis lab is right for me?
- How do I set caution and critical alarms for wear metals and additives?
- What steps can I take to ensure that I get a good sample each time?
- How do I determine the remaining useful life of my oil?

- How do I know if I should occasionally "sweeten" my oil with additives?
- What is the best temperature to trend viscosity at?
- What are the secrets to catching bearing faults with wear debris analysis?
- Are there any good field tests for oil that don't involve expensive instruments?
- What are the five most important things I should be looking for on my oil analysis report?
- What are the best cost reducing strategies using oil analysis?

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Volatility is an important engine oil property, particularly as lower viscosity oils become more popular. There is a direct association with an oil's volatility characteristics and oil consumption rates. ASTM 5800 is the standard test method for measuring engine oil volatility by the **Noack** Method. This test method covers three procedures. Procedure A uses the Noack evaporative tester equipment. Procedure B uses the automated non-Woods metal Noack evaporative apparatus. Procedure C uses the Selby-Noack volatility test equipment.

ASTM 5800 is one of the test methods being monitored by the Test Monitoring Center (TMC) based in Pittsburgh, Penn., U.S.A. There are seven participating labs with a total of 21 stands. According to the most recent TMC report, comparing the past 19 months of data on the new reference oils to the prior 19 months on the old reference oils showed a modest decrease in overall severity and "somewhat worse overall precision."

The report does not specify which procedure was actually trending severe, however it's been common knowledge within the industry that Procedure B has had poor precision for several years. However, the Surveillance Panel decided not to declare the test out of control. Instead, three task forces have been formed to address the problem, it was heard at the ASTM Subcommittee D02.B on Automotive Lubricants meeting on 24 June 2015 in Fort Lauderdale, Fla., U.S.A.

A task force has been formed to improve the performance of Procedure B and another to improve the performance of Procedure C. A third task force has been formed to propose warning and action limits and develop control charts, which will allow the Surveillance Panel to define in the future when the test is "out of control."

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Question:
What are the two wear metal detection methods employed in oil analysis?
Answer:
Ferrography (ferrous density analysis) and emission spectrometry

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OILPAS: Real Time Particle in Fluids Analyzer

Imaging system for particle-in-oil detection, classification, counting, and measurement

http://conexotest.com/conexooilpas.html

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PERMISSIBLE LIMITS FOR WEAR METALS IN USED ENGINE OILS

Samir Azzi

Owner & Managing-Director of Cedars Management Consultancy

Ashis, Please specify what type engines are you talking about..Gasoline or Diesel ...for diesel engines, normally every OEM would have different limits related to the metals used in his engine parts. Type of application of your equipment is also important:
1-on-road
2-off-highway
3-mining

Once you can identify the above, providing limits would be more appropriate..However in general, herebelow are common limits for Diesel Engines:

1-Iron >100ppm critical
2-Chromium >15 ppm , critical
3- Lead > 40 critical
4- Copper > 45, critical
5- Tin > 20 ppm, critical
6- Al > 20 ppm, critical
7- Silicon > 15 ppm, critical
8- Sodium > 30 ppm, critical

But what is more important than the above figures is to monitor the trends of your wear metals in used oil analysis...

Douglas McGregor   Lubricants and Lubricant Additives Specialist

Ashis, I second the comments provided by Samir above. In my opinion and experience, condemnation limits are meaningless. A useful and successful preventative maintenance program involving used oil analysis will utilize trend data (statistical analysis of data trends) to determine when to "flag" a given result as abnormal and in need of further investigation. Often times, the abnormal result will be well below a given condemnation limit, but may allow for an issue to be addressed prior to reaching a condemnation limit, when it will be likely too late to address and avoid more costly maintenance actions. Perhaps the information I found in my files from Cummins regarding this issue will be helpful as well... provided below:

"Used oil analysis can monitor engine oil contaminant levels and provides evidence of dirt ingress, excessive fuel contamination (dilution), coolant leaks, excessive soot accumulation, and abnormal wear.

Elevated levels of silicon in the used oil indicate dirt contamination of the oil, usually caused by faulty intake filtration. At times, dirt contamination occurs through the oil side of the engine from contaminated engine oil. The used oil can also contain abnormal levels of copper and lead from bearing material wear, without extremely elevated levels of chromium and iron.

Abnormal wear causes abnormal accumulation of wear metals in the used oil. Condemnation limits are not possible except for engines in an application operating at one site on a single engine oil. The wear metal content of an oil sample depends on the engine, the load factor, the capacity of the lubricating system, the miles or hours on the oil, the engine oil consumption rate and so on. Engines with large oil capacities result in lower wear metal levels than engines with smaller oil capacities because the particles are suspended in a larger volume of oil. Low oil consumption engines exhibit higher wear metal levels than high oil consumption engines because of the absence of dilution by new oil between oil changes. Engine oil consumption rate can change the wear metal content of the used oil by a factor of two and mask doubling wear rates which can indicate engine damage. Wear rates are unknown unless the engine oil consumption is known. Wear metal levels vary nearly linearly with the miles or hours on an oil sample. Doubling the miles or hours on an oil sample nearly doubles the wear metal content of a used oil sample. Wear metal content of a used oil sample is almost meaningless information without the engine oil consumption rate, the miles or hours on the oil sample, and a new oil analysis.

Abnormal wear, which can indicate a problem, usually only involves elevated levels of one or two metals. Detection of elevated levels of a wear metal is best performed by comparing the levels in the used oil sample to the levels in previous oil samples from the same engine. Engine components containing copper and lead can become chemically active with a change in the additive chemicals in the oil that is often accompanied by switching to a different brand of oil. This often results in dramatically increased levels, often ten times, of copper or lead in used oil. Increased levels from this source is not reason for excessive concern. These components will become passive after a few oil changes with different oil. Wear metal levels will then slowly decline back into the normal range for the engine.

Never disassemble an engine based solely on used oil analysis. Perform additional troubleshooting to determine if a problem exists. If a problem is suspected based on oil analysis, cut open the full flow oil filter and look for wear metal particles that are trapped by the filter and easily visible."

Richard Hassebrock   Field Engineer at Castrol Heavy Duty Lubricants

Wear metal limits have not much meaning at all without being referenced to a specific oil age (in miles, kilometers or hours). There's a big difference between 100 ppm of Iron at 500 hours and the same level at 250 hours oil age.

thierry chaponnay   Regional sales manager chez IESPM

I Totally agree with Richard, you have through oil analysis to mesure the tendency. I could add that each kind of motor has its own signature, on the same time/km basis 100 ppm Iron could be normal for one motor and totally abnormal for another one. You can't determine general values. Oil analysis is relevant from the third analysis and with the information below:
- using conditions
- type of lubricant
- oil hours
- motor hours
- type of motor, brand, model,...
- oil, diesel quality
- maintenance operation,...

And the most important the sample quality, it has to be taken in good conditions and representative of the bath.

Karim Ibrahim   GM & Consultant

Mr. Ghatak, no such standard exists as such as there are too many variables, for example, the levels of wear metals will vary significantly according to what protection level of oil you are using so if you get say 500ppm Iron with a cf-4 oil there will be no reason to frown but the same level with a CI-4 oil is reason for worry. Also the working conditions and quality of air coming into the engine thru different qualities and conditions of filters will definitely be a factor for each type of application the oil is running in. and finally in UOA Metal trends along with the rest of the oil's condition must be looked at; wear metals alone do not tell the whole story.

Shesby Chabaya   Head of Operations Department at Tribology Services

I agree with all the contributors above. They have raised varying points which cover the subject extremely well. Allow my to add on to the discussion by asking this question: What are the typical normal levels of copper on a Retarder for a Scania G380 Tipper Truck? Secondly what is the importance of measuring the metal content in a ratarding system?

Thomas Hanemann   Key Account Manager Industrial Lubricants bei Idemitsu Lube Europe GmbH

I agree to To the comment of Samir. Would suggest to make a Trend analalysis
For every 1000 to 2000 hours of Running Time.

Mile Stojilkovic   Marketing & Development lubricants Director at NIS Gazpromneft, Department Lubricants – NISOTEC

The engine oils, origin of elements can be of additives, wear, from fuel from the air and liquid cooling. Metals from the additives may be Zn, Ca, Ba, or Mg, and indicate the consumption of additives. Metals originating from wear are: Fe, Pb, Cu, Cr, Al, Mn, Ag, Sn and point to increased wear in those parts of the mechanism. The elements that originate from the coolant as Na and B, and increased content indicates the penetration of coolant. Increased content of Si or Ca, which originate from the air, indicates defective or bad air filter.

Damanik Ramidi   Condition Monitoring at Lenzing Group

I Have experience at Oil monitoring practice Of Heavy equiptment at mining operation, specially at Coal and Copper mining, both Open Pit and underground. The main type application of Diesel Engines with above 3000 HP:

1-Iron (Fe) >95 ppm critical and Fe > 45 warning
2-Chromium (Cr)>15 ppm , critical and Cr > 5 warning
3- Lead (Pb) > 80 critical and Pb > 25 warning
4- Copper (Cu) > 30, critical and Cu >10 warning
5- Tin (Sn) > 15 ppm, critical and Sn > 5 warning
6- Aluminium (Al) > 16ppm, critical and Al > 8 warning
7- Silicon (Si) > 35 ppm, critical and Si > 15 warning
8- Sodium (Na) > 100 ppm, critical and Na> 50 warning
9-Nickel (Ni) > 15 ppm, critical and Ni > 5 warning

Those all limits is more important to check the new oil to compare as refference standard for trending analysis.

kapil verma   Senior Executive - Technical Services (PAN India)

iron 250ppm and silicon 50ppm

Zeeshan Qureshi   Technical Advisor - Grease & Lubricants at Shell

Trend is your friend. Just follow the wear trends, history and generation rate.
Every engine will have its own permissible limits based on trend.

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Pour Point

The pour point of an oil is defined as the lowest temperature at which a lubricant will flow. It is frequently and erroneously used as the oil viscosity selection criteria.

For example, let's say an oil has a pour point of minus 30 degrees C. Most people assume that this means that the oil will flow to the bearings of the equipment even when the ambient temperature is at minus 30 degrees C. This is a fallacy. At best, this oil with a pour point of minus 30 degrees C and operating in an ambient temperature of minus 30 degrees C will merely churn at the oil pump until the churning causes an increase in the oil's temperature. This in turn allows the oil's viscosity to thin sufficiently so that it slowly begins to flow through the oil passages to the lubricated components.

Frequently, this process takes 5 to 10 minutes or more, during which severe damage can occur at various components, because the oil is actually too thick to flow. Do not select lubricants based on pour point alone.

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There is almost no relation, as **CCS is measured by ASTM D5293 and Pour Point** by ASTM D97. These are very different as you measure fluid in different state - moving and non-moving. I've seen oils in my practice that can have almost CCS limit at -25 C and pour point of -39 C. Plus ASTM D97 has an error of the method around 9 C.

I myself against statement of pour point in technical data sheets of lubricants. It gets end users confused as they try to use it at -35 and then complain about equipment failure due to "bad quality oil" disregarding everything else. "But it is usable down to -40!" is their argument.

Mahdi Vosough The Cold Cranking Simulator Test (ASTM D5293), measures lubricant viscosity under cold start conditions; since it measures viscosity at high shear rates, fragile wax structures are broken down mechanically
and do not adversely affect CCS viscosity. Therefore, the PPD is not effective in lowering CCS viscosity.

The CCS test uses relatively high shearing rates and rapid cooling and so even though wax is crystallised it does not form a destructive network that traps the oil. PPDs inhibit only the wax structure network rather than the quantity of wax precipitated they have no influence on the CCS test.

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Anti-foamants are typically **polydimethyl siloxanes** of various molecular weights and are only required at a few parts-per-million concentration to control lubricant foaming. It should be noted that the siloxanes are dispersed in the lubricant in the form of small particles or globules with specialized equipment. These small particles can be seen through the use of a phase contrast **microscope similar to that used in clinical laboratories**.

Measuring the viscosity of a diesel engine oil at 100 C, **fuel dilution** can be masked because some of the diesel may evaporate during the test?

Ramesh K   Chief Manager (Quality Control) at Indian Oil Corporation Limited

Diesel does not eveporate at or about 100 deg.C, and hence fuel dilution will be there even for KV@100 deg,C test. The IBP of Diesel is far beyond that temeperaure...

Larry Hajek   senior lubricant technical manager at Citgo

Fuel dilution is only one cause of viscosity change and Ramesh is absolutely correct. the IBP of #2 diesel is over 150 C.

Jean-Michel Demaret   Qualified Engineer / Senior Account Manager
The IBP is the temperature at which one of the main component of the diesel generates a vapor pressure of 1 atmosphere. But before this temperature a vapor pressure is generated, which means the lighter end of the fuel would have evaporated. Flash point of diesel is a minimum of 63 C. So at the flash point temperature some gas evaporates. Water in a dish would evaporate inside a house at ambient temperature, far from 100 C. I know in a Cannon viscometer there is a fan to draw the vapours out.

I looked at 17 results (between 200 and 300 hours in operation), same RVI engine, 2 diesel engine oils Shell Rimula and Mobil Delvac both mineral SAE 15W-40.

Average viscosity of the results
at 40C : 80% of the viscosity of the new oil.

at 100C : 86 % of the viscosity of the new oil.
6% difference between the ratios

2 results out of the 17 have 10% difference between the ratios

I try to understand such a difference

Now if I take another engine (Cat C15) same oils (Rimula and Delvac), no apparent fuel dilution
20 results

Average viscosity of the results
at 40C : 98.7% of the viscosity of the new oil.
at 100C : 98.9 % of the viscosity of the new oil.
0.2% difference between the ratios

Ramesh K   Chief Manager (Quality Control) at Indian Oil Corporation Limited

From the data of your own also KV@40 and KV@100 are not changing any significantly at 100deg.C as compare dto 40 deg.C , which should have been very high than 0.2% difference, if at all there is loss of fuel into atmosphere while testing.
But your study done is very good and appreciaile for proving the dilution of Diesel fuel do make difference of viscosity both at KV@40 & KV@100 significantly and also can be proved by FTIR used oil analysis test for fuel dilution etc. When both tests are positive, only then the corroboration of diesel fuel dilution in Lubricant becomes meaningful confirmation. Confirmation alone by KV40 & KV100 would not be correct without study of FTIR done on the sample of used Engine Oil.

Ramesh K   Chief Manager (Quality Control) at Indian Oil Corporation Limited

The method of ASTM D 3524 GC for diesel fuel dilution is obsolete from 2013 and only ASTM 3525-2010 is to be used and with an accuracy of some good order for those which have more than 1.64% wt dilution. Compared to this it is more easier to do FTIR for the same under used oil analysis.

Anand Gupta   Assistant Manager, R&D Lubraicants at Bharat Petroleum Corporation Limited

FTIR is easier no doubt about that, however quantification, below 2% FD is not as precise.
Based on the viscosity loss @100 deg (14%) and (20%) @40 deg data for used oil, it is difficult to say that it is because of fuel alone; soot content and other oxidation by products present in the oil may cause this variation because during oxidation process oil molecule's structure and properties changes.

Jean-Michel Demaret   Qualified Engineer / Senior Account Manager

Thank you for your comments. For the enjoyment of a technical discussion I may add few points. Using blending calculation tools I found an estimated diesel content (2 cSt @ 40 C) of around 3% at 40 C and 2% at 100 C. About 1/3 may evaporate, or should I assume 1% sample weight. The lab I am using now is at sea level. Some cities like Denver or Mexico City are at 2000 meters altitude, as well as many mining sites in the Andes, the evaporation is a lot worse (previous lab I was using was 3000m high).

Finally doing a GC may tell me that there is a fuel presence. I can do a flash point and find out if there is fuel or not and correlate to the blending data. Flash point can be done in any lab. I would use a GC if I can reproduce the heating process of a cannon viscometer, and compare a sample without heating and a sample after heating.

Last point may be more for Mr Anand, if there is an increase of relative viscosity because of the chemical reactions and change of the molecules structure of the oil or the contaminant, I should see the same phenomenon with the results of the Cat C15 engine. The Cat C15 engine results have no fuel dilution and their relative viscosity to a new oil are steady.

Larry Hajek senior lubricant technical manager at Citgo

A bigger concern is light fraction from blends of poor quality base oils.

Jean-Michel Demaret Qualified Engineer / Senior Account Manager
It has occurred to me that there was a defect in the oils. Usually Shell Rimula and Mobil Delvac "meet the specs" even if they may not be considered the best in their class.

The light fraction evaporating would have thickened the oil because only the more viscous component would have been left behind. VI improver breaking down would have had more effect in lowering the relative viscosity at high temperature than at 40 C I suppose.

So we may have a double issue, The VI improver breaking down dropping the visco at 40 C and 100 C plus the Light fraction evaporating thickening the oil at 100 C for both oil brands. We do not have these phenomena with the Cat engines.

Peter Weismann Technical Director bei OelCheck GmbH

European laboratories detemine the fuel content. In the OelCheck laboratory report (also in China) you will find the fuel content as Diesel fuel, biodiesel in the diesel fuel as well as gasoline. The values are created by GC acc. to DIN 51454 (ASTM D3524 or ASTM D3525) only. Nobody would trust a viscosity measurement @100 °C only. If viscosity should support, at least 2 viscosities should be measured and the VI should be calculated. IR was a good method app. 20 years ago when Diesel contained aromates and much more sulfur. Also the Spectro Fuel Sniffer was good for some years for old type diesel fuels. The GC or the 2 viscosities is making oil analysis more costly, but also more credible.

Ramesh K Chief Manager (Quality Control) at Indian Oil Corporation Limited

Condition monitoring of used lubricants by Fourier transform infrared (FTIR) spectroscopy is reviewed and placed in context of the recently approved ASTM Practice E 2412-04 developed by the Joint Oil Analysis Program (JOAP) as a standardized means of trending oil/lubricant condition. I vouch for this method for the Fuel Dilution also as projected and hence is also very authentic method and the Software of Pekin Elmer and Thermo are excellent aids for the used oil analysis for knowing data including Fuel dilution etc... the only thing is you should know & have the original oil too....for test.

Peter Weismann Technical Director bei OelCheck GmbH

The ASTM is more than 10 years old. Usually ASTM-standards are reworked after 5 years. We are member in this working group for renewing the standard, but no consense could be found. Especially in diesel fuel calculation the method could never be used for the diesel type in Europe. In the Standard you find the note 10: Fuel calibration ..... needs to be adjusted to the local variations in fuels. Even so PE or Thermo have included those methods into their calculation it does not mean that those are valid all over the world. We checked in a used oil group in Germany, were the laboratories of the engine and car manafucturers as well as the laboratories in test rigs are participating in round robins (head is Mr. Trick from Mercedes) and could not find any correlation of the IR-methods to the real world results or the GC-methods.

Ramesh K Chief Manager (Quality Control) at Indian Oil Corporation Limited

ASTM E2412 - 10 Standard Practice for Condition Monitoring of Used Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry
Use the latest 2010 method which is more authentic for used oil anaylsis , where in Fuel dilution is also one of the predictions of Oil condition montitoring. If combined with physical tests, this tool of

FTIR can be useful for sure. And we can not have more methods of our own when already ASTM has taken into their standards from JOAP to confirm the usabality after many years. If any other better method is known to any one the same can be shared for the guidance of all. But in absence of any authentic better method than this it would be usable only for all, whether in Europe or US or any other country..

Mark Smith, CLS
When using Gas Chromatography to measure fuel dilution, the standards should use the lower end of a distilled fraction (of the regional blend, as Peter says) to simulate any heat-related losses (mainly crankcase oil heat and combustion heat). FTIR analysis looks directly at the parts of the fuel that are not highly affected by losses of the volatile portions. You should see a viscosity correlation if any sheardown of multigrades has not happened. Viscosity alone is not a measure of fuel dilution, since there are factors influencing an increase in viscosity that can be happening at the same time.

Larry Hajek   senior lubricant technical manager at Citgo

we are all thinking on the lab side of this question, what if we look at the engine. the engine has a oil temp of at least 90 C and the main gallery of 150 C with areas underneath the piston of above 200 C. if volatiles were present they would be vented from the crankcase.

Jean-Michel Demaret   Qualified Engineer / Senior Account Manager

On the Fuel by FTIR, I must admit that result I obtain for Fuel (and Glycol) through FTIR are imprecise enough to be disappointing. GC is quite an expensive test.

In large plant/mining sites, with their own testing equipment, we usually confirm the low viscosity with a measure of the flash point. From the flash point and viscosity you get 2 estimation of the fuel dilution and they should correlate.
I believe the drop of viscosity because of fuel dilution has more impact on viscosity than the thickening by oxidation. Fuel dilution will prevail.

The initial question is really: could a measure of the viscosity at 100C mask partially a fuel dilution. The example given was 2 engines, one with fuel dilution and showing a different relative difference of viscosity with a new oil measure at 40 C and 10 0C. One without fuel dilution showing no relative difference with a new oil at 40 C and 100 C. In the first case the oil seems to thicken, in the way that it has a higher VI than the original oil (144 instead of 136).

Larry has a point saying when the engine is operating, the temperature of the oil in a diesel engine reaches 90 C and intermittently up to 150 C,

Anand the viscosity of the new oil is 114 cSt@40C / 15cSt at 100C (VI 136). Used oil is average 89.5 cSt/ 13.0 cSt (VI 144) I would have expected 89.5cSt/11.8 cSt

Peter Weismann   Technical Director bei OelCheck GmbH

What you described is exacly what we found when we compared the GC-results (http://www.oelcheck.de/en/oil-analysis-tests/fuel-content.html) with VI and other parameters. In used SAE 15W-40 and 10W-40 an increase of the VI by 4-5 points per percent of fuel is a typical result.This means we find usually a VI of 145 instead of 140 for SAE 15W-40 oils if they contain app. 1% diesel fuel. We also found the change in the density is a much more accurate value as the numbers produced by the FT-IR. More information about fuel dilution you will find in the OelChecker, but when this was issued in Summer 2001 we had it only in German. However you may understand the diagrams on the bottom of page 5 and 6.
http://www.oelcheck.de/fileadmin/oelcheck/pdf/oelchecker/sommer2001.pdf
The described Spectro "fuel sniffer" did work well when it was calibrated against the real diesel fuel

(like in test rigs) which did change with ervery shipment. Therefore for samples coming from more than 3.000 different customers it was not accurate egnough. This was the reason why we were looking into the comparison with fresh oils on density, calcium content, viscosity change at 40°C (100 is not sensitive egnough) and even flash point. After PE came out with a fast dedicated GC (app. 5 inutes per sample because of a backflush function, we went away from the inaccurate methods to the GC.
The German way is different to the American and getting more detailed information. Of course the price will be higher than with cheap methods, but it is useful to pay more for quality and a better interpretation.

Hi Peter, Finally got round to this old discussion, "Hello Everyone. Would you think that when measuring the viscosity of a diesel engine oil at 100C, fuel dilution can be masked because some of the diesel may evaporate during the test?"

http://www.oelcheck.de/en is a wonderful web site with many pages useful for training. A real treasure!

I understand the diagrams on the bottom of pages 6 and 7 in http://www.oelcheck.de/fileadmin/oelcheck/pdf/oelchecker/sommer2001.pdf - very good stuff. But it is not printed without errors on page 6. As you say, the first should be density. Calcium content not %.

Thank you! Petr

Jean-Michel Demaret Qualified Engineer / Senior Account Manager
TBN is an odd test .If the calcium deplete hence the TBN drops as well. If the engines operate with 5000 ppm sulfur diesels, the TBN results have a very wide standard deviation for engines with similar operating parameters.

For these 2 tests, the positive fuel dilution symptoms can be seen only if the leak is "old". New ingress or sudden increase, say in the last 50 hours before oil change , may not be seen as the additives won't have time to react. This is how I understand some of the results.

I looked at visco-temp charts for Diesel, MDO and MGO and I calculated that the VI is in the 80 range. I am not clear why we have this increase of VI when the oil is diluted beside of evaporation.

Peter Weismann Technical Director bei OelCheck GmbH
My article was written when sulfur in diesel fuel was app. 1.000 ppm. Nowadays sulfur in fuel all over Europe is less than 10 ppm. Only in Italy and France it is permitted to operate farming and off-road construction equipment by using heating fuel, which basically fulfills the diesel spec but has app. 1.500 sulfur content. As long as the "old" diesel fuel was in use, the IR and the other methods were more precise, but now it is very hard (and by using the GC-method not necessary) to find strong correlations. We do more than 20.000 diesel engine samples (each including IR and GC) a year out of a total of more tahn 300.000 mainly industrial oil samples.
The Calcium content in our lab is very precise also at the high end because we set 6 calibration points at 10, 100, 500, 1.000. 2.500 and 5.000 ppm.

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Jean-Michel Demaret

Qualified Engineer / Senior Account Manager

The reproducibility of RPVOT test is around 22%. You can have your oil tested by 2 reputable labs giving you acceptable measured values between 936 and 1464 minutes. (ASTM D2272 mentioned that the repeatability is 12% (same sample same lab same operator) and reproducibility is 22% (same sample 2 different labs))

Is the Base Line from the data sheet (specs may change) or measured from a lab?

In my experience 100% of strange RPVOT were due to the poor reproducibility and the expectation of the user that RPVOT will follow a straight decreasing line enabling an accurate forecast of the time for an oil change. **RPVOT is a bitch test.**

Otherwise you may have had some amine anti-oxidant finding their way in the system, even ammonia bubbling could increase the RPVOT if you operate a circulation system for a large ammonia compressor in chemical plant.

Try alternative testing for example RULER to map your anti-oxidant additives package. sludge by ultra centrifuge, Visco, TAN and oxidation will confirm the trend.

Guillaume Kalfon

General Manager Middle-East & Africa at Fluitec

Siemens and others have made written statements as to how RPVOT's value is not so strong any more for oils made with base oils other than Group I. I agree with Jean-Michel above, why not research the use of D-6810/6971 instead (aka RULER)? To his comment, I will just add D-7843 (aka MPC).

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Ghasem Shilati

Technical Manager at Naham Pala Engineering CO.
My experience in practice shows taking bottle samples manually gives results one or two class dirtier than on-line particle counting for the same oil reservoir. It is unavoidable because of oil droplets contact with surrounding or even few dusts existing inside the bottle during sampling. So online particle counting should be always preferred.

Finally I remember particles settling time in Newtonian viscose liquids are proportional to 3rd power of particle diameter which means for micronic objects its a huge time and negligible to affect your sample up to received by oil labs.

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Alexey Muralev

Head of Technical Service Department at Kulan Oil

Top Contributor

Basically paper chromatography is very simple and qualitative method of separation of different phases.
Basically you just put a drop of oil into the center of round fine paper filter and wait for capillary forces to spread it. You can see visually the quantity of soot (center), fuel and water (edges). This method won't give you wear metals or any chemical parameters of the oil, just fast check.
This process is incorporated in following commercial products:
http://www.gwrauto.com/One%20Drop.htm

http://www.deutz.com/file/8a85818a244834f601249b491d9201ef.en.0/oil_performance_check.pdf

HPLC and gas chromatography are definitely more complex and precise methods, but they are very expensive and are used for base oil or chemicals, even more for crude oil. As they can show fractional and specific organic compounds content.

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lab must be selected that can perform the following tests for in-service **transformer oils:** interfacial tension (ASTM D971), acid number (ASTM D664), dielectric breakdown voltage (ASTM D877 or D1816), Karl Fischer water (ASTM D1533), oxidation inhibitor (ASTM D4768 or D2668) and dissolved gas analysis (ASTM D3612).

In addition, the following tests should be conducted upon receipt: liquid power factor (ASTM D924), specific resistance (ASTM D1169), corrosive sulfur (ASTM D1275), gassing tendency (ASTM D2300), oxidation stability (ASTM D2440) and particle count (ASTM D6786).

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Manufactured lubricant

Hussam Adeni Business Head and Lubricant Specialist at P N Trading

((For used oil analysis, I would rate Ferrography way above/over ICP, as Ferrography adds another dimension, that of colour photography and also some indicative structure of probable wear.

One distinct advantage of Induction Coupled Plasma analysis is that it can detect below 10 micron sized elements, and can be used both for fresh, in-production and used oils.While Ferrography highlights larger particles, above 10 mu particles in used oil analysis, but more important, it also captures "shape/geometry of wear particle to ascertain type of wear taking place, as well as detect onset of rust and corrosion. The use of color imaging is a distinct benefit and which can be shared with maintenance team members for mutual benefits; as against sharing of ICP derived elemental "data." which is a series or increasing or decreasing numbers.))

I would particularly like to use ICP in a Lube blending operations, where after a class or family of products are made and tank flushed to cater to a new family of products. Typically when a producing ZZ (Zero Zinc) range, or the Railroad oils. ICP would also be a great QA/QC tool for checking "carryover" of additives lodged in the crevices and pipeline.Another area of interest, for using ICP would be to capture additive treats on all batches for analysis, or when changing an additive supplier, as well as for competitors oil analysis.

Since I am not familiar with the tribological tests, you plan to conduct, I cannot comment.

Particle Counters - Industrial Lubricants Which one do YOU like, and why?

Robert Bowden, CLS RWB Consulting LLC, CEO

There are many particle counters available for determining hard particle contamination levels of industrial lubricants. I would like to hear/read opinions, pros and cons, about some of them. I have worked with most of them but not all. I'm interested in experiences, not advertisments.

> Bob Smith
>
> Managing Director of Clean Oil Services Pty Ltd and Lubemaster Australia Pty Ltd
>
> Hi Rob, I use many different particle counters all of the time and have done for many years on a very wide range of equipment and none of them can give me reliable and repeatable information in real time. If you want accuracy and repeatability, use a microscope and a good quality image analysis program - what you see is what you get - an experienced operator will recognise ISO4406 numbers through the microscope and remember, one change in the number of any category represents a 100% change in the number of particles; so if the count is variable from 16 to 17 in the first category, there is twice as many particles in the second sample - too much variation for a lot of my work. This is a very interesting topic - I look forward to seeing further comments. Cheers, Bob Smith (Bob@cleanoilservices.com)

Doug Harris

Tactical Marketing Manager at Pall Corporation

Hi Robert,

Very interested to see how this conversation develops - thanks for putting it out there! I'm professionally biased towards one particular technology, but will try to keep my thoughts objective.

Light obscuration counters (laser or LED based, online or bottle samplers) can give quick results, and the highly numeric output can look very scientific - especially in the case of multichannel counters. But to get best results, the oil needs to be free of entrained gas bubbles, water droplets (both of which will be counted as particles) and fine silts (which can overload the finer size ranges) which in practice can be very difficult. In addition, heavily coloured oils and water/glycols may cause significant light beam absorption, reducing sensitivity or preventing analysis completely. Price wise there's a big spread from low cost workaday models, outputting limited size ranges targeting high turnover oil sampling, to quite expensive research level machines with channels ranging from 0.3µm upwards to 200µm+ - if you really really need to know size distribution rather than cleanliness level, the latter are what you need (or a microscope and well trained technician, see below). The technologically heavier end of the spectrum may also need a lot of operator training to get best results, and careful calibration. Check carefully on optical cell pressure limitations, particularly on the 'research' models as many are designed for off-line sampling and only rated to 10 bar, and be careful to ensure good flushing between samples and especially if changing fluid type.

Mesh blockage devices are generally quick & easy to deploy, unaffected by air, water or oil colouration and much less affected by silts, if at all. The technology has been around long enough to be industry accepted, and operators do not need very detailed training to get reliable results. Note that these devices offer only cleanliness code outputs, accurate to +/- half a cleanliness code, and cannot give you numbers of particles or true size distribution. Online & offline devices are available, and will still need flushing if changing fluid types.

Microscope + technician is still considered the ultimate answer, but takes time - an hour or more to prepare, analyse & report, vs a few minutes for the automated devices - and the technician needs detailed training, good equipment & plenty of experience to produce reliable results. I've seen sites using low magnification viewers and 25 year old reference slides to sign off equipment flushing to cleanliness class 6...

You might also want to get input from my former colleagues Mike Day & Martin Wilkinson.

Robert Bowden, CLS

RWB Consulting LLC, CEO

Thank you. While I am very familiar with particle counting and counters, nobody has "all" the information. Your addition to this conversation is exactly the feedback I was looking for and is much appreciated. Thanks again.

Evan Zabawski, CLS

Reliability Specialist

I have had very positive experience with syringe-driven HIAC/ROYCO particle counters in a laboratory setting, and with CO2-driven PODS in the field.

My laboratory experience with the CO2-driven CLIMET was fraught with calibration issues, repeatability errors and an ultimate decision that dice could give better numbers.

In the field I frequently use a microscope and count manually, but find the greatest value comes from being able to identify the particles.

Luca Angeli

Technical Sales Manager - Hydraulic Filtration Specialist & Oil Monitoring Analyst

Hi Robert!
I use Parker ICount OS and Parker ICount BS, both struments are very easy to use.
ICount BS is suitable to be used in the laboratory while the ICount OS is suitable for making analysis directly on the customers's industrial plants.
Very important: both the strument can be connected in line with pressure up to 400 bar!

Don McNeil

Industrial Sales - STLE Certified Lubrication Specialist at Apache Oil Company

My experience has been similar to that reported. The use of field instruments produced widely variable results and the only reliable method seems to be using a microscope and an experienced lab technician to analyze the sample. Down side is that it takes time to produce accurate results.

Ricardo Hein

President at Conexo Inc.

We work with OILPAS that reports particles, bubbles and water droplets, and oil turbidity, all separate from each other, so we do not count air or water as particles. All of this is done online, and at a rate of 1 analysis per second with a resolution time of 1 second to the monitor for real-time measurement. A very specialized instrument for understanding processes that might be developing in the fluid and with very precise results. There is a method for measuring aeration in the oil with this instrument. Here is a paper at the SAE with how it works: http://papers.sae.org/2010-01-1528/

Conor Jaunsen

Business Manager at Fluid Analysis Technologies

I've run particle counts for Chevron, Mobil, John Deere, etc. In my experience, a variability in results is caused by improper sample prep and/or sample dilution, particularly for industrial oils which tend to have thicker viscosities (producing a higher percentage of entrained air bubbles) as well as higher allowed water values (same effect on automatic particle counting). We used laser particles for hydraulics with water less than 400ppm and at most, 1:1 dilutions with Conostan's SCF (super-clean fluid). We now use a Spectro LNF for engine and gear oils for both particle count and wear identification, zeroed with VHG's NIST traceable filtered kerosene.
Also, using a laser particle counter on industrial oils reduces the life of the laser. Higher RMS noise approaches the 4-micron voltage field and the only solution is a new laser. Manual counting just introduces more human error.

- Richard Hassebrock

Field Engineer at Castrol Heavy Duty Lubricants

This discussion illustrates a dirty little secret in the oil analysis industry, the poor accuracy of automatic particle counters and how easily the results can be skewed by improper sampling and handling practices. The problem is that it seems every periodical in the oil analysis and equipment maintenance business has published articles about the importance of fluid cleanliness, and promoted ISO 4406 as the standard; training sources like Noria and others teach students about the same, and in the heavy off road equipment industry manufacturers like Caterpillar have established cleanliness limits for new and in use lubricants; which is all well and good, but they've all failed to stress that in order to achieve accurate and consistent results requires exceptionally clean sampling and handling practices, and even then the results from an APC in the lab are far from ideal.

This has resulted in a lot of confusion in the field among well intentioned but poorly trained workers who believe the results of their poorly drawn samples with dirty sampling equipment and containers and a lab with substandard practices are gospel. This is especially true in the off-road world if the lab happens to be a Caterpillar lab, because Cat customers tend to take every word from Cat as if it was from The Almighty himself.

I appreciate the increased awareness of the importance of fluid cleanliness and the trend towards improved conditions in the field and the concerns being shown by users, these are all good things; but the industry has done itself no favors by failing to educate about the limits of this technology, and the requirements that users upgrade their sampling/handling practices to world class levels if they expect to receive world class results. Oil analysis labs also need to improve their practices, and even then the limits of the technology leave particle counting best used as a trending tool, to be used to help improve conditions, not an absolute measurement.

- Bijendra Rawat

  adviser at Assam Company Ltd & Pandit Deen Dayal Petroleum University Gugarat, India

  I agree with richard ....using improper and / or non standard way of drawing samples particulalrly from the field service and that too deploying an unskilled / untrained personnel and then talking of nano level / micro level wear particle measurement in in-use oil would be a joke ? there has to be a fool-proof and scientifically proven method prescribed by the manufacturer besides providing an easily approachable sampling point in the machine / or engine to draw sample under dynamic running condition ......only then we can discuss and interpretate scientifically........

  Mark Latunski

  Lab Manager at American Chemical Technologies, Inc.

  In evaluating many technologies for group V synthetic lubricants, it is the Coulter method and the Multisizer that shows the best performance.

  The technology is ideal for PAG's, synthetic esters, phosphate esters, and water glycol fluids. If I were specializing on Group I-IV lubricants, laser technology is sufficient. But where premium lubricants are concerned, the coulter method is much better for predicting fluid contamination.

  Guillaume Kalfon

General Manager Middle-East & Africa at Fluitec

Not an expert in particle count, I'll just share a key thing I'm seeing. One difference between optical and pore block is that optical may count as particles things that are in fact soft, such as water or soft contaminants, i.e. the degradation products of the base oil and/or the additives. If you have elevated first class of ISO 4406 and high D-7843 (MPC), then that may be the case. Comparing optical with pore block will then give some confirmation (optical giving higher count). Another way is to re-run ISO 4406 after heating the oil (e.g. 1 hr at 80C): if the particle count drops, then you have soft contaminants that have re-dissolved, and no longer show on the optical sensor.

Perry Thiessen

C.C.JENSEN Offline Oil Filtration Systems (Canadian distributor)

Hi Malcolm, I think the ISO 4406 particle count is an ok reference point. Of course if you have particles larger than 14 micron then this is of serious concern. As others have mentioned usually a clean particle count such as 15/13/11 is indicative of a system clean of larger particles.

Something we have seen in hydraulic press systems is varnishing of the oil because the systems run very hot. We supply offline filter systems that continually filter down to 3 micron absolute and also remove varnish from the system.

In my opinion the ISO 4406 particle count will fall out of mainstream use in the future (at least I hope so). As we learn more about oil contamination and equipment failures it is apparent that small particles are the most damaging. Going forward I believe the focus should be on cleaning oil from particles 1-5 micron in size. In addition cleaning submicron particles and soft contaminants is also important (varnish). I take it for granted that any critical oil system should be free of any particles greater than 15 micron (if this is not the case then a solution should be implemented).

This is a case study we just completed here in Canada on a hydraulic press system in a pulp and paper mill.

https://docs.google.com/presentation/d/1Vo3IC0_JFwDR9AkYjpMO5mi_WwA2yYAW8QNUKnaVHro/edit?usp=sharing

==

toby taylor • Gentlemen, the **FZG** rig needs higher loads to evaluate adhesive wear/ fatigue mechanisms . The whole slide roll wear mechanism is what it is with grossly modified dedendum profile with fog. the 4 ball simply measures the sliding hertz load until wear scar until adhesion wear begins. Both tests are extreme but merely give guidance as to a EP additives performance. To simulate real gear mechanisms at a particular point within the slide roll wear cycle you need to test on a disc machine. Ideal for evaluating all the wear mechanisms that takes place ie adhesion and fatigue both micro and macro.

fzg rig a four square rig with grossly modified profile to create high sliding to accelerate the propensity to cause slugging. Job done. But the surface finish was of mash grinding and that produced micro pitting at the lower load stages whoopee another test condition to examine.additives developed to beat load stage 12. Correlation please this test is too expensive. The 4 ball evaluates scuffing its cheap what if test....nothing like simulating real dynamic conditions .. Bring back a simple disc machine david brown gear company still have the original design.Fzg and 4ball both are simple. Wear tests and that's all they are.there is nothing like testing the real dynamic of your sliding or rolling condition.a seasoned lubricants tester and developer.

By toby taylor

Correlation of FZG and Four-ball test

> Greetings Jorge, Mitjan and everyone,
>
> Yes, this is a good debate but I think it is worth reminding ourselves of where it started. Let’s revisit Petr Vavruch’s original comment with regard to correlation between the four ball test and the FZG test: “The short answer is no. If there was a correlation, nobody would bother to run FZG”.
>
> There are two elements to this comment that perhaps need teasing out a bit:

> There is no correlation between a four ball test and an FZG test:
>
> Agree. But there is also no correlation between a four ball test and an engine test etc, so that is no surprise. But that does not mean that there is no correlation between other types of tribological test and an FZG test.
>
> If there was a correlation, nobody would bother to run FZG:
>
> Disagree. The whole reason for running cheaper and simpler tests is to screen candidates before running expensive tests such as FZG, a full engine test or, indeed, a field trial. That is the whole purpose of bench tests; not to replace the full-scale tests but to reduce their number and increase the through-put of candidate samples. There is another purpose, of course, and that is to make the wear and failure mechanisms more accessible and controllable, so as better to understand what is going on in the real process. For example, it's a lot easier to measure friction in some kind of bench test than it is to measure friction at some point in an engine. It is a lot easier to control critical processes in a bench test than it is in an engine or gear test. Etc.
>
> So it is worth trying to develop meaningful bench tests and, with sensible design, this is feasible. This is something recognised by Vern, Jorge and Mitjan. So what is sensible and what will work? That is the question!
>
> I think the right starting point is to decide what definitely will not work; I am not, for example, convinced that any sliding point contact test is a model for anything in the real world, and that is not just the sliding four ball test. Sliding point contact tests have their uses, mostly for EP testing, but they also have significant limitations. For example, in a reciprocating test, the beneficial effects of some friction modifiers are not obvious with point contact specimens, but are present at lower contact pressures, using line contact specimens.
>
> For a reciprocating test my preference would be to use line as opposed to point contacts, resulting in:
>
> 1. Much lower and hence more realistic contact pressures.
> 2. Sampling of more of the specimen surface.
> 3. Avoidance of plastic deformation and work hardening of the counter-face.
>
> For sliding/rolling tests, then once again, my preference would be for a two roller test or an energy pulse slide/roll adapter with a line contact.
>
> To be of any use the test must model the friction, wear and/or failure mechanism apparent in the real application, in this case the FZG test. The test variables must be selected and adjusted in order to obtain correlation. If the wear/failure generated in the bench test looks nothing like the wear/failure in the real system, the model is likely to be wrong. Hence, before we start, we need to be able to analyse what is going on in the real application and then to see if there is a sensible way to model this in our bench test.
>
> Good understanding of real process to be modelled followed by sensible bench test! Sounds easy, doesn't it!!!!

By George Plint

Petr Vavruch • We have to run modified FZG because the standard test is no good for modern EP gear oils, they all pass stage 12. One has to run stage 11 for 30 hours to select the really good formulations.

Charles du Bois • Pleebe advised that there is actually no correlation between FZG and 4 -ball. Bothe tests are completely different. If you would like a answer, ask Dr Michaelis in Munich at FZG as he is the inventor/ devoloper of the test. We should stop guessing, ask the expert.

Mitjan Kalin

Head, Laboratory for Tribology and Interface Nanotechnology (TINT) at University of Ljubljana

I'm following you and I did not intend to comment as I will probably repeat previous comments a lot, but I would like to point out few things also from the additives' point of view (I think this was the interest of Zheng Wang from Shell in the initial question).

Since in our group we use almost all above mentioned tests, including FZG (quite extensively), I can say that it is hardly possible to get quite the same contact conditions (with many parameters defining "conditions") - on the absolute values scale - at different tests, which was nicely explained before. In this respect, the additives will probably not perform exactly the same in different tests, because:

We should note that additives have specific chemistries and so the mechanisms and tribochemistry with activation barriers, kinetics, film property, etc. Since we all typically notice some performance differences between even slightly different additives at the same test conditions or with the same additive under even slightly different test conditions (in "exactly" the same tests), this suggests that they are indeed sensitive to contact "conditions" and chemistry variations and therefore, all these things matters - this is the key, right?

Therefore, since it is not really probable that exactly the same contact conditions will be achieved in FZG and four-ball or other tests, different effect of additives should be expected.

However, if the additives of interest are "different" enough (i.e. perform differently), then the contact conditions on two devices may indeed discriminate them in the same way (if good contact conditions match is obtained) - and so the result on two devices will be the "same" or "similar". On the other hand, if the additives react in a very "similar" way, then their performance in different tests will probably be "different" because the differences in contact conditions may be already so high that they trigger their activity in a different way. And quite small variations can put the ranking and tribochemical activity in very different perspective.

But how different is "different" is also relative and depends on what should be deduced from specific test, as well. Is it the same wear mechanism, the ranking among different samples/additives, the "exact" tribochemistry, film thickness, its chemistry & properties, etc. ?

Thus, well defined conditions in the same test type can compare very well the behaviour of additives and describe when and how they operate, but comparing directly the results from different test devices - it can work only if the above contact similarities are satisfied, which is hard to predict or satisfy, and the answer depends also on the magnitude of differences that we look for.

Best wishes,
Mitjan

The fundamental point is that friction and wear are system responses (Czichos), so the behaviour of a lubricant or tribological pair depends on the system in which you place them. You can alter the performance of any test system by, for example, changing the stiffness or inertia of the loading system, the thermal mass of the test adapter etc. So nominally identical systems (for example, nominally identical pin on disc machines) can produce wide variations in friction and wear coefficient, when running with nominally identical test samples and test conditions. Now put the material pairs in different test systems and you will, inevitably, get a different system response.

If you want to model a real tribological contact, you first need to understand the wear and failure mechanisms you are attempting to model, then try to reproduce those mechanisms in a laboratory scale test. The problem with tests like the four ball is that it was not designed for this purpose. It was invented long before the advent of the term tribology or (somewhat earlier than Jost) tribo-chemistry and tribo-physics. Here are some dates worth noting:

1929: Pin on Vee Block Test
1933: Shell Four Ball Test
1935: Timken Block on Ring Test
1937: IMechE - General Discussion on Lubrication and Wear
1937: Blok - "Flash Temperature"
1946: Bowden and Tabor - "Tribophysics"
1953: Archard - "Wear Law"
1966: Jost Report - "Tribology"

The four ball and Timken tests were basically invented as scuffing tests, to demonstrate the presence of EP additives in a lubricant. This makes sense; the tests use the same material on either side of the contact (52100 bearing steel), so the specimen pairs are by definition mutually soluble and will thus have a tendency to scuff: they have poor tribological compatibility (Rabinowicz). This is thus a good set up if you want to demonstrate the presence of an anti-scuffing additive, but it’s not much use as a model for any real engineering contact, for a number of simple reasons:

1. There are no real engineering contacts that involve a sliding hertzian point contact, so the four ball configuration does not model anything in the real world, other than a four ball test. This point is recognised in the bias statement in many ASTM standards, which typically follow the form: “The evaluation of “Property X” by this test method has no bias because “Property X” can be defined only in terms of the test method.” In other words, it does not correlate with anything else. This is an inconvenient truth, so we frequently feel tempted to ignore the bias statement!
2. There are few, if any, continuous sliding line contacts in the world, fewer still (if any) involving bearing steel against bearing steel. Engineers would choose different materials, if such a contact was unavoidable, to improve tribological compatibility and avoid the risk of scuffing failure.
3. There are few if any sliding area contacts involving bearing steel running on bearing steel etc.

Finally, and then I’ll shut up, how anyone can think that any kind of sliding test involving bearing steel (a steel ball, for example) sliding against another material can model anything in an engine is beyond me. Exactly how much bearing steel do we find in an engine?

To conclude, very simple screening tests have their purpose, but can rarely be used as models of real systems. Things usually go wrong when we try to use simple tests to model complex systems. Because tribological properties are system responses, we need different test systems and test parameters to generate these different responses.

By George Plint

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James McAllister

For real time analysis of **moisture** during vacuum dehydration we use a Vaisala moisture meter that measures aw (water activity) as measuring ppm or saturation can be tricky unless you program in propoerties for each different oil you encounter. They have a good article here that explains the process-
http://www.vaisala.com/Vaisala%20Documents/Application%20notes/OilMoistureExpressedasWaterActivity_B210806EN-A.pdf
We also use a n LPA2 desktop instrument measuring Relative Humidity and % sat.
Link to the LPA2 is here http://www.mpfiltri.com/media/wysiwyg/pdf_products/2601.pdf

KF is great if you have a lab nearby but we don't often have that luxury in field applications.

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Viscosity test - Liz Considine

According to ASTM D445 the following rules apply:

Repeatability: Duplicate results with the same operator using the same or different viscometers should not be considered suspect unless the deviation is greater than 0.35% of the mean.
Reproducibility: Samples run in different labs, different viscometers and different operators should not deviate more than 0.7% of the mean.

On-site oil analysis

Richard Hassebrock • Which basic tests? Good question. The most basic test is Viscosity, often called the most important characteristic of a lubricant. A simple Visgage is inexpensive allows quick checks of viscosity in the field (note that this is a "comparative" analysis, not absolute). Just do a google search for more information on that. Or you could spend more for something more sophisticated, just google "portable viscosity meter" and you find several options. After Viscosity you probably want to know the condition of the oil, and there are several different ways to go with that, depending on how sophisticated you want to get with it. The simplest way to go is with using a blotter test, which is simply placing a drop of oil onto chromatography paper and letting it absorb, then reading the results; to field test kits for TBN & TAN, to portable FTIR machines such as those available from Spectro (Fluidscan Q1100); and then there is the RULER device which measures anti oxidant additive depletion. And finally you'll want to measure for contaminants, either particulates or water contamination. For water you can simply use an electric hot plate heated up to at least 212 degrees and place a sample of the oil onto a flat plate and if there is water present it will boil and be evident (that's the Crackle Test). For particulate contamination you can use a filter patch test, where a sample of the oil is drawn across a fine filter patch, then the patch is viewed under a microscope. And finally a simple test that really should be employed regularly by everyone with a maintenance operation is filter examination. Cut open the filter and examine it for visible particles, which can give you advance warning of impending failure. I have customers that this is part of their regular maintenance practice, at each filter change interval the filters are cut open and inspected before the machine is returned to service. It only takes a few minutes and more than once has paid off in finding some kind of damage that was starting to occur.

None of these tests take the place of a full lab analysis of the oil, but they can give you immediate information that can be helpful to make decisions on maintenance/repair actions. I.e. if the oil fails the crackle test, you don't need to wait for the sample results to come back from the lab, you can start addressing the problem immediately. But you still want the actual lab results to be added to you used oil analysis database, and to confirm what your field analysis told you.

ROBERT SMITH • I appreciate all information. I would like to continue using an off site
lab but will look at the best provider available.
In relation to oil analysis say we used the Fluid Scan Q1100 and found an issue with the oil and needed to be changed immediately. Before changing oil we would obtain a sample for full analysis at the offsite lab.
I have beenasked about carrying out full analysis on site but to get the dedicated people plus the investment I believe it is not the way to go.

Sandy Mitchell • I would agree with Andrew. A Parker/Kittewake Filed Test Kit offers a reasonable indication of oil condition at a reasonable price.
http://www.kittiwake.com/DIGI-Field-Test-Kit
Combine this with a particle counter to check on ISO or NAS codes
http://www.parker.com/portal/site/PARKER/menuitem.7100150cebe5bbc2d6806710237ad1ca/?vgnextoid=f5c9b5bbec622110VgnVCM10000032a71dacRCRD&vgnextfmt=EN&vgnextdiv=&vgnextcatid=7456515&vgnextcat=BOTTLE+SAMPLING&Wtky=Monitoring

http://www.filtertechnik.co.uk/resources/fs9001-portable-fuel-cleanliness-analyser#node-342
and this will give you a good idea of whether the oil needs to be changed

(Alternatively, see my comments about oil changes)

Tim Roadnight • Robert, Some oil majors have had success in implementing converted shipping containers into basic on-site labs. Some of the equipment is Perkin Elmer, some Kittiwake / Parker, but more importantly you could utilise the OSA equipment, 'On SIte Analyzers' based out of Florida.

As Sandy, Richard and Andrew have mentioned, on site analysis should only be your first line of defense, an external lab with professionally trained lab technicians and tribologists, equipped with calibrated state of the art equipment can never be replaced.

In addition, having looked at the CAPEX for on site labs for a number of clients (in my past role), the cost of set up, implementation and running costs (including training two staff on each shift) soon mounts up. Unless you are undertaking >6K samples/year I son't see any cost benefit.

Michael Dines • Thank you to all for the recommendations for the Parker Kittiwake solutions. We always say that in normal circumstances on-site should not be used to replace the laboratory testing and the two compliment each other. For me, on-site testing is an ideal tool for identifying issues occurring between scheduled laboratory tests, for getting immediate feedback on trouble-shooting and for double checking a laboratory warning/alarm before taking action (in case the sample was incorrectly taken or mislabeled). Some people also use it to extend the monitoring program to equipment where they feel regular, full laboratory analysis isn't justified.

It can also increase "ownership" in a program by workers in the field. A laboratory report can seem a little detracted from what they are doing day to day. By doing the simple tests themselves, they develop a trust in oil analysis, which then increases trust and understanding of laboratory reports.

For the particle count; this is a great, simple option
http://www.parkerhfde.com/pdf/conmon/fdcbB528uk.IOS.English.pdf

The IcountBSplus (link from Sandy) is a very advanced product and is a full particle counter (providing lots of information). If something simpler, just providing ISO codes or NAS class is needed my link above shows a portable particle detector.

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Joel Teixeira Cunha   Sócio na Fundamental Manutenção Sustentável Ltda.

In my opinion you have mixed two different type of oils and the first step is to check if top up with the same oil. My teory is that the results of new and used oil are not coerent. And according with the problem related maybe you mix two incompatible type of oils and this could be a problem too. Lets go to the oil chemistry. First of all FTIR is not a good technic to do TAN, because the amount of ester will contribute with the "increase" of TAN results because the presence of C=O in the esters are the same in organic acids and show absorbance in the same region, for exemplo, is commom to find high values of TAN by FTIR in oil with high amount of VI improvers or Fat Acidsand they are not acidic compounds. Second, poliol is a alchoolyc compound and will show absorbance in the same region of water and the only way to quantify water in this oil is by Karl Fischer or Destillation.

For oils in service: The neutralization number NZ (or acid number AN) is one of the important tests in oil condition monitoring. But it should never done or interpreted isolated. Otherwise it can lead to wrong diagnosis.
Here two examples:
The NZ/AN increases mainly by oxidation or in combustion engines combustion by-products. But often it decreases by additive depletion. If the lubricant is mixed with another one, it can decrease or increase the NZ/AN too. You should

perform evereytime a element analysis, FT-IR and viscosity too. In gas compressors you should degass the oil before measurement to avoid influences of the compressed gas, e.g. CO2..
In synthetic base stocks the NZ/AN reacts often very different. Some esters show a very significant increase, some not. PAO based hydraulic fluids or gear oils show often nearly no increase of AN and/or viscosity, but tend to form deposits. Therefore: Put all parts of oil analysis together to get a real picture of the individual oil ageing process of the application.

By Rüdiger Krethe

http://www.astm.org/Standards/E2412.htm

Evan Zabawski, CLS • FTIR is frequently employed to measure USED engine oil samples for:
Oxidation - ASTM D7414
Nitration - ASTM D7624
Sulfation - ASTM D7415
Soot - ASTM D7844

Sometimes:
Anti-wear - ASTM D7412

It can also detect water, glycol and fuel contamination, and be calibrated to correlate with other testing such as Base Number.

There is not likely to be much reason for the manufacturer of new oils to employ FTIR and measure any of the above parameters.

Josef Barreto-Pohlen • I agree with the previous comments and I want to add something:
FTIR is THE analytical method for a lubricant laboratory in QC and R&D.
First off all it is a method to identify substances, being it raw materials, intermediates, finished or used products.
During development and production it can be very helpful, e.g. producing poly urea grease, because one can check the content of free isocyanate and take corrective actions.

Hussam Adeni • Details of FTIR pertaining to Oils analysis is available on Slide No 29.
http://www.slideshare.net/hussam57/testing-of-lubes-and-its-significance-nov-2011

Josef Barreto-Pohlen • I am not familiar with the ASTM-Tests Evan mentioned. As long as the lubricant doesn't contain any esters it is possible to check the peaks at 1705-1720 cm-1 (carboxyl-group of organic acids). Ketones is also possible around 1710-1720 cm-1. The appearance of these peaks in the used lubricant indicates oxidation. In the presence of esters it will be difficult because they appear around 1735 - 1750 (additional doubble peaks between 1000 -1300). In this case it is useful to look for antioxidants/additives in genral in your spectra. You can also see if you have water or solids (soot, shift of the base-line) in the lubricant.
Important: Always compare with the IR-spectra of the fresh lubricant.

Evan Zabawski, CLS • Farhan,

I re-read your comment and realized you were asking a question, and the answer is that yes, some FTIR software packages will do spectra comparisons and tell you how closely an unknown spectra matches those in your database.

As to whether or not you can definitively say that the unknown spectra is a certain oil is largely dependent on what spectra you have in your database, the larger and more representative it is, the greater confidence you will have in your answer.

Siddiqa Mahaaf • I agree with Evan.

FTIRs are commonly used for identification, qualification, comparison and reproducibility of materials in quality control and production applications. FTIR techniques include fingerprinting, and the qualitative and quantitative analysis of unknown mixtures.
http://www.chemguide.co.uk/analysis/ir/fingerprint.html
HTH

TEST METHOD for TBN that utilizes some kind of TABLETS

Daniel Ríos • Look for kittywake, they have field test equipment. Based in the UK, see if they have distributors in your region. For TBN they have a reaction capsule which uses some reagents and the capsule has a pressure gage where you take the reading and calculate the BN, based on th ereading for a new oil sample.

George Abernathy • I agree with Daniel. In the field we used Hach(brand name)capsules that one cuts the end off, adds/mixes then titrates(there is a chart) with either drops or a biurette with an auto-refill system, and a portable mixer and if necessary-a pH meter. Contact Hach.

Evan Zabawski, CLS • Dexsil also makes a kit version: http://www.dexsil.com/products/detail.php?product_id=38
I have used the Hach one and found it to be superior to Dexsil's in terms of accuracy and comparability to off-site lab results, but the Dexsil kit offers simplicity that is preferred by some users.

Nancy Barrantes • For analysis of TBN Titralube Dexsil field exists, astm method d5984, very accurate and easy to use.

Jorge Villafuerte • Dear friends, I recommend using these fast test kit only for trend monitoring. If you need accuracy use D 4739 (USED OIL) or D 2896 (NEW OIL AND USE OIL).

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Larry Ludwig, CLS, OMA, CMFS • An engine oil's **volatility** characteristics are directly related to the type of base oil and the particular base oil viscosity cut used in the formulation of the engine oil. The volatility characteristics of an engine oil as measured by the ASTM D-5800 Method have been found to correlate with oil consumption in both passenger cars and heavy-duty diesel engines.
In this test, a measured quantity of sample is placed in an evaporation crucible or reaction flask that is then heated to 250° C with a constant flow of air drawn through it for 60 minutes. The loss in mass of the oil is determined. The test meth-od covers three procedures for determining evaporation loss. Procedures A and B use an electrically heated block to heat a steel crucible where Procedure C heats the glass reaction flask with an electric heating element. A vacuum pump is used to maintain a constant flow of air across the surface of the sample. To meet API CJ-4; ACEA E-7 and ACEA E-9 requirements an engine oil cannot exhibit greater that a 13% evaporative loss. To meet API SN Resource Conserving and ILSAC GF-5 and engine oil cannot exhibit greater than 15% evaporative loss.

Metal Particles

Zainudin Yahya   Managing Director at CbM Solutions Sdn. Bhd.

Iron in ppm in theory should cover ionic (oxides) and magnetic Iron, PQ Index which rely on magnetic properties of Iron particles in the sample will only show magnetic Iron.

Depending on what technology being used typically spectrographic (XRF, ICP, RDE etc) Fe in ppm will be measured fairly "completely" for Ferrous particles of 5 microns and smaller, but as it get bigger some instrument may not be able to present the bigger ferrous particles (typically 10 micron and bigger) to be "excited" hence limits the detection of large ferrous particle.

This limitation is complemented by PQ instrument, which the very reason it was designed .... primarily to detect large ferrous particles. Good lab will typically have both techniques.

To understand this in greater depth you may want to visit: http://cbmsolutions.com.my/?p=960

Lubricant Specialists

What is the best way to ***test the presence and amount of metal particles of a lube oil*** *in the laboratory?*

Alexey Muralev • That depends on size distribution of your particles.
Here is link to Machinery Lubrication (http://www.machinerylubrication.com/Read/1308/large-particles-gear-oil)
Basically it is:
Emission Spectroscopy (< 8 mkm)
Particle Count (5 - 100 mkm)
Analytical Ferrography and Patch test (> 100 mkm)

Panagiotis Moraitis • If you need high accuracy definitely ICP.

Alessandro Paccagnini, CLS / MLT-I • As stated by Alexey, it depends on the size of the particles.
Using AES, be it ICP or RDE, you will detect metals only up to 5 microns (8 microns in the case of RDE), but you will have an invaluable trending tool, which is more or less unaffected by sampling errors (in my experience).
Particle counting and particle imaging (analytical ferrography and membrane patch microscopy) are a lot influenced by the sampling technique. Particle counting from 4 to 100 microns, particle imaging (including ferrography) from 10 to 500 microns.
But particle counting alone will tell you nothing about particle metallurgy or composition.
Another successful technique is PQ, which detects only magnetic particles in the mid-high size range.
At the end, metallic particle monitoring in used oil should always be performed using a multi-technique approach, with AES being the first and essential test.

Robert Bowden, CLS • Fast, easy, cheap and FIRST, use a magnet to see if magnetic metals are even present.
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What is your preference? WDX is way too expensive so can not be compared to ICP or EDX

Dennis Hoogendonk

Instrument sales and support at OmniTek

Being distributor for RDE, we, of course, prefer RDE ! But it is not straight forward. First you have to find out : (1) what elements are present, (2) what shape do they have and (3) can I find standards that match with my fluids.

(1) A lubricant will consist of 'dissolved' metal soaps based on Zn, Mg, Ca . They may have concentrations up to 2000 ppm. Then you will find undissolved particles like Fe, Cu, Al, Ni, Cr having presence up to 100 ppm.
(2) Size of wear metals may be from 0.1 - 100 microns.
(3) There are a few standards available up to 24/32 elements. But all based on 'dissolved / dispersed' metal soaps.

Now comes the tricky part.
With WD-XRF or ED-XRF you can run a sample straight-away from the sample cup. In a few

minutes it is analyzed. With ICP you have to create a nebula, so you have to dilute it with e.g. diesel or kerosene. With RDE you can fill a 3ml sample cup and run it staright away and qithin a minute you have a result.

ICP is very sensitive, but due to dilution you loose some of it. But main drawback it that wear particle > 4 microns will not be taken into account. They either lie down on the bottom of your sample tube or will not be fully atomized in the nebula.

WD-XRF (ED-XRF) require a couple of minutes for analysis. In between all large particles have settled to bottom. So these will influence measurement; most often you will analyze too much. Also XRF has some element interactions which need to be mathematically compensated. But if you know fluid composition it may work well for non-wear metal containing fluids.

With RDE you have your result fast with little prepararation. Also, due to rotation of electrode, sample remains homogeneous. So all heavy wear particles are swirled around. Electronic sparc will energize all metal atoms up to 10 microns, so a far better result is obtained. In case of filtered base analysis, even up to 100 microns can be analyzed, but only for trending applications.
Another advantage of RDE is that instrument can be in-service within a few minutes, as XRF can. ICP requires a quilified analyst to have it run.

Drawback of RDE is that it is more difficult to automate.

George Diloyan

Director of Technology at Nanotech Industrial Solutions

Thank you Dennis. How about elements that can be analyzed in Rotrode? I guess there are 25 (max 32). ICP, EDX or WDX analyzed almost all elemental table (besides radioactive and very light elements). Also seems like precision of ICP (due to high sensitivity) is in order of magnitude higher than rotrode.
I agree with EDX - there could be a lot of overlapping of peaks, however in WDX this problem seems to be eliminated. Another problem with EDX is that same concentration of the same elements in different oils could give totally different results. This can be solved by building a calibration curves, but if you do not know composition and need a run a quick check, you can be easily misled.

The drawback of WDX is the price - over $150k

Dennis Hoogendonk

Instrument sales and support at OmniTek

RDE runs 24 elements standard and can be expanded to 32. More elements are possible in theory, but as RDE is originally developed for fuel analysis or fresh / used oil analysis not many standards are available to cover entire gamma. Advantage of RDE and ICP is ability of analyzing Li.

About WDX the next story. I was yesterday with a customewr for installation of an LNF Q200. I was told that their WD-XRF has annual costs of 15-20.000 euro to keep it up and running. Modern ED-XRF can do the same as some older WD-XRF for less than half the price.

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Standardized tests for oxidation, metal corrosion, seal attack, viscosity, and acidity give reasonably repeatable and meaningful measurements of lubricant properties. On the other hand, the usefulness of bench testing to assess wear resistance, friction, scuffing, and rolling contact fatigue is controversial. Therefore, it is incumbent on tribologists to find tests for friction and wear that correlate best with engine operating experience.

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Typical sources of copper within an engine include the wrist-pin bushings, thrust washers, oil pump, governor, valve-train bushings, cam bushings, oil cooler and bearings. Copper can also be an anti-wear additive in some oils. Additionally, high copper readings may occur during the run-in of a new engine (after 500 to 1,000 service hours) and when changing from one oil brand to another.

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PORTABLE OIL PARTICLE COUNT KIT

Hopefully someone in the group can help me .We are considering to procure portable particle count kit for analysing particle contamination and water contents % in oils being used in our Gas turbine (Synthetic oil) , Generator & Gas compressors (Mineral oil) .Have come across a portable kit i,e Contamination Control system (CCS 4) manufactured by Internormen Technology GmbH .Does any one has experience in using CCS 4 ? .Is it user friendly ? .
Request comments ,advice & suggestions on any other mobile onsite oil diagnostic kit , which is capable to measure water saturation and particle contamination per ISO 4406:99, NAS 1638 and SAE AS 4059 standard .
Regards,
Danish

Dennis Hoogendonk • You could also consider Spectro LNF-Q200, also known as LaserNet Fines. It not only counts particles, it has also capabilities to picturize particles larger than 20 mu. Besides, it can determine to some extent water content, if present as bubbles. I give you wear shape details. Unit can measure viscosity as well and has a special kit for analyzing Skydrol (phosphate esters). Portabe but not battery operated. Best of all: calibration is not required.

If you only need particle count, we have a dedicated Filter Patch Scan unit available based on CCD camera technology.

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what is the right equipment **to test a transformer oil**, is it user friendly?

Steffen Bots, CLS / OMA I • Hi Mogopodi, let me add some comments regarding the observation of transformer oils. First of all you should define whether you want to do online (e.g. sensor), onsite (e.g. RULER ASTM D-6971) or offline (laboratory) analysis. Another question is what kind of information you need. Are you focused on the transformer condition, the oil condition or both? If you want to do both you will have to measure a couple of different parameters like breakdown voltage, precise water detection acc. K.F., dielectric strength, surface tension, antioxidants, gas in oil analysis and more. You find an overview about typical test composition under the following link:
http://www.oelcheck.de/fileadmin/oelcheck/pdf_eng/prices_Transformer_oils.pdf
All those tests can be performed very precise within a laboratory, but you need already special material like aluminum bottles or gas tight syringes and apply a proper sample taking technique in order to take a representative oil sample. Some of those test are also available onsite or online. If you want to observe mainly the transformer condition the most important test is the gas in oil analysis. There are a couple of different instrument suppliers on the market for laboratory, onsite and online equipment. But I fully agree with Benoit, that this is high end equipment. Usage and also interpretation of the results require definitely a certain level of knowledge and experience.

Jorge Villafuerte • Dear, power factor ASTM D 924,@25°C and 100°C, Dielectric Breakdown @60HZ ASTM D 877 (delivery), ASTM 1816 (fill tranfsformer), Interfacial Tension ASTM D 971, and water PPM ASTM D 1533.

Praise Afunor Agbi • Praise Afunor Agbi Well transformer oil is not the easiest to test and, depending on what you are looking for, normally requires high end equipment such as Gas Chromatograph. I'm not sure what's your definition of "user friendly", but I would probably reply that no lab instrument is user friendly and requires the proper knowledge and experience to be applied, power factor ASTM D 924,@25°C and 100°C, Dielectric Breakdown @60HZ ASTM D 877 (delivery), ASTM 1816 (fill tranfsformer), Interfacial Tension ASTM D 971, and water PPM ASTM D 1533.following the correct procedures and quality system to ensure consistency of results.

Guillaume Kalfon • Hi Mogopodi. Transformer oils can be either inhibited (containing antioxidants) or uninhibited. In the former, monitoring antioxidant health is a good idea. This can be done by linear sweep voltametry. Adapting ASTM D-6971 (meant for turbine oils) to transformer oils is one way of doing it.

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Richard Widman • I have written extensively on this subject, and if you can handle the Spanish, you can start here: http://widman.biz/Analisis/interpretacion.html

There are several pages where I talk about contamination, degradation, etc, and also sample analysis results for certain cases, selected from about 5000 I have done for my customers.

Among the 115 bulletins I've published, there are also several that explain these problems and how to extend intervals. (and how to implement a sampling program) Those start here: http://widman.biz/boletines/boletines.html

You need to take into consideration that the lab will give you results that are very "cold", or average. They will compare your results with norms (which I explain in the first link). BUT only the person who is familiar with the goals of the end user and the conditions where the equipment works can really tell you what is too much. I disagree with a lot of what is published, as my customers do not want to be average. They want extended intervals with long life spans for equipment. We have successfully extended drain intervals in construction and agricultural equipment to 500 hours that sometimes get stretched to 700. But first you have to get a hold of contamination. You need top tier filters (I use only Donaldson) and top tier oils (group II). You could use synthetics, but that is long after you control contamination.

A good CI-4 group II oil can easily take you to those levels even with 2000 ppm of sulfur in the diesel and give you 25,000 hours of engine life between overhauls.

In general I alert customers when their silicon (dirt) levels pass 7 ppm for the 500 hours and flag them at 10 ppm. I alert when the fuel dilution is at 1% and flag to stop the equipment if it causes the viscosity to fall below 12.5 (with a 15W-40).

Here in the tropics (400 meters above sea level, I alert above 0.5% and flag 1% soot, but equipment operating in the west of the country at 4000 meters has a normal soot of around 1 to 1.5%, getting my alerts above 2%.

In hydraulics the conditions are similar all over, but I strive to get them to use breathers and good filters. I tell the ceramic plants to change their hydraulic oils when I can determine there clay mix from the elements in the oil. Again, filtration is the key, since this can vary between 4000 and 8000 hours in different plants with the same equipment but different maintenance programs. I will also note this is with group II hydraulic fluids. I've had experience where one plant decided to use a cheaper group I product and was not able to get through 400 hours without doubling the viscosity and the oil turning black with oxidation.

V.S.S. Sarma • I Thank Richard Widman for taking time to give his expert advice. In desert conditions, in dusty environments like what we have here in the Gulf countries, Silicon is an issue. Even the fresh oils may show 5 ppm of Silicon and can go upto 25-30 ppm while the oil is in service in a car. Is it then safe to keep on extending oil life from about 5,000 KMs to say 13,000 KMs ?

Richard Widman • When looking at silicon, up until the SN category you normally would not see any. Most of the SN oils, to meet the anti-foam characteristics use about 6 ppm of a silicon anti-foam agent that the analysis can't differentiate from dirt. To my knowledge, no diesel formulations use silicon.
One identifier is aluminum. Aluminum, in an average environment, will be about 1/3 of the dust particles in the air. So if you have 10 ppm of silicon, and only 2 ppm of aluminum, you probably only have 6 dirt. If aluminum is more than 40% of the silicon, you definitely have a wear problem (hope that is clear).

The km you drive (or hours used) between changes is also related to the technology of the engine. Today's computer fires injected engines run a lot cleaner and will produce less soot than their carburated cousins of a few years ago. Since only about 5% of our roads are paved, I generally change diesel oil in my fleet at about 6000 km and gasoline engines around 8000, except for my Grand Cherokee where I use the computer or time factor. But I never allow anyone to open a filter housing. I have restriction sensors on every vehicle and only open the housing to change the filter when the sensor turns orange.

V.S.S. Sarma • Thank Robert Widman for his elaboration.

This point on Aluminium & Silicon relationship is noted. Where Silicon alone raises but not Aluminium, we should think that entry of dirt is not a problem. Entry of dirt would mean that both would be increasing. The Silicon may be getting generated internally from components such as seals and it does not indicate potential for catastrophic wear.

On use of Silicone anti-foam - Robert Widman should know what we blenders do. We take 1 Kg of Silicone fluid (12,500 molecular weight), add to it 99 Kg of Kerosine and make a diluted anti-foam fluid. We use about 3 ppm of this diluted fluid as an anti-foam to suppress foam. This is used across all lubricating oils (Passenger Car Motor Oils, Diesel Engine Oils, Hydraulic Oils, Gear Oils, etc.) except Turbine oils wherein the Silicone anti-foam can affect air-release value adversely and hence we use a polymer as an anti-foam (this is some 40 times costlier than the Silicone fluid approach).

Robert uses diesel engine oils for 6,000 KMs ? This is very low, my friend. Volvo recommends use of VDS-4 level oils till about 140,000 KMs. 8,000 KMs in passenger car is again low. Synthetic SM 5W40 level oils can last till about 25,000 KMs and mineral oil can last atleast till 10,000 KMs in passenger cars. Suggest Robert to review the oil drain suggestions. Europeans have great experience with their ACEA and OEM speciifed oils which last for very long times.

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toby taylor • Mark, viscosity is very important subject to the stability of the soap structure to hold a realistic temperature via continuous or interrupted running. The thixotropic nature of grease whichAlessandro Paccagnini, CLS / MLT-I • As stated by Alexey, it depends on the size of the particles.
Using AES, be it ICP or RDE, you will detect metals only up to 5 microns (8 microns in the case of

RDE), but you will have an invaluable trending tool, which is more or less unaffected by sampling errors (in my experience).
Particle counting and particle imaging (analytical ferrography and membrane patch microscopy) are a lot influenced by the sampling technique. Particle counting from 4 to 100 microns, particle imaging (including ferrography) from 10 to 500 microns.
But particle counting alone will tell you nothing about particle metallurgy or composition.
Another successful technique is PQ, which detects only magnetic particles in the mid-high size range.
At the end, metallic particle monitoring in used oil should always be performed using a multi-technique approach, with AES being the first and essential test.
(Imho)
Regards,

Alessandro

means it thickens on shear unlike normal oils so shear rate is the real control of the oils "viscosity" AR Wilson papers explains its mechanism and how it can be measured using dielectric constant of the oils properties. toby or not toby

later: Thixotropy is greases secret weapon it separates out not by chance to seal the surround of the bearing whilst it shears to its eventual stable viscosity within the rolling elements. This shear rate is very critical to obtain this wall of sealing .No one suggested that the oil was not its ultimate control of film thickness where EHL dominates with a very thin film which sustains its lubrication. Its the journey of shear rate to arrive at a this thin oil film that distinguishes it from simple viscosity change with oils. Toby

Toby,
I disagree about the importance of thixotropy here. If you use greases on high shear rate (high speeds) then soaps don't have time to reorganize structure, it breaks all the time due to shear stress. It is typical for Non-Newtonian fluids, see Rheology section of Colloid Chemistry book.
So the base oil viscosity will be the only thing keeping surfaces one from another at high speeds.
By Alexey Muralev
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If corrosion is a problem due to condensation, a small amount of a volatile amine vapour phase inhibitor, such as Morpholine /Di-n-Butylamine should be present, as it will co-distill with the water and adjust the pH to the passive range for ferrous metals.

Ahmet Guven • Addition of rust inhibitors to oils, if not too much necessary, needs to be avoided. Most of the rust inhibitors are very polar substances and will effect oil surface properties. This will efect oil's water shedding properties and in a water contamination incident oil will form steady emulsification with water. Again most of the rust inhibitors if not added in the right ratio might cause foaming which is a big head ache in gear boxes.It can not be detected by naked eye but air release property will be effected negatively which shortens oil life.
I will reco to operate gear boxes regulary for 15 to 20 minutes idle every 3 to 4 days which will help gear oil to cover the gear boxe surfaces.

David Stevenson • Kashif,
You would normally encounter this type of vapour phase corrosion in the reservoirs or header tanks of hydraulic systems, such as those used in diecasting and hot metal industries, as well as in motion compensator systems on oil platforms and fire control equipment on military ships. It's anywhere that condensation might form as the fluid gets warm and water starts to evaporate and condense on colder surfaces.
HFCs are required where sources of ignition exist that may ignite mineral oil types, particularly those under high pressure, as an aerosol generated from a leak under high pressure can result in a

massive fireball. One company that condicts testing into these types of fluids is Factory Mutual and a product that has passed FM testing is allowed to display the FM logo (the diamond with FM inside it) on the containers.

joel reber • The general rule I always used was if the machine laid dormant for 1 week between operations, then consider a vapor phase rust inhibitor. I always considered this quite conservative and believe the interval could be stretched to 30 days with a highly rust inhibited oil. Basically ,the remaining oil film on all parts after a shutdown contains enough rust inhibitor to last at least a week, usually more. Motor oil is probably more in the 3 month range and is highly rust inhibited. Industrial oils generally contain less rust inhibitor. Most industrial systems, even standby's are started monthly just to ensure proper operation for when they are needed.

Paul Michael • There is a standard test method for measuring this property. It is the ASTM D5534 - Standard Test Method for Vapor-Phase Rust-Preventing Characteristics of Hydraulic Fluids. This method may be helpful if you are trying to verify the effectiveness of a vapor-phase corrosion inhibitor.

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We have sn 150 and sn 500. Flash point of sn 150 is 180 C & flash point of sn 500 is 240 C. Is there a formula which can give flash point of blends of these 2 base oils?

John Neale   Director at John Neale Ltd

If you are using 150SN in the blend at more than a small percentage then the closed flash point will reflect the presence of the 150SN and be around 180DegC.

SANJAY KUMAR   Dy General Manager-R&D at Gulf Oil Corporation Limited

If the component with lower flash point has its significant presence (around 10%, my experience) flash point will be closer to flash point of component with lower flash point. In this case it will be around 180 deg C

Stephen Boyde   Chemical Technology Consultant

Just to add it will depend slightly on whether you use a closed cup or open cup test.
Using closed cup FP (ASTM D93) you'll definitely just measure the FP of the lowest flash ingredient as per John's original comment.
Using open cup (ASTM D92) you will see more dependence on blend composition, but it will still be dominated by the low flash component, and there isn't a universal way to predict it.
My experience has been that open cup is used more often for new oil specifications, closed cup used more for condition monitoring to pick up eg fuel contamination.

dhimant shah   CEO

Unlike viscosity , there is no way one can calculate the Flash point of the blend .As Flash point will depend on the vapours coming first while testing the flash point.Lighter fraction will vaporize first and give the flash ,even if you have the small percentage of low flash material blended into higher flash point oil it will still reflect the flash point depending on the vapourization of the lighter fraction.

Panagiotis Moraitis   Recovery and refining of precious metals

Dear Dilip, the best way to study FP variations of a blend is always the experimental one.Perhaps you should do a blend study with different proportions of SN-150 & SN-500 (I would sugget the addition of the additives since ,depending on the formulation, they also contribute to FP of the mixture) and determine the FP of each blend.

Since we are talking of virgin base oils (therefore no fuel or volatilew contamination) we can actually calculate the FP of the mixture taking under consideration the FP's of base oils and their proportions. The mathematical equations are logaritmic and the theoretical results are in agreement with the experimental ones.

Stephen Boyde   Chemical Technology Consultant

Jean-Michel - you have assumed (2) that partial pressures are proportional to weight (or volume) fraction. Actually to a first approximation they'll go with mole fraction. And if your oils are different viscosity & FP then they probably have different average molecular weight. The mole fraction of the lower FP / lower MW component is higher than its weight fraction, which pulls the calculated blend FP closer to the low end.
The approach you've used was proposed by Thiele way back, assuming that FP corresponded to vapour pressure = 10 mm Hg, and you'll find lots of canned calculation routines based on his formula. As long as the two FPs aren't too far apart it's not a bad approximation given the inherent variability of the FP test. For Dilip Shah's original question Thiele would predict FP = 195C for the 50 / 50 blend, which will certainly be closer than the straight line average of 210C. Realistic assumptions about the MW ratio will give a better estimate.
For any 2 oils, if you measure the FP of the 50/50 mix and fit the result to get an effective MW ratio then you can estimate a pretty good fit to the whole blending curve for those 2 oils. But you can't extrapolate the result to any other oils because they may have a different MW spread, so different effective MW even if same FP.

Jean-Michel Demaret   Qualified Engineer / Senior Account Manager

Thank you Stephen for your input.I am still stuck in a situation where I need the Molar Weight of a component knowing only his Flash Point. Even if deal with alkane, it seems that I still need a gas chromatography of the vapors.
I use the calculations to confirm that a low viscosity in used oil analysis results is due to diesel dilution and not a mix of different oil. The precision of the measurement is not highly important, I need an estimate only.

I found this book on Google, Encyclopedia of Chemical processing and Design which gives a formula for estimating the flash point, page 52 part 4
http://books.google.co.nz/books?id=z2NLRf0ROx0C&pg=PA54&lpg=PA54&dq=relation+flash+point+and+molar+weight&source=bl&ots=s0qBS9Rhjz&sig=vUxzZtAgAJUz09rWRR-B9RiGgNM&hl=en&sa=X&ei=xW0PVLfxPM-yuATwqoKQCA&ved=0CEIQ6AEwBA#v=onepage&q&f=false

Stephen Boyde   Chemical Technology Consultant

Blend components will normally show some variability & that Flash point is not the most accurate or reproducible measurement. In real life you are normally working with specification values for the components, or at best with CoA values measured in someone else's lab. To get good enough data points for the blend components to justify a complex calculation you would need to measure them yourself, and then why not just measure the blend as well, or instead.
The modified Thiele method does work quite well if you are working with blends of 2 synthetic fluids of the same type; the chemistry is known, you know the molecular weights accurately and the ignition chemistry is similar. But for mineral oil cuts, even if you can estimate the average MW it may vary between different lots. & the basic assumption that flash point corresponds to a critical value of vapour pressure may not be valid.

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Bill Guynes • In a fluid you are looking for high aniline point the higher the better. Next eliminate contamination entry points with desiccant breathers. Get a good oil analysis program with particle count. Every 3 to 4 months use a kidney loop filtration system. A petroleum product (hyd oil) should run 10 to 20 years with no problems if this is done, I personally have seen 27 to 30 hyd oil samples that were fine by just doing the few things above and keeping the hyd oil temp below 130F. You do not have to purchase high priced hyd oil to do this, just test for certain add packages until you are satisfied of the anti wear, de foament, r&o and high aniline point.
