

Used oil analysis



InfineumInsight.com/Learn

© 2018 Infineum International Limited. All Rights Reserved. 2018012



Outline

- Overview
 - Why analyze oil?
 - What can go wrong?
- Economics
 - Not quantitative
- Critical Factors
 - Oil Sampling
- Properties and Tests
 - Key properties
 - Alarm limits
- Oil Evolution and Oil Changes
- Interpretation
 - Real-life examples
 - Oil consumption
- Sample Oil Reports
 - What they say and how to read

© 2018 Infineum International Limited. All Rights Reserved. 2018012



Overview

- Oil analysis for an engine is like a blood test for a human
 - It is an overall check of health
 - Unusual results may lead to more tests
- A single oil analysis program will not fit all situations
 - Depends on equipment, fuel, oil, aspirations, value
- Oil analysis requires attention to detail
 - Sample timing, sample labelling, good lab, reading the reports
- Analysis of trends is the best method
 - A single point has much less value
 - Understanding equipment history leads to a better program
 - Fresh oil properties are a necessary comparison
- Often, the goal is to increase Oil Drain Interval (ODI)
 - Not always the most important target



Why analyze oil?

- New Oil also called Fresh Oil
 - Conformance with specifications
 - SAE viscosity grade, OEM ash limits, catalyst element limits, etc.
 - Manufacturing Tolerance
 - Did you make what you intended to make?
 - Contamination
 - Anything there that shouldn't be?
 - “Fit-for-Use”
- Used Oil also called In-Service Oil
 - Detect contamination
 - Fuel leaks, water leaks, broken air filter, incorrect oil added, etc.
 - Assess condition of oil
 - Rate of degradation
 - Predict timing of oil change
 - Assess condition of equipment
 - Indirect wear measurements
 - Indirect assessment of combustion



Economics for trucks, trains and large engines

- Need to balance short-term and long-term costs
- Oil Analysis \$
 - Cost of analytical tests
 - Amount depends on lab
 - Amount depends on number of tests included
- Oil Change (Planned) \$\$
 - Cost of oil, filters, labor
- Oil Change (Unplanned) \$\$\$\$
 - Cost of oil, filters, labor (maybe overtime)
 - Cost of unscheduled downtime
- Engine Failure \$\$\$\$\$\$\$
 - Cost of repairs
 - Cost of downtime
 - Cost of contractual penalties (possibly)



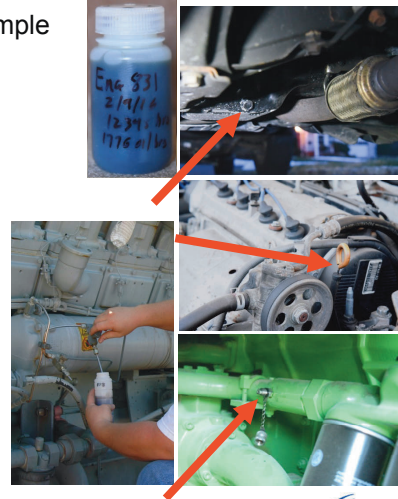
Critical factors

- Proper sampling technique
 - Representative of in-service oil
- Timely sampling
 - Sample timing depends on equipment criticality
 - Send the sample for analysis!
- Sample labelling
 - Unit, date, equipment miles/hours, oil miles/hours, at least
- Lab response time
 - Rapid enough to take action, if problem is suggested
- Reliability of lab results
 - Accuracy, precision, bias
- Fresh oil references
 - Basis for trend analysis
- Interpretation and Trending
 - A whole education in itself
 - Examples later →



Oil sampling

- Oil analysis is useless without a good sample
 - Representative of the in-service lubricant
 - Properly labelled and submitted promptly
- Sampling will depend on equipment
 - Quantity, bottle, location, and method
- Oil pan drain valve
 - Stagnant sample
 - Usually inconvenient and messy
- Vacuum pump
 - Suck a sample up the dip-stick tube
 - Convenient for cars and trucks
- Sample port
 - Tap into a pressurized oil line
 - Sample while engine is running
 - Most representative of in-service oil
 - Convenient for large, stationary engines



© 2018 Infineum International Limited. All Rights Reserved. 2018012

7

Properties and test methods



8

Used oil – what happens?

- Engine oil degrades normally in service, until it's time to be changed
 - We want to ensure it doesn't degrade too much
- Combustion
 - Acids
 - Acid Number (AN) increase
 - Base Number (BN) decrease
 - Oxidation and Nitration
 - Viscosity increase
 - Soot and insolubles
 - Radicals
 - Incomplete combustion
- Engine wear
 - Metals
 - Specific to the engine design
- Contamination
 - Fuel
 - Leaky fuel injectors or blow-by
 - Water and coolant
 - Worn gasket or breached pipe
 - Airborne debris
 - Broken air filter
 - Wrong oil added
 - Abrupt change in oil properties



Properties

- Viscosity
 - Usually 100°C or 40°C or both
- Elements
 - Additive depletion: calcium, magnesium, phosphorus, zinc, *etc.*
 - Wear: iron, copper, lead, aluminum, chromium, *etc.*
 - Contamination: sodium, potassium, silicon, *etc.*
- Neutralization Numbers
 - Acid Number, Base Number, ipH (Initial pH)
- Infrared
 - Oxidation and Nitration
 - Water and glycol
 - Fuel dilution and soot
- Physical and chemical tests
 - Water and glycol
 - Fuel dilution and soot
- Others depending on circumstance and purpose

▪ Not all tests are necessarily done on all samples

▪ Pick the suite of tests appropriate to the situation



Test methods

- Some laboratories use strictly ASTM or CEC test methods
 - Some laboratories use modified or alternate methods
- Either can give good (or bad) results – know your lab!
- Common methods:
 - Viscosity ASTM D445 or D7042 or D7279
 - Elements ASTM D5185 or D6595
 - Acid Number ASTM D664
 - ipH ASTM D664 (initial reading from AN test) or D7946
 - Base Number ASTM D2896 or D4739
 - Infrared (FTIR) ASTM E168 or E2412 or D7414 or DIN 51453
 - Water ASTM D6304 (Karl-Fischer) or FTIR
 - Glycol ASTM D7922 (Gas Chromatography) or FTIR
 - Fuel dilution ASTM D7459 (Gas Chromatography) or FTIR
 - Soot ASTM D7844 (Thermo-Gravimetric Analysis) or FTIR
 - Insolubles ASTM D893 or D4055 or D7317



Oil analysis and oil drain intervals



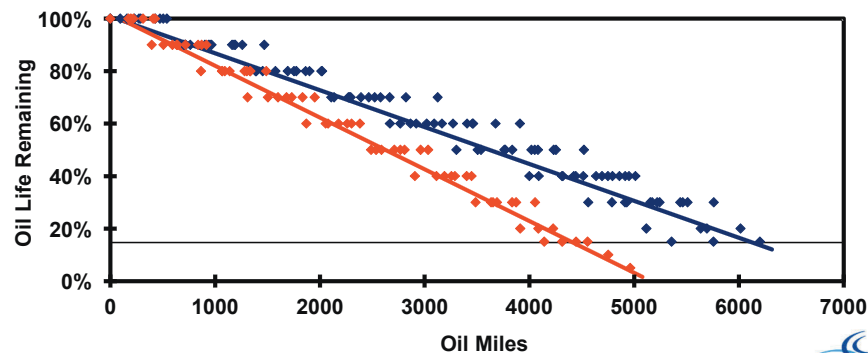
Philosophies of oil analysis and oil change

- No Oil Analysis
 - Oil changed at scheduled intervals
 - Based on manufacturer recommendation or experience
 - Makes sense for non-critical, relatively inexpensive equipment
- Validation
 - Scheduled oil changes
 - Based on manufacturer recommendation or experience
 - Oil analysis at (before) oil change (at least)
 - Extra samples during oil drain recommended
 - Intermediate samples may detect contamination or engine failure
 - “Drain only” samples will only confirm a failure has occurred
 - After-the-fact interpretation may lengthen or shorten oil drain interval
- Predictive
 - Oil changes determined by examining oil analysis results
 - Lowest cost for large equipment
 - Significant labor and judgement required



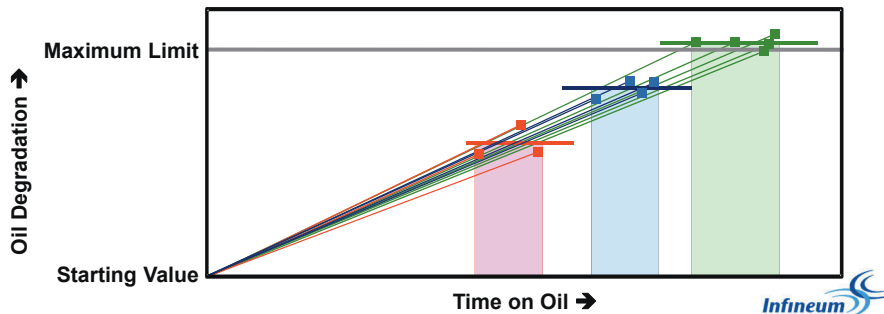
Oil change meter

- Modern cars have an oil condition digital read-out
- Different cars or driving cycles will give different results
- Algorithm depends on engine operation, not oil quality



Validation oil analysis and drain extension

- Oil drains scheduled using manufacturer's guidelines
- Oil drain sample analysis shows remaining oil life
 - Drain interval increased in small increments
- Notice: Oil performance ("slope") remains the same
 - Just taking advantage of the performance not previously captured
- Oil drain extension requires more attention
 - Less margin for error



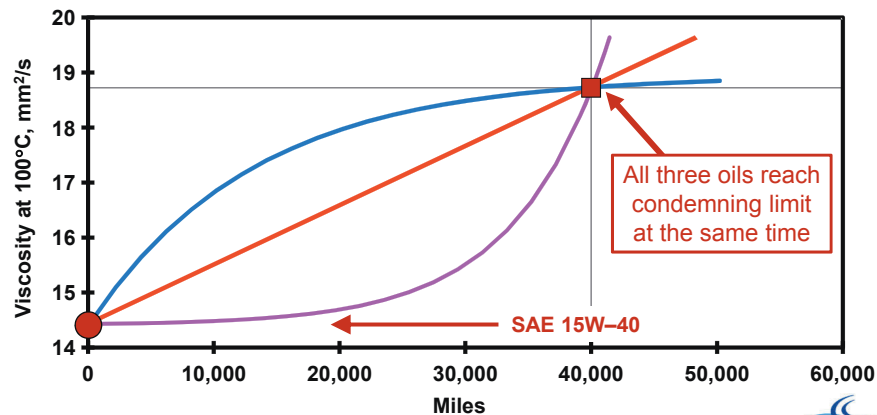
© 2018 Infineum International Limited. All Rights Reserved. 2018012

15



Oil evolution

- Single-point analysis doesn't tell the whole story
 - Which oil would you rather have?

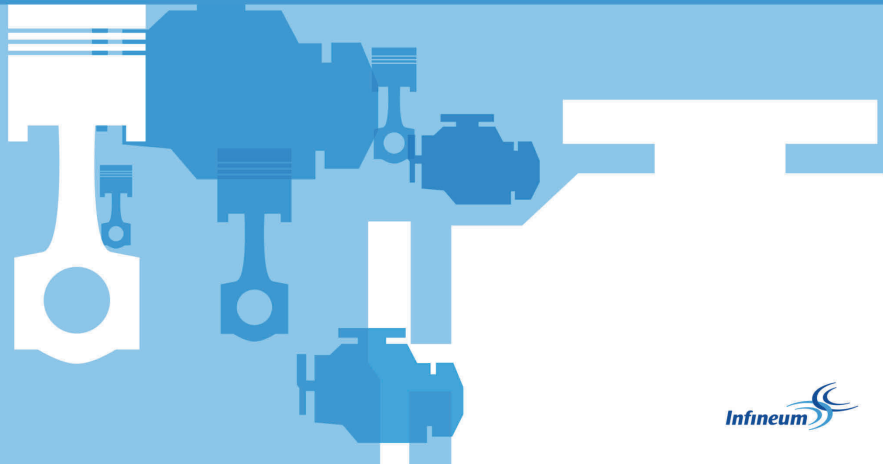


© 2018 Infineum International Limited. All Rights Reserved. 2018012

16



Examples and interpretation



17

Viscosity

- Viscosity is resistance to flow
 - See: “Viscosity and Viscosity Modifier” section of Additive Seminar

Cause

- Decrease can be caused by
 - Shearing of Viscosity Modifier
 - Fuel dilution (gasoline, diesel)
 - Contamination with improper oil
- Increase can be caused by
 - Oxidation
 - Soot
 - Insolubles
 - Deposit pre-cursors
 - Coolant contamination
 - Fuel dilution (marine)
 - Contamination with improper oil

Differential

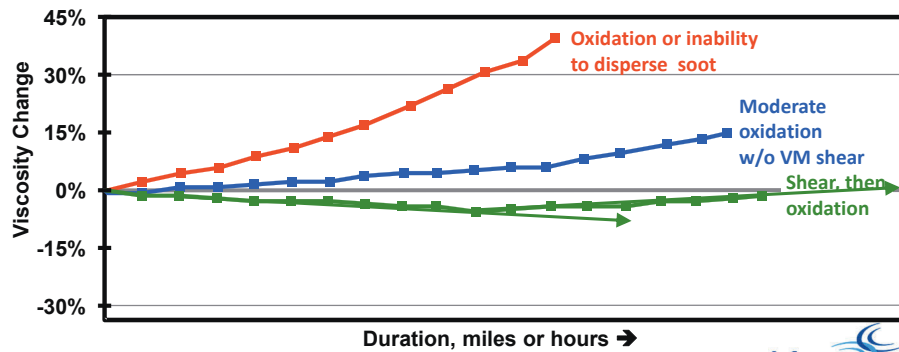
Absence of other signs
Fuel dilution, flash point
Elemental signature

Infrared, Acid Number
Soot content
Insolubles content
Infrared, Acid Number
Water, glycol, sodium, potassium
Flash point, nickel, vanadium, sodium
Elemental signature



Viscosity changes in service

- Viscosity can increase or decrease
 - Depending on balance of factors
- Increase usually oxidation or soot agglomeration
- Decrease usually Viscosity Modifier shear or fuel dilution



© 2018 Infineum International Limited. All Rights Reserved. 2018012

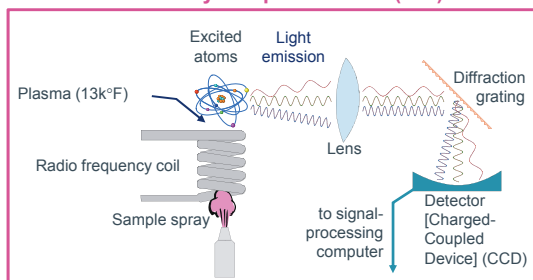


19

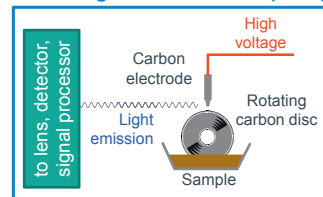
Elemental analysis

- Two common methods of Atomic Emission Spectroscopy (AES)
 - Excite atoms at high temperature, which emit light of distinct wavelengths
 - Inductively-Coupled Plasma (ICP), ASTM D5185
 - Dissolved elements and particles < 3 μm (micrometers)
 - Rotating Disc Electrode (RDE), ASTM D6595
 - Dissolved elements and particles < 8 μm (micrometers)
- X-ray AES (ASTM D4927) only suitable for new oils

Inductively-Coupled Plasma (ICP)



Rotating Disc Electrode (RDE)



© 2018 Infineum International Limited. All Rights Reserved. 2018012

20

Elemental analysis

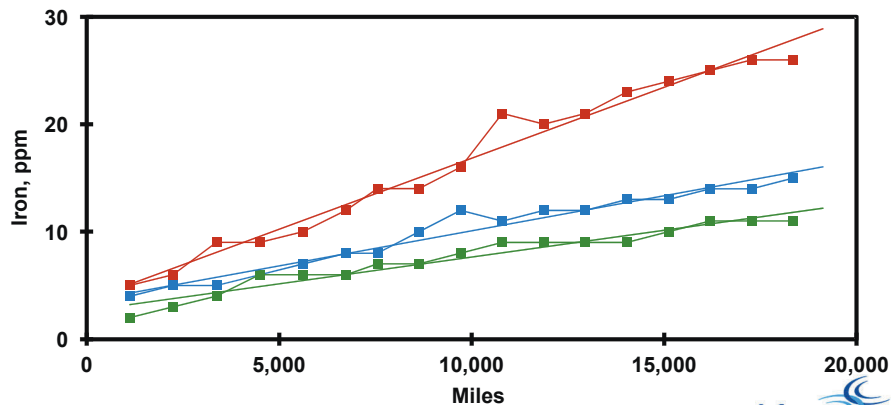
- Important to know: new oil, engine metallurgy, and coolant signatures

| Element | Possible Sources | | | |
|------------|------------------|--------------------------------|---------|--------------|
| Aluminum | | Wear (block, piston, bearings) | | |
| Barium | Additive | | | |
| Boron | Additive | | | Coolant |
| Calcium | Additive | | | |
| Chromium | | Wear (rings) | | |
| Copper | Additive | Wear (bearings) | | |
| Iron | | Wear | | |
| Lead | | Wear (bearings) | | |
| Magnesium | Additive | | | |
| Molybdenum | Additive | Wear (rings) | Coolant | |
| Nickel | | Wear | | Marine fuel |
| Phosphorus | Additive | | | |
| Potassium | | | Coolant | |
| Silicon | Additive | Wear | Coolant | Dirt or sand |
| Sodium | Additive | | Coolant | Marine fuel |
| Tin | | Wear | | |
| Vanadium | | | | Marine fuel |
| Zinc | Additive | | | |



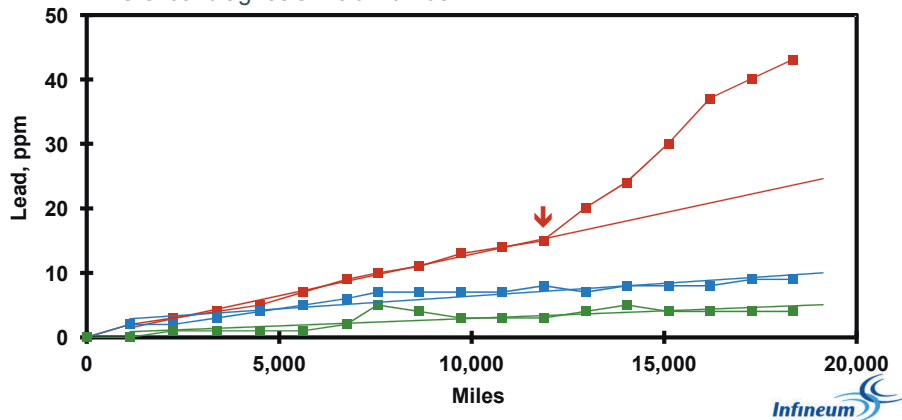
Normal wear example – Iron

- Linear, but different, controlled rates of wear
 - Probable mechanism is abrasive wear of rings, liners, cams
 - Wear rate can be extrapolated to predict engine life



Abnormal wear example – Lead

- RED oil has higher lead wear rate than BLUE or GREEN
 - Particularly troubling is the departure from linear trend
 - Probable mechanism is corrosive bearing wear: more investigation →
 - Differential diagnosis: Acid Number

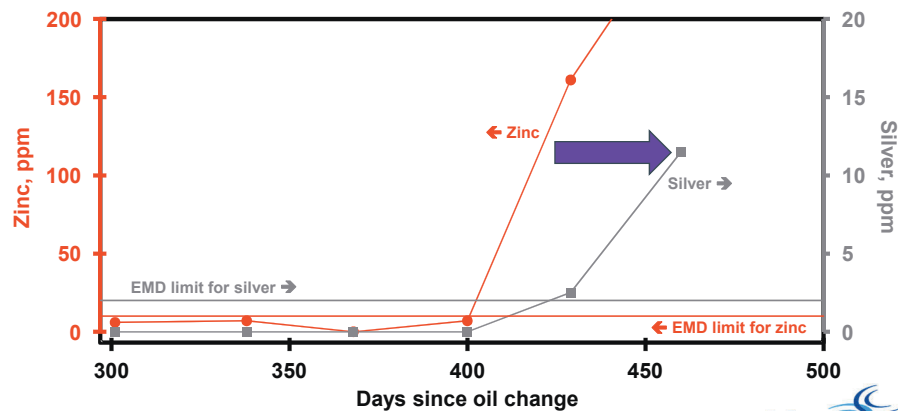


© 2018 Infineum International Limited. All Rights Reserved. 2018012

23

Elemental contamination and consequences

- EMD engines with silver bearings are corroded by zinc
 - Railroad oil contaminated with Heavy Duty oil



© 2018 Infineum International Limited. All Rights Reserved. 2018012

24

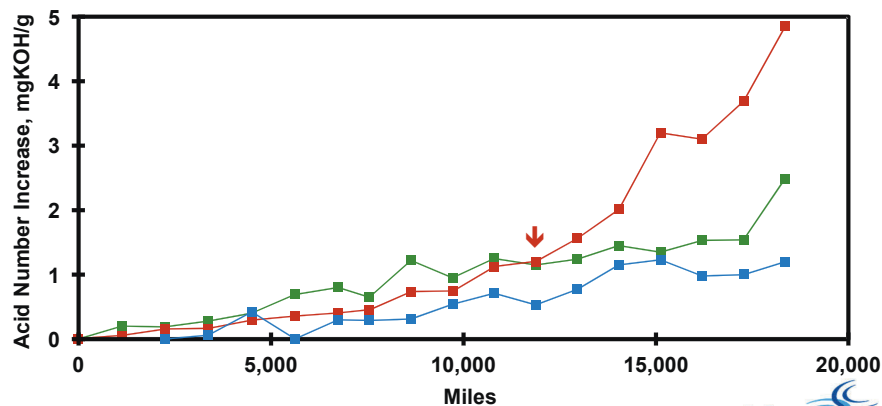
Acid Number (AN)

- Acid Number by ASTM D664
 - Titration method
 - Base is added until acid is neutralized
 - Detected by electrode
 - Amount of base needed to neutralize is the Acid Number
 - Expressed as mg KOH/g (milligrams potassium hydroxide *per* gram of oil)
- Acids are formed by:
 - Oxidation of hydrocarbons (weak organic acids)
 - Oxidation of sulfur (if any) in fuel (strong inorganic acids)
 - Oxidation of nitrogen from air (strong inorganic acids)
 - Exhaust gas re-circulation (EGR)
- Some additives are acidic by this measurement
 - Acid Number Increase is a better measure than absolute Acid Number



Acid Number example

- Sudden increase in **RED** Acid Number coincides with lead increase
 - Supports corrosive bearing wear hypothesis



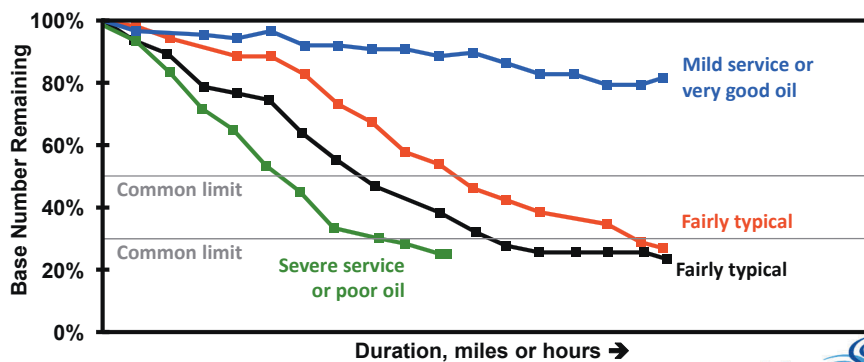
Base Number (BN)

- Base Number by ASTM D2896 or D4739
 - Titration method: Acid is added until neutral, as detected by electrode
 - Amount of acid needed to neutralize is the Base Number
 - Expressed as equivalent mg KOH/g (milligrams potassium hydroxide *per* gram of oil)
- Methods use two different acids:
 - D2896 uses Perchloric Acid (very strong) – detects strong and weak bases
 - D4739 uses Hydrochloric Acid (strong) – detects strong bases only
- Bases are provided by additives to neutralize acids:
 - Detergents (strong base)
 - Dispersants (weak base)
 - Some antioxidants (weak base)
- Base Number will decrease as acids are neutralized (doing their jobs)
- Different manufacturer's specify may different BN tests
 - Sometimes percent decrease, sometimes absolute minimum



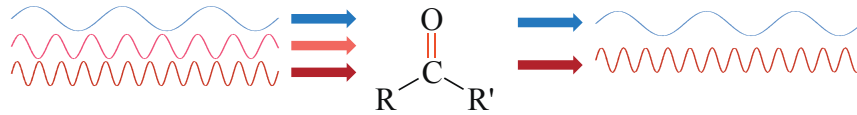
Base Number example

- Base Number can decrease fast or slow
 - Depending on engine, fuel, service, and oil
- Specific limits usually suggested by engine manufacturer
 - Either percent decrease (remaining) or absolute minimum number



Infrared

- Infrared is light with longer wavelength than visible red
 - 2500 – 20,000 nm (nanometers) vs. 400 – 800 nm for visible
 - Corresponds to frequency of molecule vibrations
 - Can be used to identify specific molecular combinations
 - Oil industry methods focus on oxidation, nitration, and contaminants
 - More detailed analysis can give more information
 - Usually measured as “Fourier Transform Infrared” (FTIR)
 - Instrumental technique to increase resolution and decrease time
 - Several different test methods, with lack of industry standardization
 - ASTM E2412 and DIN 51453:2004 common, but variations exist →
 - Good for trend analysis from a single lab
 - Caution comparing across laboratories
- Very good for detecting oxidation and nitration
 - Somewhat less reliable – but cost-effective – for water, fuel dilution, soot
 - Usually good enough for routine condition monitoring

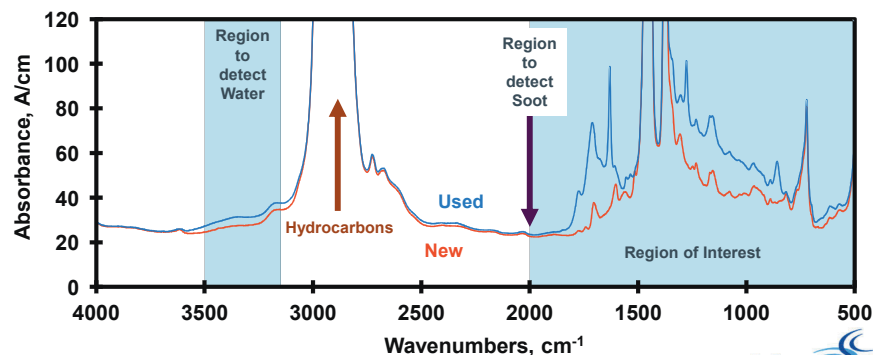


© 2018 Infineum International Limited. All Rights Reserved. 2018012

29

Infrared example – full spectrum

- Plot of amount absorbed against wavenumbers (light frequency)
 - Note: X-axis is backwards – wavelength increases from left to right
- Most of the interest is between 500 – 2000 cm^{-1}
- Expand the region of interest for further examination →

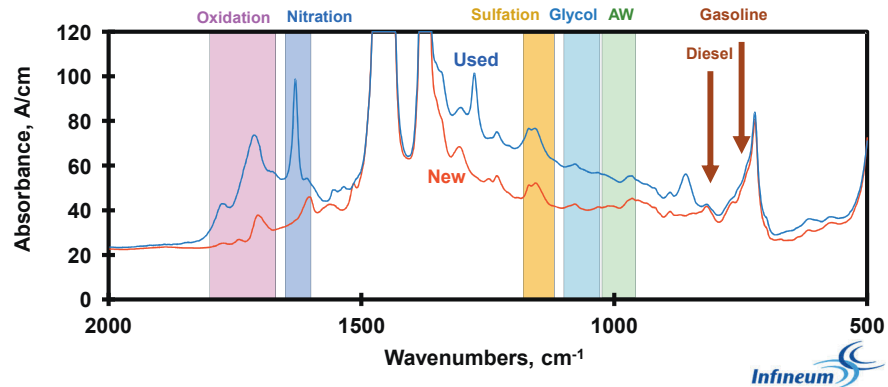


© 2018 Infineum International Limited. All Rights Reserved. 2018012

30

Infrared example – region of interest

- Region of interest shows many features important for used oil analysis
- Interested in changes from **fresh** oil
 - Necessary to have a fresh oil reference spectrum



© 2018 Infineum International Limited. All Rights Reserved. 2018012

31

Water and glycol

- Water can enter the oil through condensation or coolant leaks
 - Or rain!
- Glycol is often a component of the coolant treatment (anti-freeze)
 - Coolants treatments also have anti-corrosion chemicals
- Important to know the composition of the coolant
- Water and coolant contamination can be detected by:
 - Distillation
 - Most accurate, but lengthy and expensive
 - Karl-Fischer (titration)
 - Good choice for water
 - Gas Chromatography
 - Good choice for glycol
 - FTIR
 - Questionable accuracy, but inexpensive
 - Elemental analysis
 - From coolant anti-corrosion treatment
 - Common elements: sodium, potassium
 - Less common: boron, molybdenum
 - Single element may not signal coolant contamination
 - Important to compare to fresh oil (which may have similar elements)

© 2018 Infineum International Limited. All Rights Reserved. 2018012

32

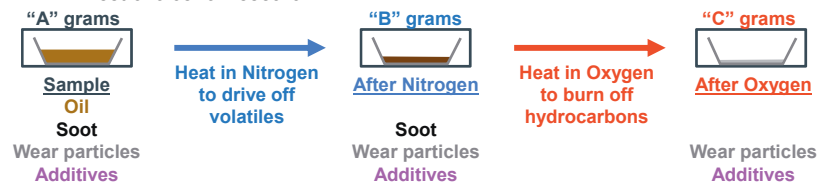
Fuel dilution

- Fuel contamination will lower flash point
 - Safety concern
 - Flash point not commonly included in routine oil analysis
- Fuel contamination will affect viscosity
 - Decrease: gasoline, diesel
 - Increase: marine diesel
- Fuel contamination can be detected by
 - Viscosity
 - Not specific: too many other contributors
 - Flash Point
 - Not usually included in analysis
 - Gas Chromatography
 - Good choice, but expensive
 - FTIR
 - Questionable accuracy
 - Needs calibration for quantitation



Soot

- Soot is a by-product of incomplete combustion
 - Unburned hydrocarbons plus coked ("cooked") fuel
 - Usually only seen with heavy, liquid fuels = diesel
- Soot can be measured by:
 - Thermogravimetric Analysis (TGA)
 - most accurate, but expensive
 - Soot = B – C
 - Best choice for research



- FTIR
 - fast and convenient
 - Adequate for condition monitoring, but requires calibration
 - Sometimes called "Wilkes Soot"
- Insolubles tests
 - not exactly "soot"



Insolubles

- Insolubles includes soot plus:
 - Oxidation products
 - Varnish pre-cursors
 - Additive drop-out
- Insolubles not usually included in engine oil analysis programs
 - Unless manufacturer requires it for approval
 - Sometimes part of a bench or engine test
- Insolubles can be measured by:
 - Centrifuge tests – standard, but imprecise ($r = 7\%$ to $>100\%$)
 - Oil is mixed with solvent (pentane or toluene) and centrifuged
 - Sometimes, coagulant is added to promote drop-out
 - Filtration tests – lengthy and questionable accuracy
 - Oil is mixed with solvent (usually pentane) and filtered
 - Sometimes vacuum-filtered
 - Sets limits on particle size detected (usually $8\ \mu\text{m}$, but can be changed)
 - Blotter tests – might be useful for field check



Other properties

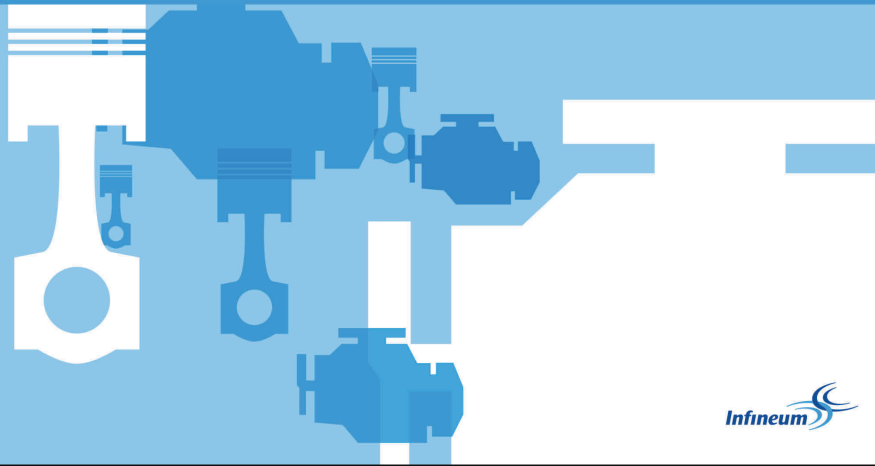
- Many other properties can be measured
- Need to consider cost of analysis vs. value of information
 - Research field tests include many more tests than routine fleet programs
- Some tests available for used oil analysis programs include:

| | |
|---|--|
| <ul style="list-style-type: none"> – Flash point – Pour point – Aniline point – Bench oxidation tests – Ferrography – Color | <ul style="list-style-type: none"> – Fuel dilution – Anti-oxidant depletion – Measure of aromatic content – Antioxidant remaining – Wear mechanisms – Supposedly degradation |
|---|--|
- Some tests commonly used for industrial, not engine, oils:

| | |
|--|--|
| <ul style="list-style-type: none"> – Rust or corrosion – Particle size – Remaining Useful Life (RULER) – Filterability – Emulsion or demulsibility – Foaming | <ul style="list-style-type: none"> – Anti-corrosion depletion – Amount and type of wear – Anti-oxidant depletion – Alternate for particles – Water separation – Antifoam depletion |
|--|--|



Oil analysis reports



37

Oil reports

- Each laboratory has their own format and conventions
 - Samples across and results down
 - Samples down and results across
 - Last sample first or first sample first
 - Familiarity comes quickly
- Oil report may or may not specify test methods
 - The lab will be happy to provide details and interpretation
- Oil report will usually have some flag for unusual results
 - Unlikely to explain what the limits are or how arrived
 - The lab will be happy to explain
- If changing labs, double-sample for a period
 - To establish cross-correlation
- If using multiple labs, cross-check periodically



38

Summary

- Oil analysis can be a very useful technique to:
 - Assess the condition of engine oil
 - Assess the condition of the engine
 - Predict and schedule maintenance
 - At lower cost than unscheduled maintenance
 - Extend the life of the oil
 - Extend the life of the engine
 - Save money
- Oil analysis must balance:
 - Cost of analysis vs. risk and cost of equipment failure
- Interpretation of oil analysis requires attention to detail
 - There are many training courses available
 - Your oil analysis lab is a good place to start



Permission is given for storage of one copy in electronic means for reference purposes. Further reproduction of any material is prohibited without prior written consent of Infineum International Limited.

The information contained in this document is based upon data believed to be reliable at the time of going to press and relates only to the matters specifically mentioned in this document. Although Infineum has used reasonable skill and care in the preparation of this information, in the absence of any overriding obligations arising under a specific contract, no representation, warranty (express or implied), or guarantee is made as to the suitability, accuracy, reliability or completeness of the information; nothing in this document shall reduce the user's responsibility to satisfy itself as to the suitability, accuracy, reliability, and completeness of such information for its particular use; there is no warranty against intellectual property infringement; and Infineum shall not be liable for any loss, damage or injury that may occur from the use of this information other than death or personal injury caused by its negligence. No statement shall be construed as an endorsement of any product or process. For greater certainty, before use of information contained in this document, particularly if the product is used for a purpose or under conditions which are abnormal or not reasonably foreseeable, this information must be reviewed with the supplier of such information.

Links to third party website from this document are provided solely for your convenience. Infineum does not control and is not responsible for the content of those third party websites. If you decide to access any of those third party websites, you do so entirely at your own risk. Please also refer to our Privacy Policy.

'INFINEUM', the interlocking Ripple Device, the corporate mark comprising INFINEUM and the interlocking Ripple Device and 润英联 are trademarks of Infineum International Limited.

© 2018 Infineum International Limited. All rights reserved.

