Performance you can rely on.

Used oil analysis

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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

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

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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)

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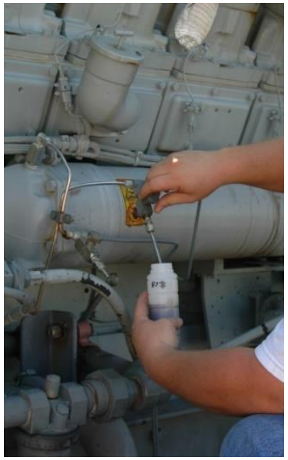
Critical factors

- 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



Properties and test methods

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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
  - Radicals
  - Incomplete combustion
- Engine wear
  - Metals
- Contamination
  - Fuel
  - Water and coolant
  - Airborne debris
  - Wrong oil added

Acid Number (AN) increase
Base Number (BN) decrease
Oxidation and Nitration
Viscosity increase
Soot and insolubles

Specific to the engine design

Leaky fuel injectors or blow-by
Worn gasket or breached pipe
Broken air filter
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

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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
  - Acid Number — ASTM D664
  - ipH — ASTM D664 (initial reading from AN test)
  - 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

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Considerations common to all test methods

• Accuracy	– agreement with accepted values
• Precision	– agreement among repeated measurements
• Bias	– systematic difference from reference values
• Detection Limit	– lowest value that can be measured
• Saturation Limit	– highest value that can reliably measured
• Time	– how long until you get the results
• Cost	– obvious

- Not reviewed here in detail for each test
- Keep in mind when selecting tests for your monitoring program
- Keep in mind when interpreting results

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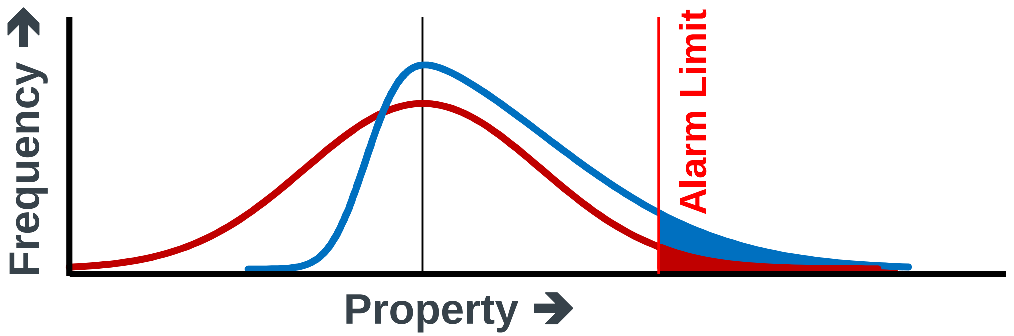
Setting alarm limits

- Alarm – or condemning – limits – are the points where action is required
- Sometimes one limit: Pass/Fail
- Sometimes tiered limits: Good, Caution, Action, Danger
  - Or: Change Oil at Next Fueling, Change Oil Now, Remove from Service
- Limits can be set several ways:
  - Engine manufacturer's specification or guidelines
  - Let the analysis lab do it
  - Statistical analysis of historical database
  - Often, some or all of these are synonymous
- Alarm can be a fixed number, or a rate of change
  - Can be a point where a known bad thing happens
  - Alarms can be set using multiple parameters in combination – complicated



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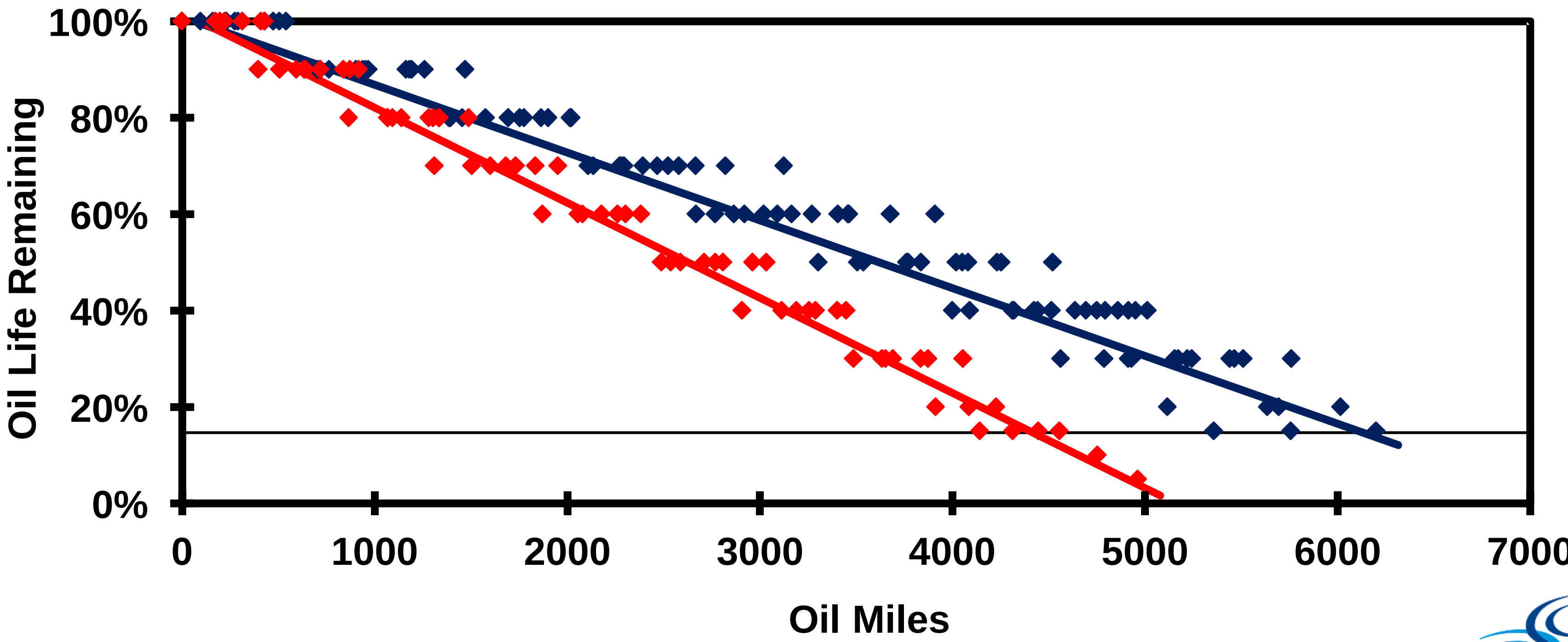
Oil analysis and oil drain intervals

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

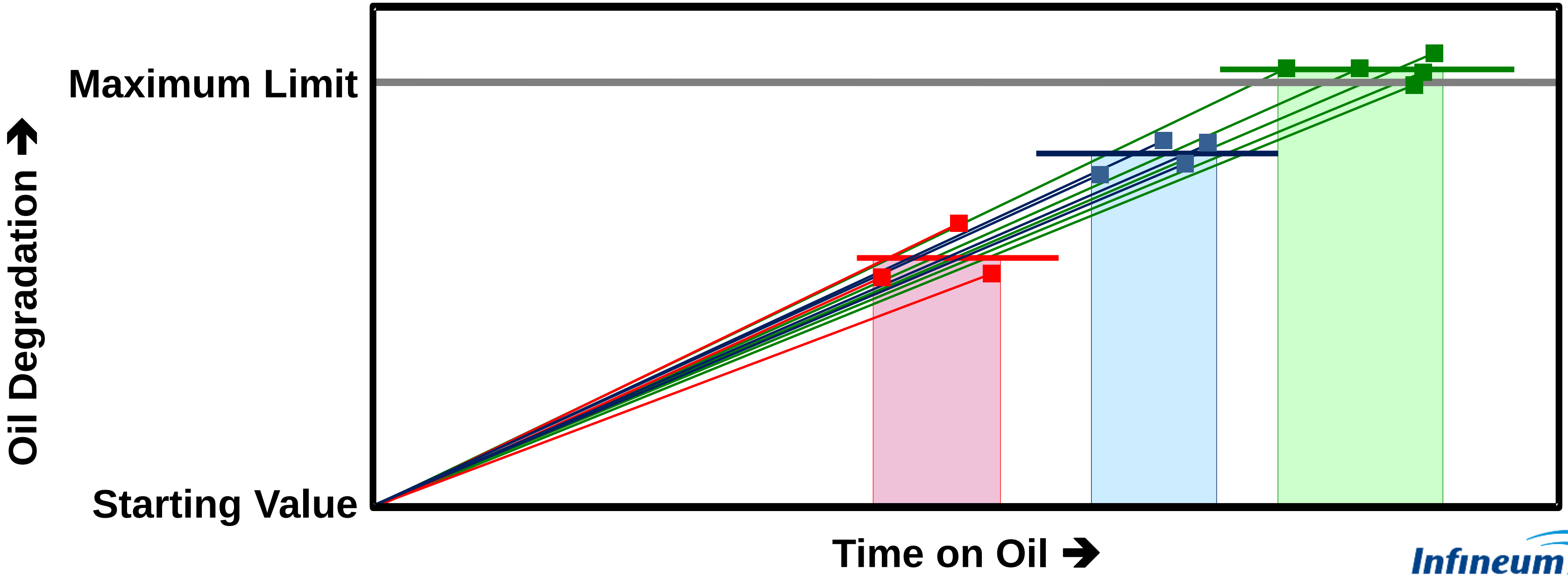
Oil change meter

- 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



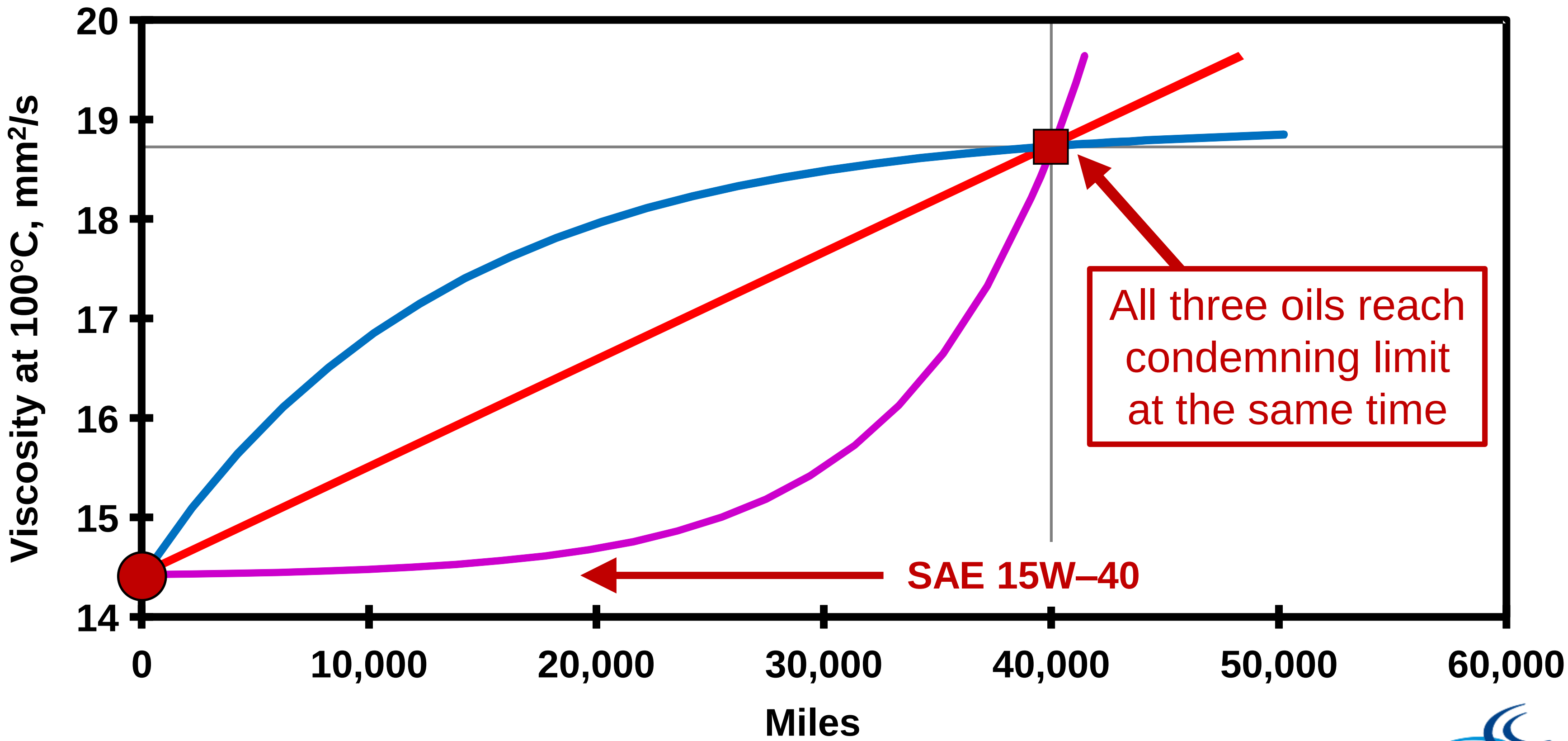
Validation oil analysis and drain extension

- 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



Oil evolution

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



Examples and interpretation

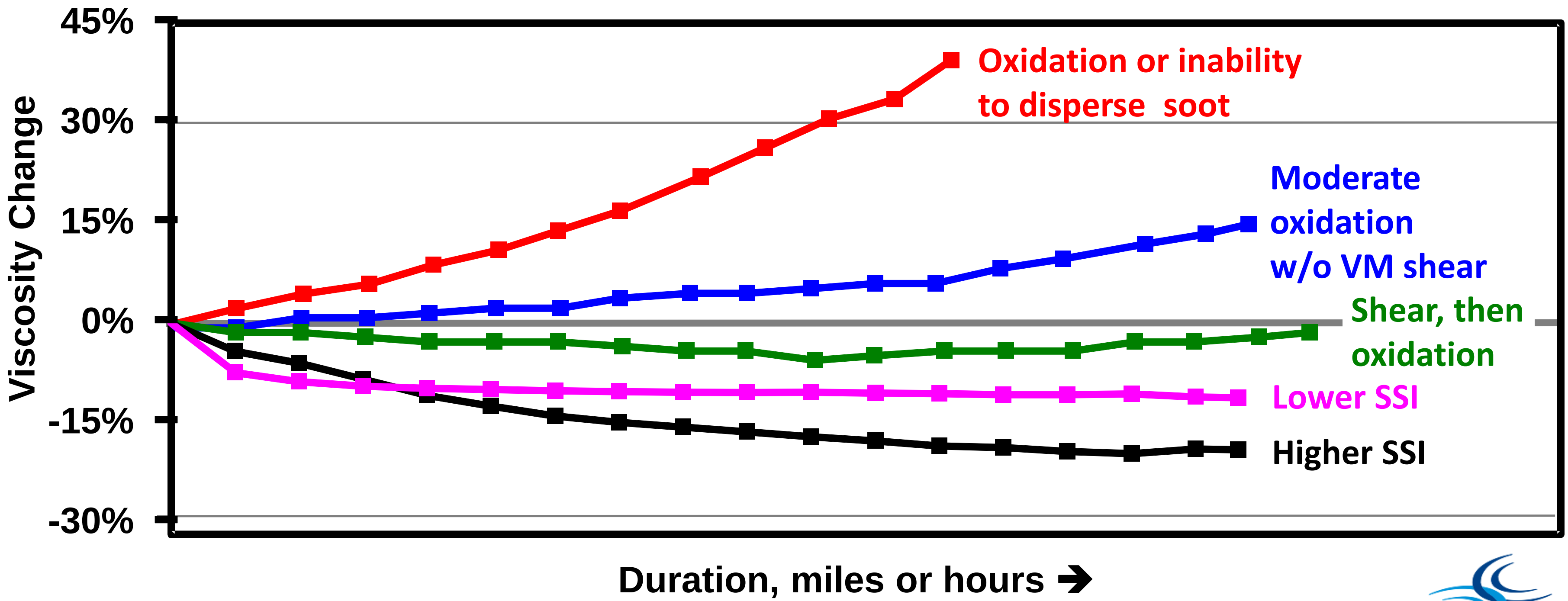
Viscosity

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

Cause	Differential
Decrease can be caused by	
– Shearing of Viscosity Modifier	Absence of other signs
– Fuel dilution (gasoline, diesel)	Fuel dilution, flash point
– Contamination with improper oil	Elemental signature
Increase can be caused by	
– Oxidation	Infrared, Acid Number
– Soot	Soot content
– Insolubles	Insolubles content
– Deposit pre-cursors	Infrared, Acid Number
– Coolant contamination	Water, glycol, sodium, potassium
– Fuel dilution (marine)	Flash point, nickel, vanadium, sodium
– Contamination with improper oil	Elemental signature

Viscosity changes in service

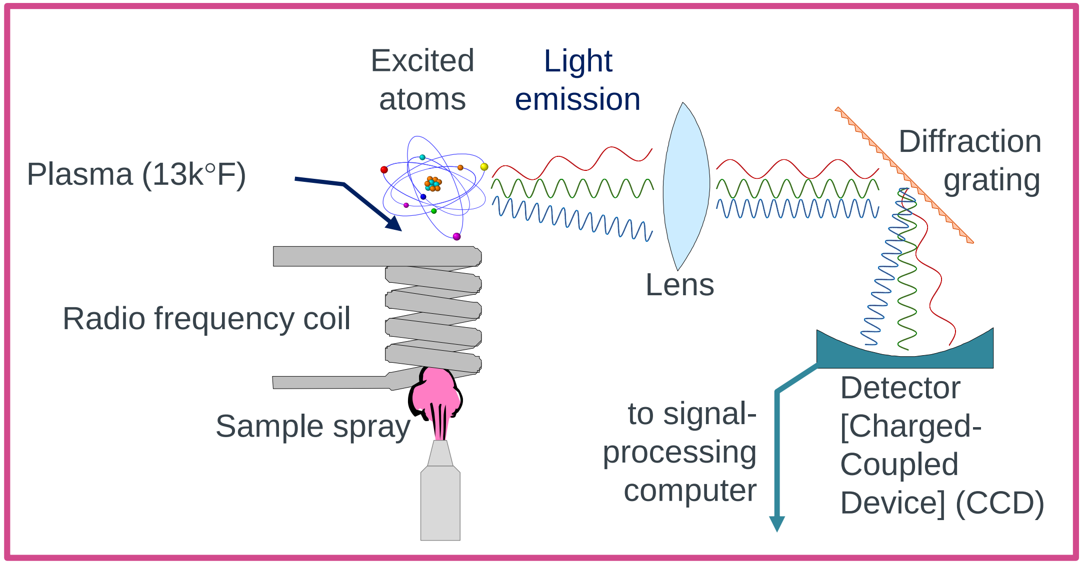
- Viscosity can increase or decrease
  - Depending on balance of factors
- Increase usually oxidation or soot agglomeration
- Decrease usually Viscosity Modifier shear or fuel dilution



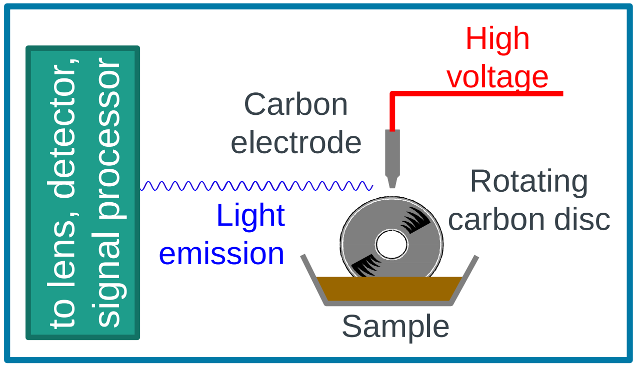
Elemental analysis

- 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)



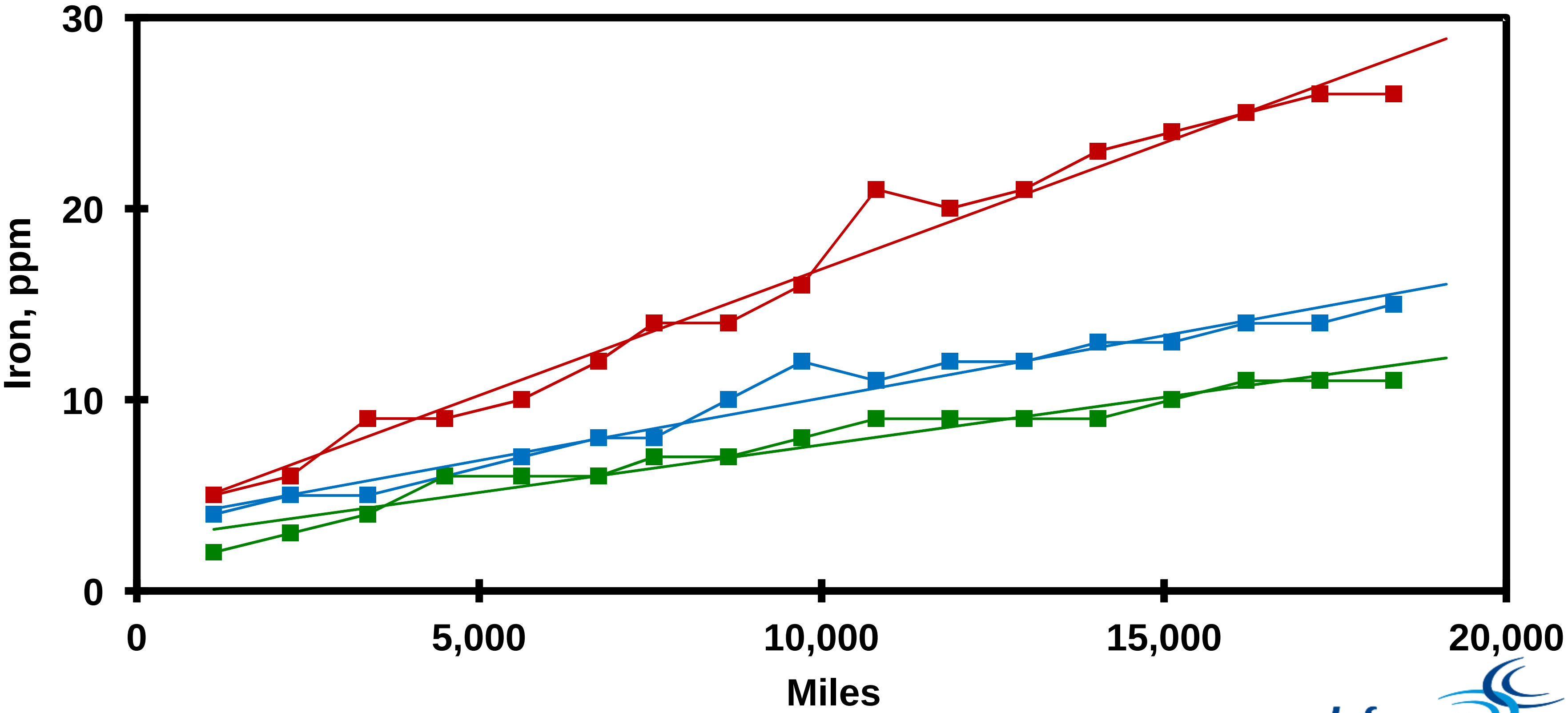
Elemental analysis

- 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



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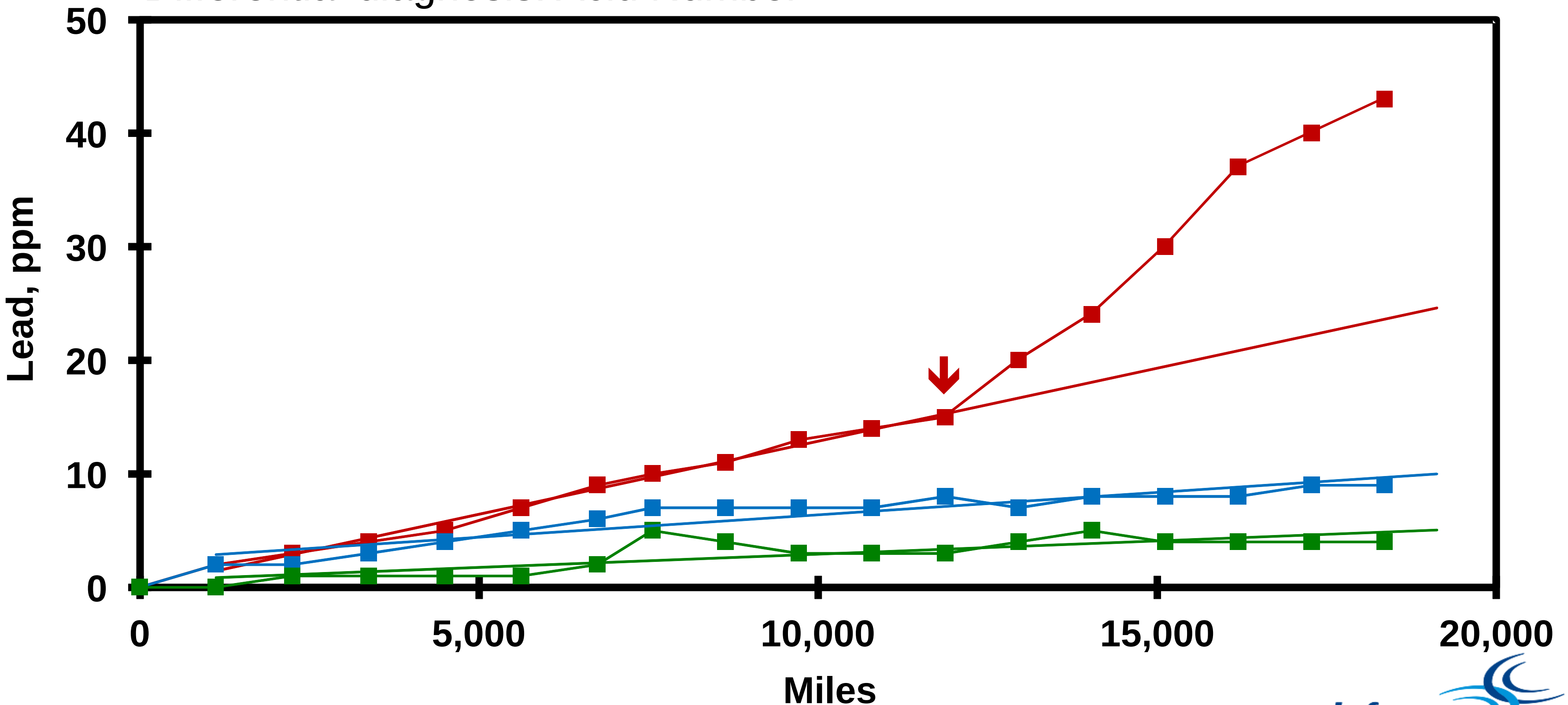
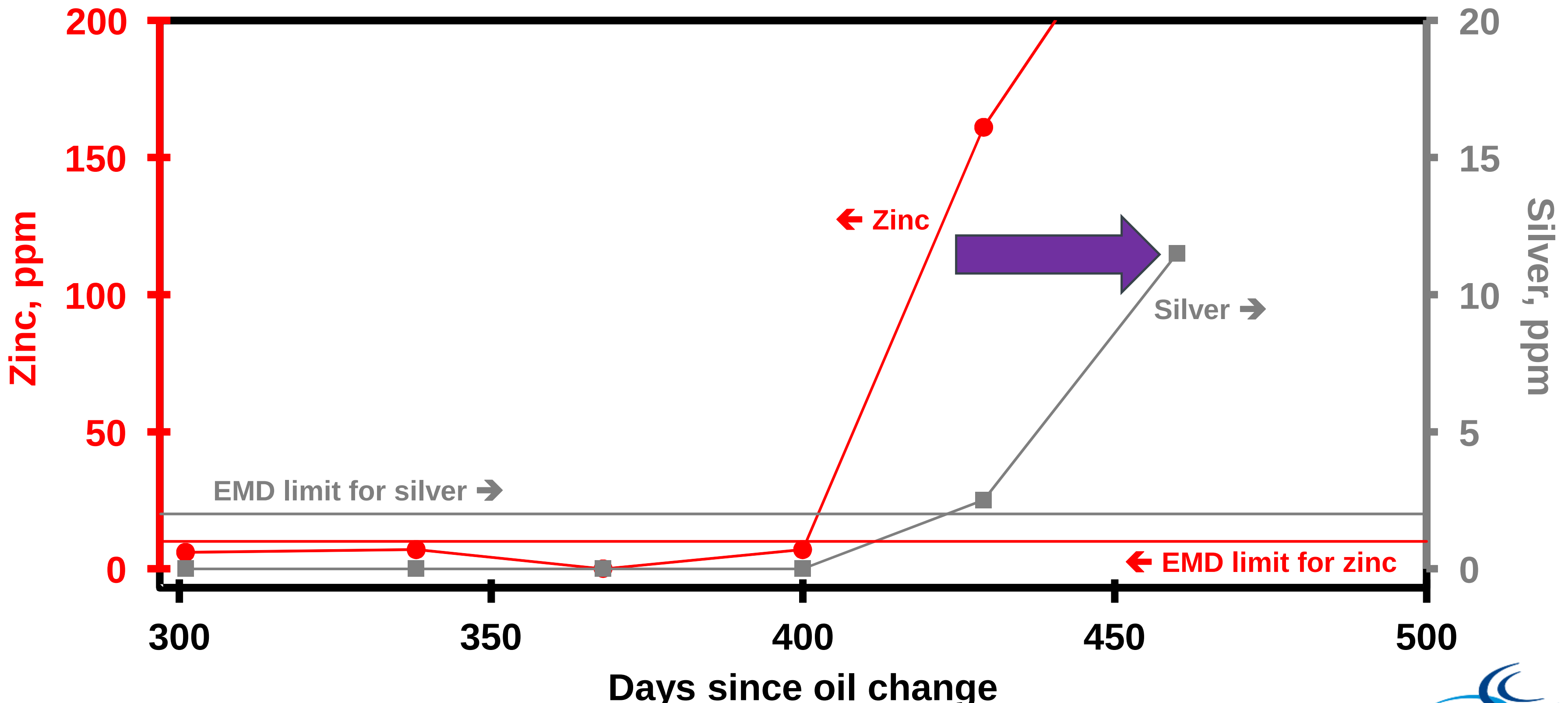
Abnormal wear example – lead

- 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



Elemental contamination and consequences

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



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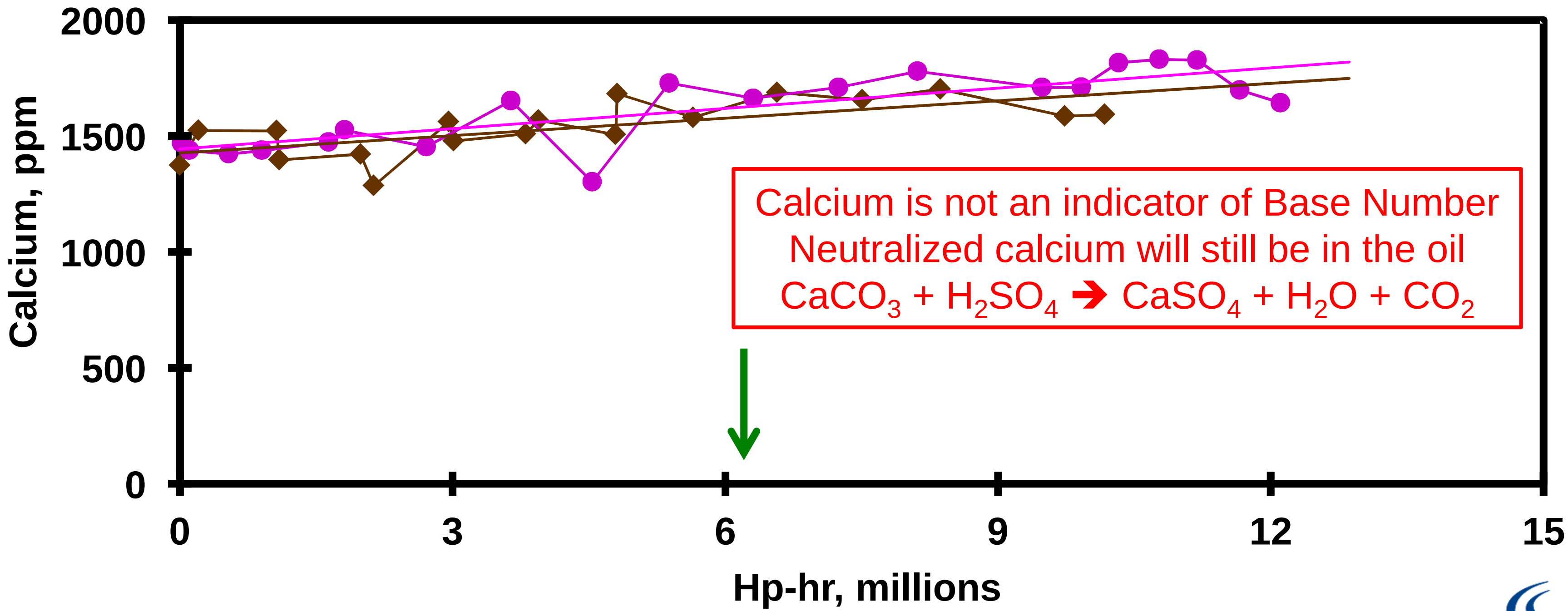
Elemental evidence of oil consumption

- Non-volatile elements concentrated by base stock evaporation
  - Does not reflect total oil consumption – only evaporative loss
  - Physical transport past piston rings is the other major contributor



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

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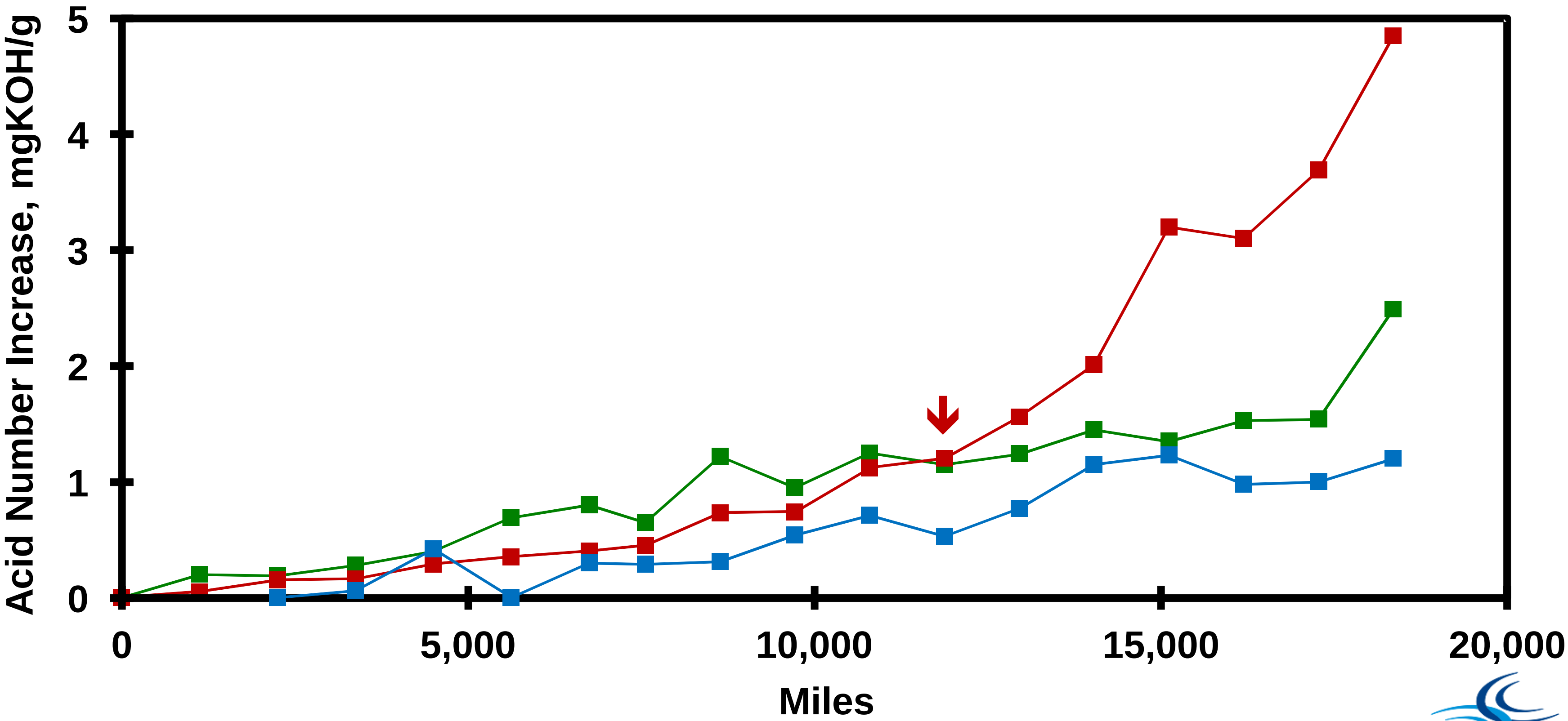
Acid number example

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



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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

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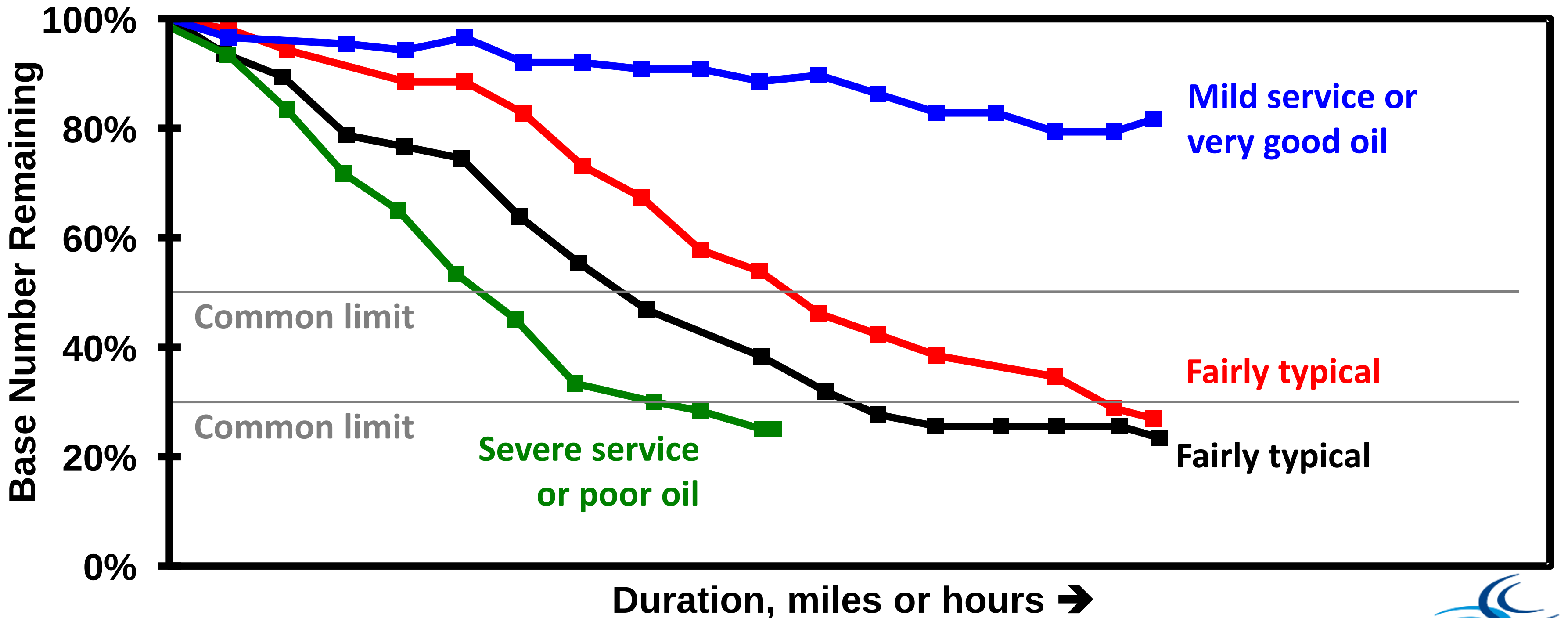
Base number example

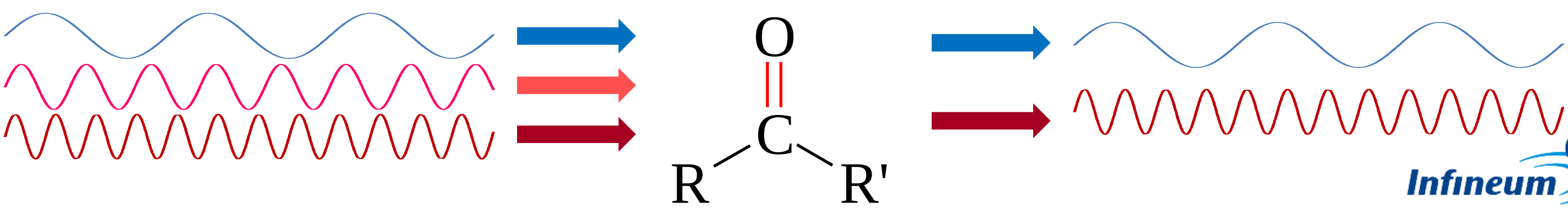
- Base Number can decrease fast or slow
  - Depending on engine, fuel, service, and oil
- Specific limits usually suggested by engine manufacturer
  - Either percent decrease (remaining) or absolute minimum number



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



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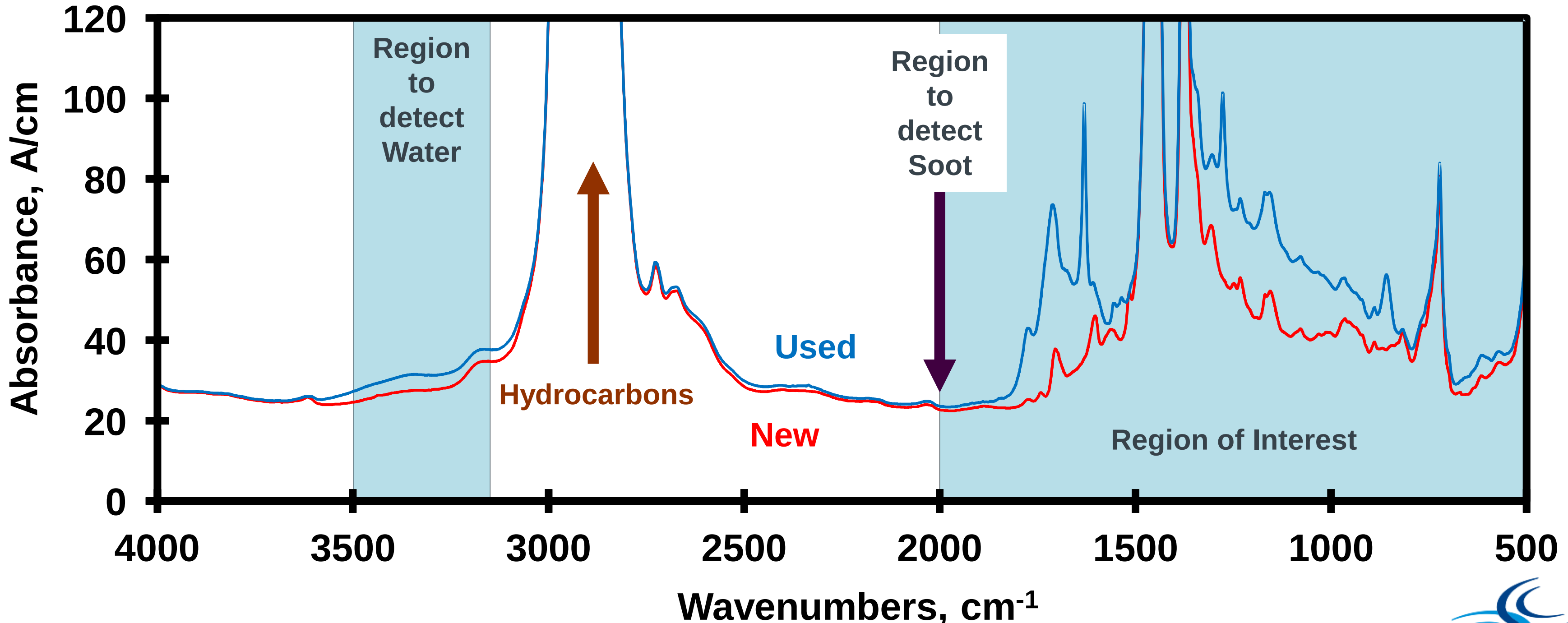
Infrared

- Amount of infrared light absorbed is plotted against light frequency
  - "Wavenumber" is 1/wavelength in "reciprocal centimeters" = cm^{-1}
  - Frequency = speed of light / wavelength
  - Wavenumber is proportional to energy
- Amount of light absorbed can be expressed as:
  - Either Peak Height or Peak Area ➔
- Amount of light absorbed depends on length of sample
  - Peak Height expressed as "Absorbance *per* centimeter"
    (Not the same centimeters as in wavenumber) – can be confusing
  - Commonly, some labs use an instrument with a 0.1 mm cell
    They might express as "Absorbance *per* 0.1 mm" – be careful
  - Peak Area expressed as "Absorbance *per* centimeter squared"
  - Very often, units are not given – you must infer from values
    Ask the lab
- For used oil analysis, the fresh oil "background" must be subtracted
  - It is critical to have a fresh oil reference sample

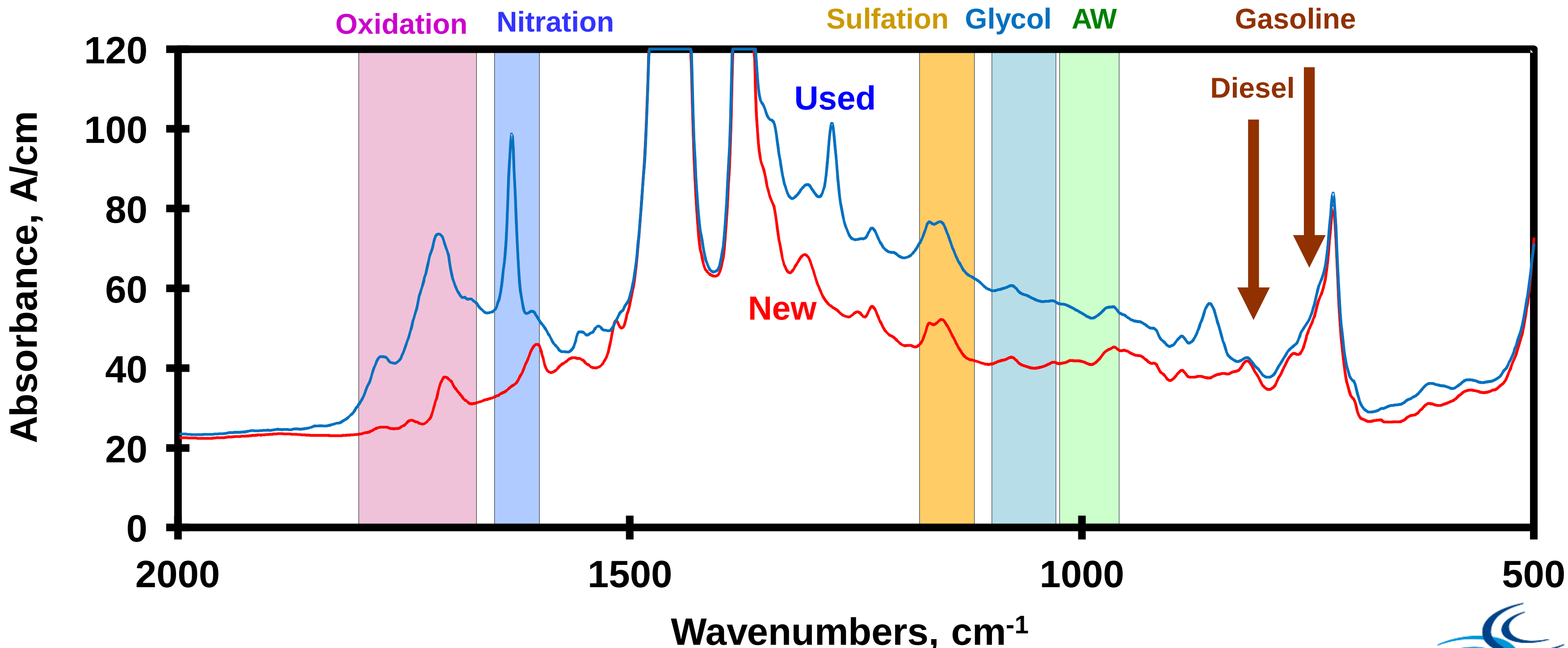
Infrared example – full spectrum

- 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 ➔



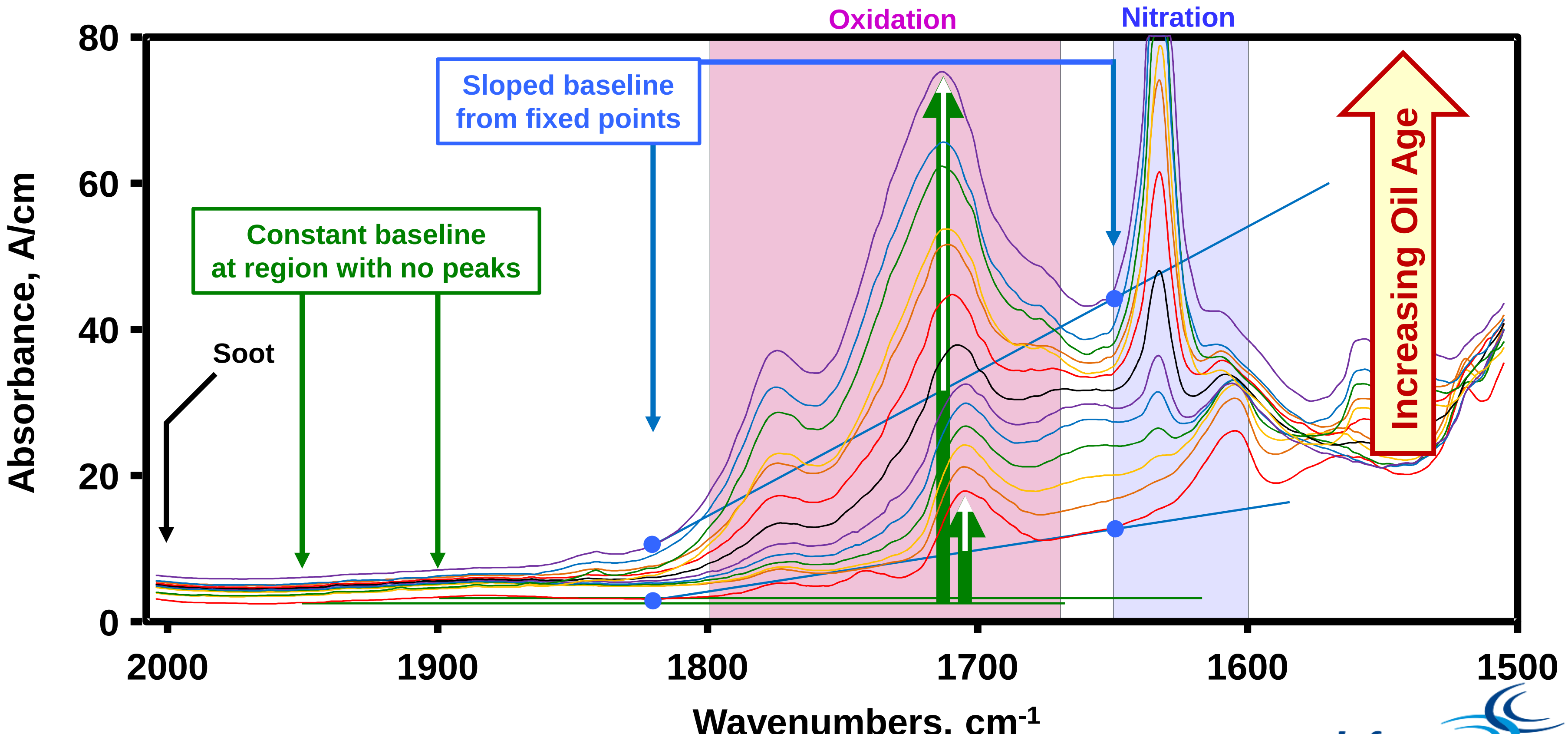
Infrared example – region of interest

- 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



Infrared example – oxidation

- As peak height increases, so does baseline ➔
  - Also note: oxidation peak location is shifting slightly to the left with age
- Severe nitration also evident



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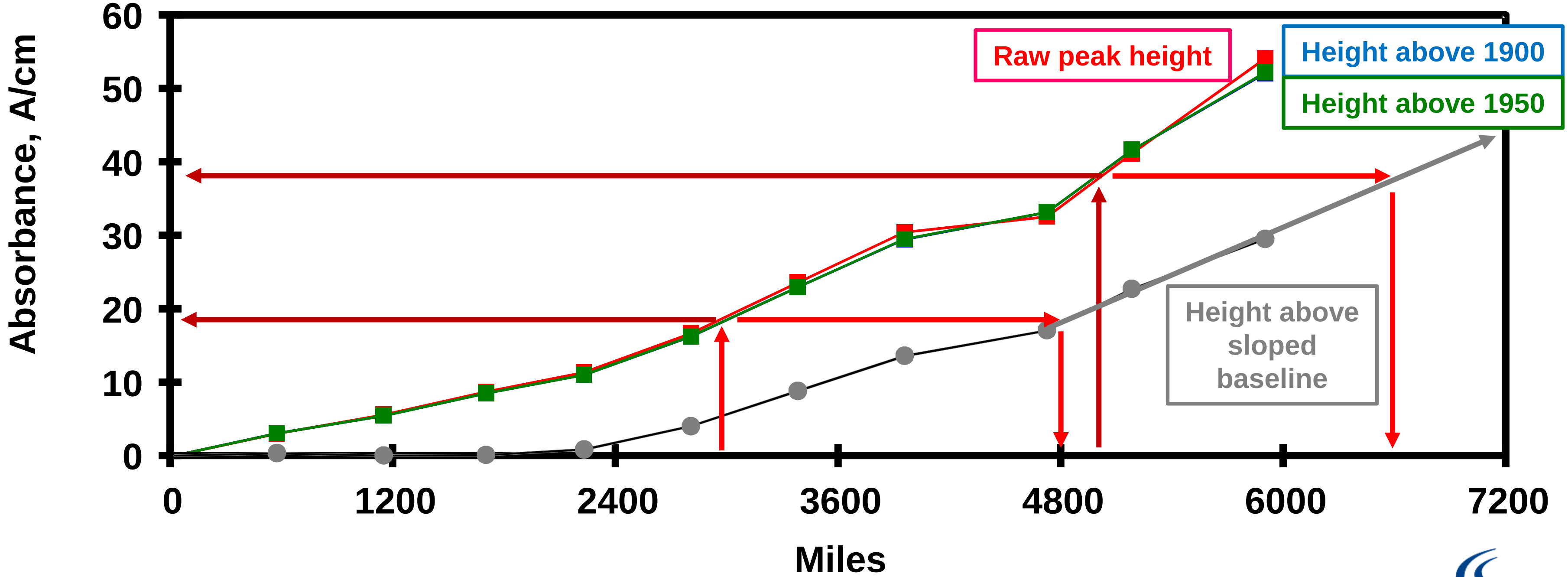
Infrared example – correlation of methods

- Four methods correlate, but one gives different values
- Limits and oil change decisions must be made consistently
  - Or else value will be lost or equipment put at risk



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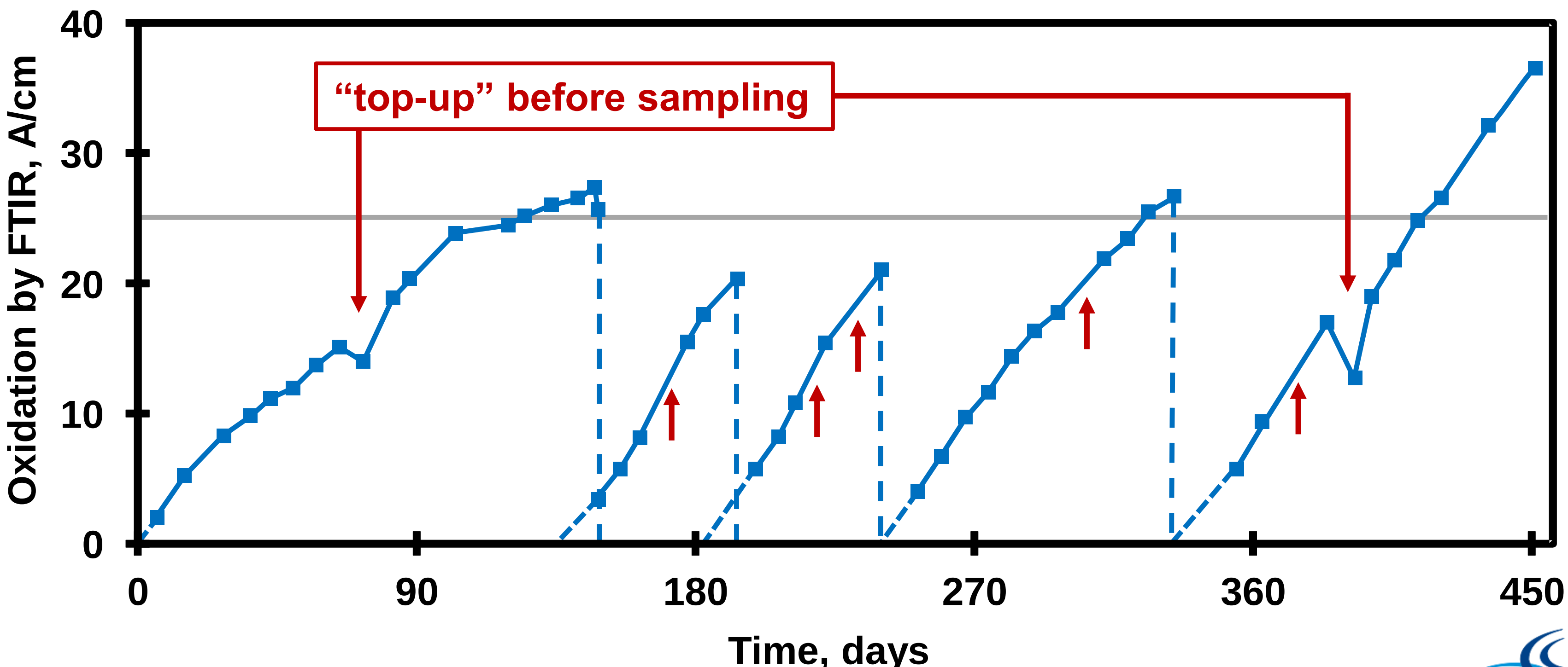
Oil sampling – good practices

- Take oil sample before "topping-up" or results are distorted
  - Cannot easily assess oil or engine condition
- Incomplete oil change will shorten next oil change interval



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Water and glycol

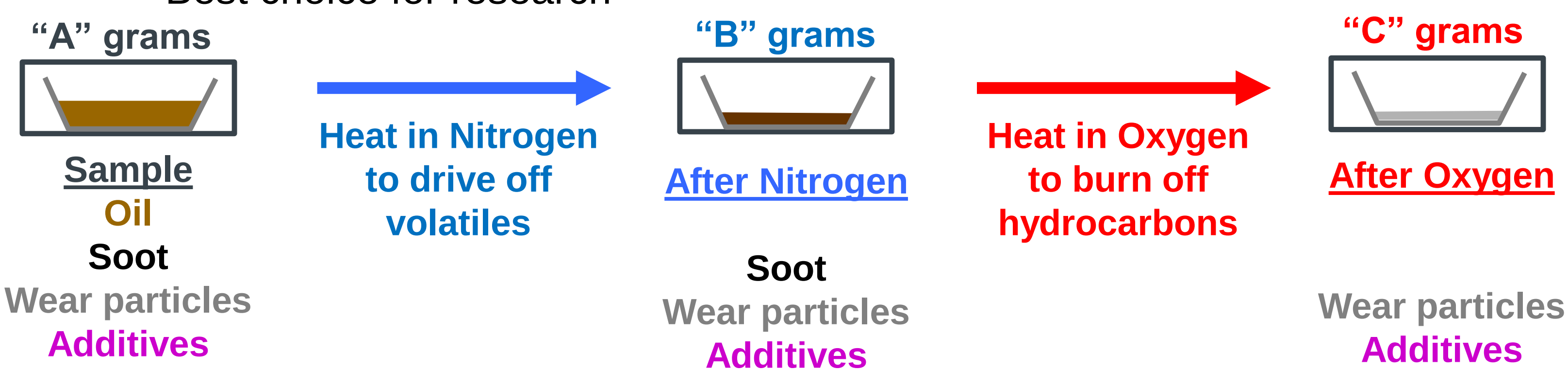
- Water can enter the oil through condensation or coolant leaks
  - Or rain!
- Glycol is often a component of the coolant treatment (anti-freeze)
  - Coolants treatments also have anti-corrosion chemicals
- Important to know the composition of the coolant
- Water and coolant contamination can be detected 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)

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"

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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 μm, but can be changed)
  - Blotter tests – might be useful for field check

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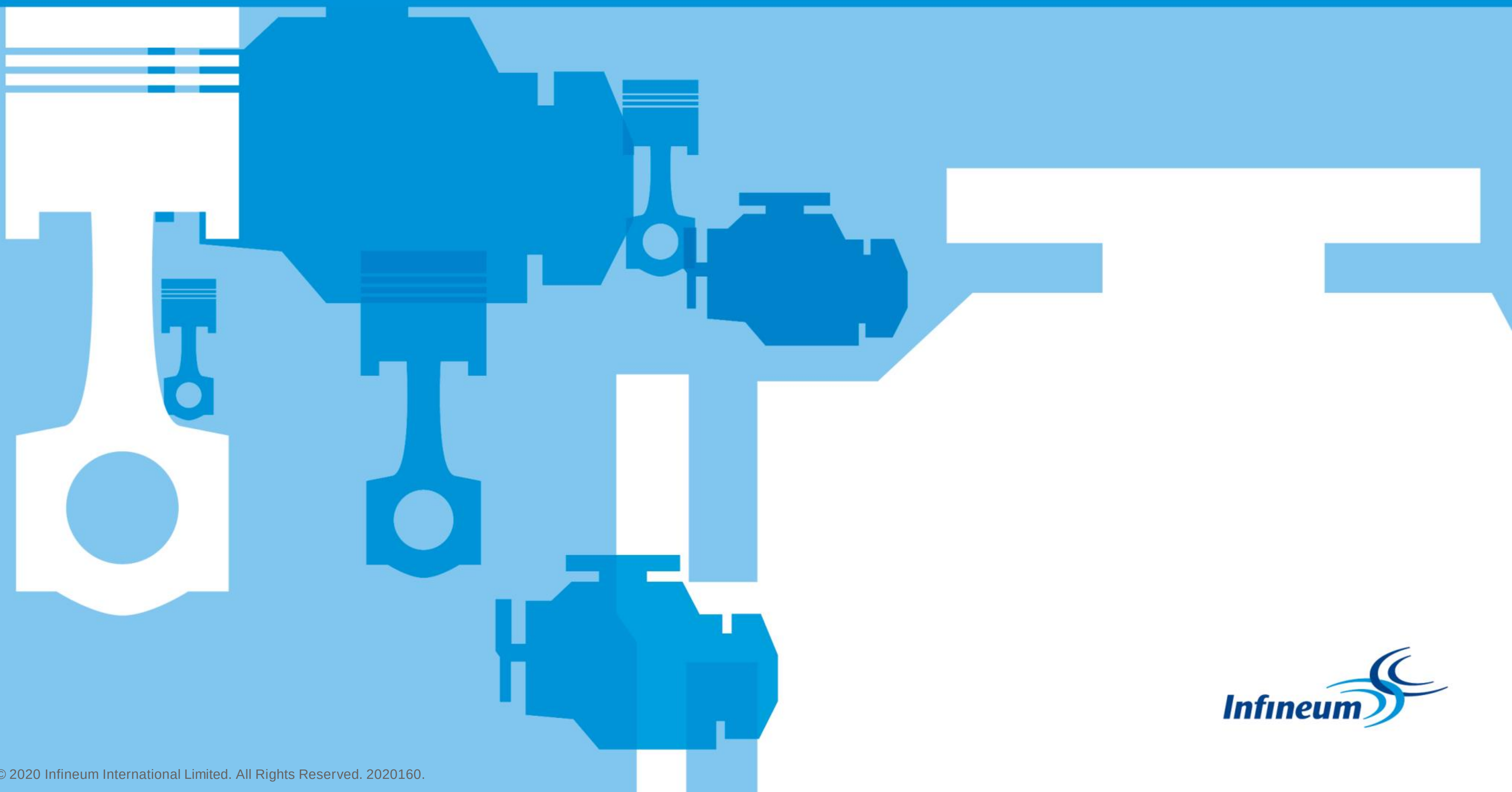
Other properties

- 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:
  - Flash point – Fuel dilution
  - Pour point – Anti-oxidant depletion
  - Aniline point – Measure of aromatic content
  - Bench oxidation tests – Antioxidant remaining
  - Ferrography – Wear mechanisms
  - Color – Supposedly degradation
- Some tests commonly used for industrial, not engine, oils:
  - Rust or corrosion – Anti-corrosion depletion
  - Particle size – Amount and type of wear
  - Remaining Useful Life (RULER) – Anti-oxidant depletion
  - Filterability – Alternate for particles
  - Emulsion or demulsibility – Water separation
  - Foaming – Antifoam depletion

Oil Analysis Reports



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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
- Where to test your own vehicle used oil?

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Sample oil report – header information

- For this hypothetical, ideal example:
  - Samples taken about once *per* week
  - Lab response is two days
  - Engine and oil miles are consistent
  - Last oil changes were at 83,384 and 109,399 miles
  - Unit accumulates about 400 – 450 miles *per* day
  - Consumption is less than ½ unit *per* 1000 miles

Customer:	**Company**
Contact:	**Person**
Unit Number:	**Serial Number**
Equipment:	**Engine Model**
Oil:	**Brand Name**
Grade:	**SAE**

Sample Number	Date Taken	Date Reported	Lab Tracking	Engine Miles/Hrs	Oil Miles/Hrs	Change	Oil Added (units)
7	4 Feb 16	6 Feb 16	CJK56F	103,045	19,661	No	1
8	12 Feb 16	14 Feb 16	CJK57F	106,244	22,860	No	1
9	19 Feb 16	21 Feb 16	CJK58F	109,399	26,014	Yes	2
10	27 Feb 16	29 Feb 16	CJK59F	112,713	3114	No	1

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Sample oil report – physical and chemical

- For this hypothetical, ideal example:
  - Properties follow consistent trend, then “re-set” after oil change
    - Questionable result for Acid Number
  - Appears to be mild oxidation
    - Viscosity, acid, oxidation increase / Base decreases
  - No evidence of water, coolant, or fuel leak
  - Appears to be lower-temperature combustion (Ox/Nit ratio)

Sample Number	Visc 100°C	Acid Number	Base Number	Oxidation	Nitration	Water	Glycol	Fuel	Soot
7	13.6	1.67	8.25	8	3	0.0031	Neg	Neg	0.08
8	13.8	**4.85**	8.01	11	6	0.0044	Neg	Neg	0.06
9	14.1	1.94	7.92	16	7	0.0031	Neg	Neg	0.06
10	13.5	1.21	8.44	1	0	0.0006	Neg	Neg	0.03

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Sample oil report – elements (excerpt)

- For this hypothetical, ideal example:
  - Properties follow consistent trend, then “re-set” after oil change
    - High lead in sample 7: re-check results for sample 6 and before
  - Suggestion of low oil consumption (Ca increase)
    - Questionable calcium for sample 10? Not flagged by lab
  - Appear to be low wear rates (Fe, Al, Cr, Cu, Pb)
  - Evidence of water or coolant leak? (Na would probably be higher)
  - No evidence of air filter leak (Si)

Sample Number	Fe	Al	Cr	Cu	Pb	Mo	Ca	P	Zn	Na	Si
7	7	<1	<1	3	**12**	2	1523	802	884	<1	8
8	7	<1	<1	4	<1	1	1561	807	887	2	8
9	10	<1	<1	3	1	1	1592	753	835	**7**	6
10	<1	<1	<1	<1	<1	<1	1373	781	852	<1	3

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

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