



Used oil analysis

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What can go wrong?

2

Economics

Not
quantitative

3

Critical Factors

Oil Sampling

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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

Oil analysis requires attention to detail

- Sample timing, sample labelling, good lab, reading the reports

A single oil analysis program will not fit all situations

- Depends on equipment, fuel, oil, aspirations, value

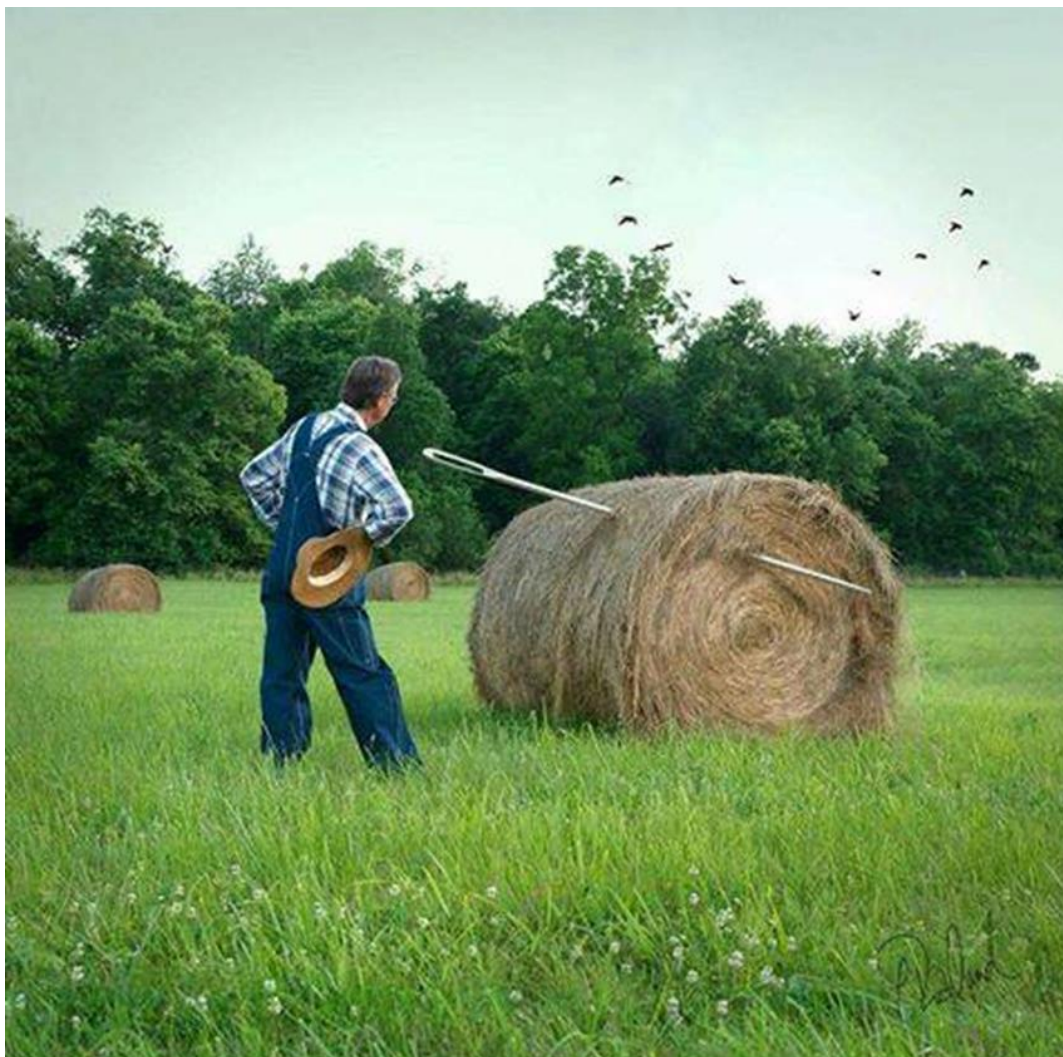
Analysis of trends is the best method

- 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

Finding needle in a haystack



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, & 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)

Oil sampling



Importance of proper sample labeling

A sample is only as good as its label

- Equipment identification
- Oil age
- Mileage or hours
- Sampling date
- Lubricant/oil information

Properties and test methods

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

Radicals -

Oxidation and Nitration
Viscosity increase

Incomplete combustion

Soot and insolubles

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

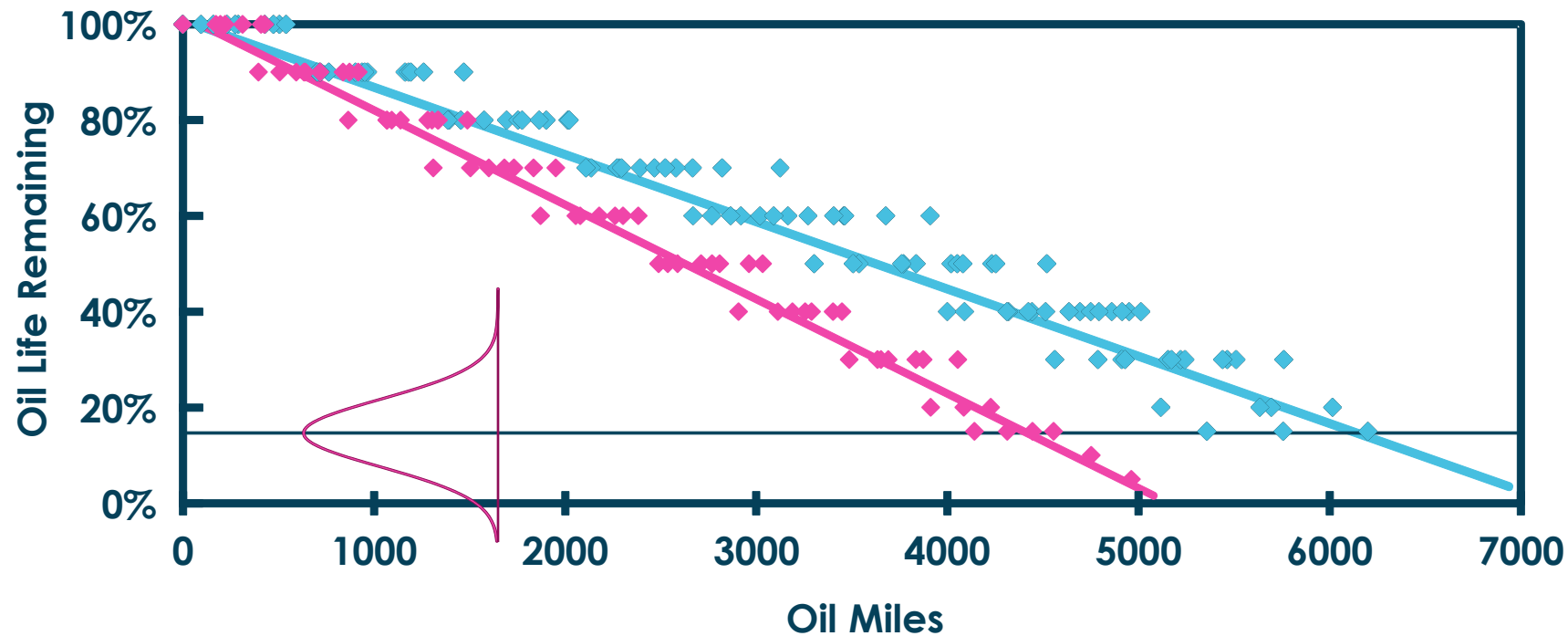
- Scheduled oil changes
- Based on manufacturer recommendation or experience
- Oil analysis at (before) oil change (at least)
- Extra samples during oil drain recommended
- Intermediate samples may detect contamination or engine failure
- “Drain only” samples will only confirm a failure has occurred
- 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

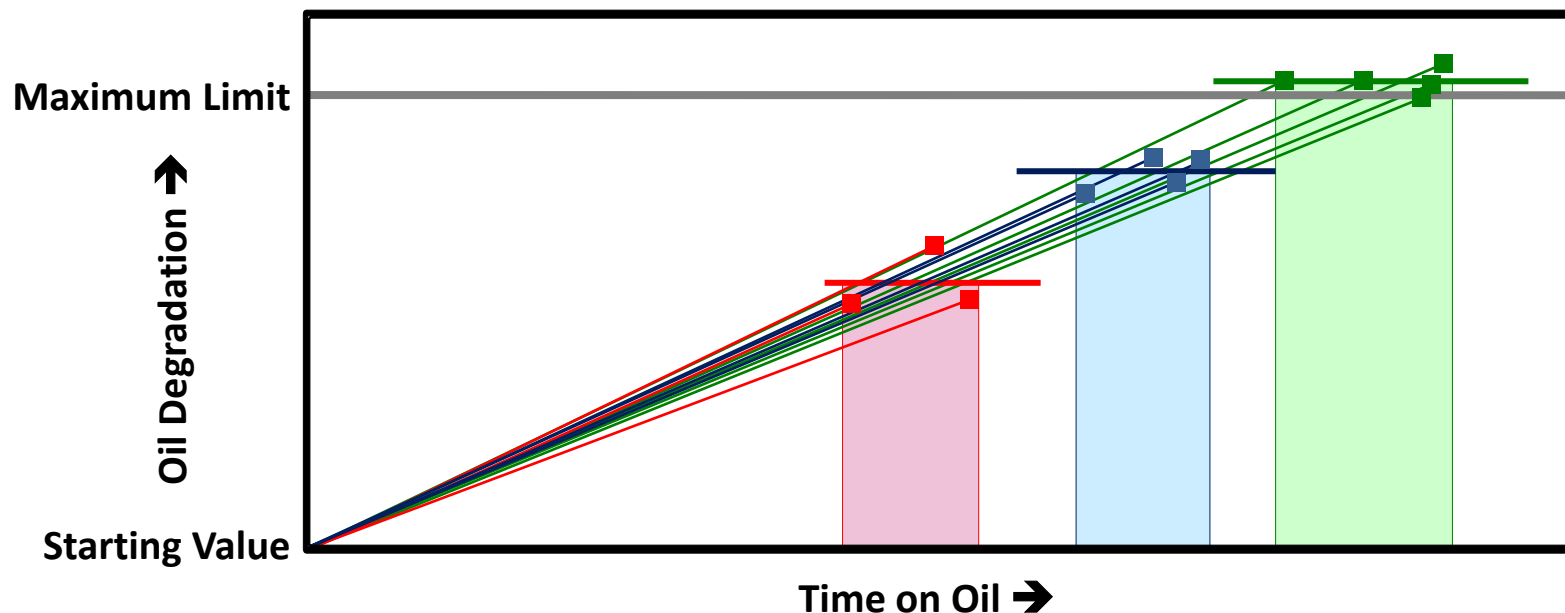
Oil change meter

- 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

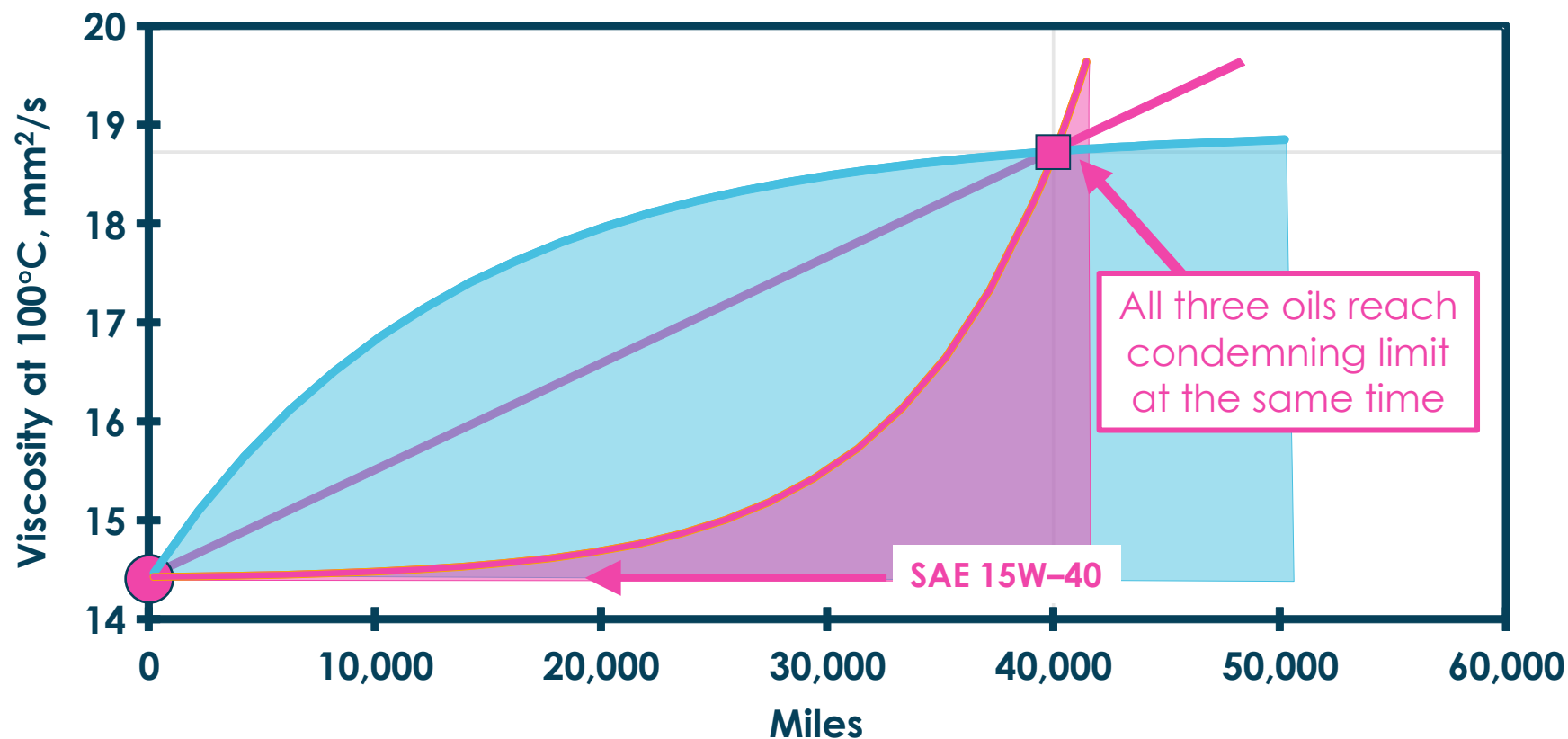
- Oil drains scheduled using manufacturer's guidelines
- Oil drain sample analysis shows remaining oil life
 - *Drain interval increased in small increments*
- **Notice:** Oil performance ("slope") remains the same
 - *Just taking advantage of the performance not previously captured*
- Oil drain extension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



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

Decrease can be caused by:

Cause:

- Shearing of Viscosity Modifier
- Fuel dilution (gasoline, diesel)
- Contamination with improper oil

Differential:

Absence of other signs

Fuel dilution, flash point

Elemental signature

Increase can be caused by:

Cause:

- Oxidation
- Soot content
- Insolubles
- Deposit precursors
- Coolant contamination
- Fuel dilution (marine)
- Contamination with improper oil

Differential:

Infrared, Acid Number

Soot content

Insolubles content

Infrared, Acid Number

Water, glycol, sodium, potassium

Flash point, nickel, vanadium, sodium

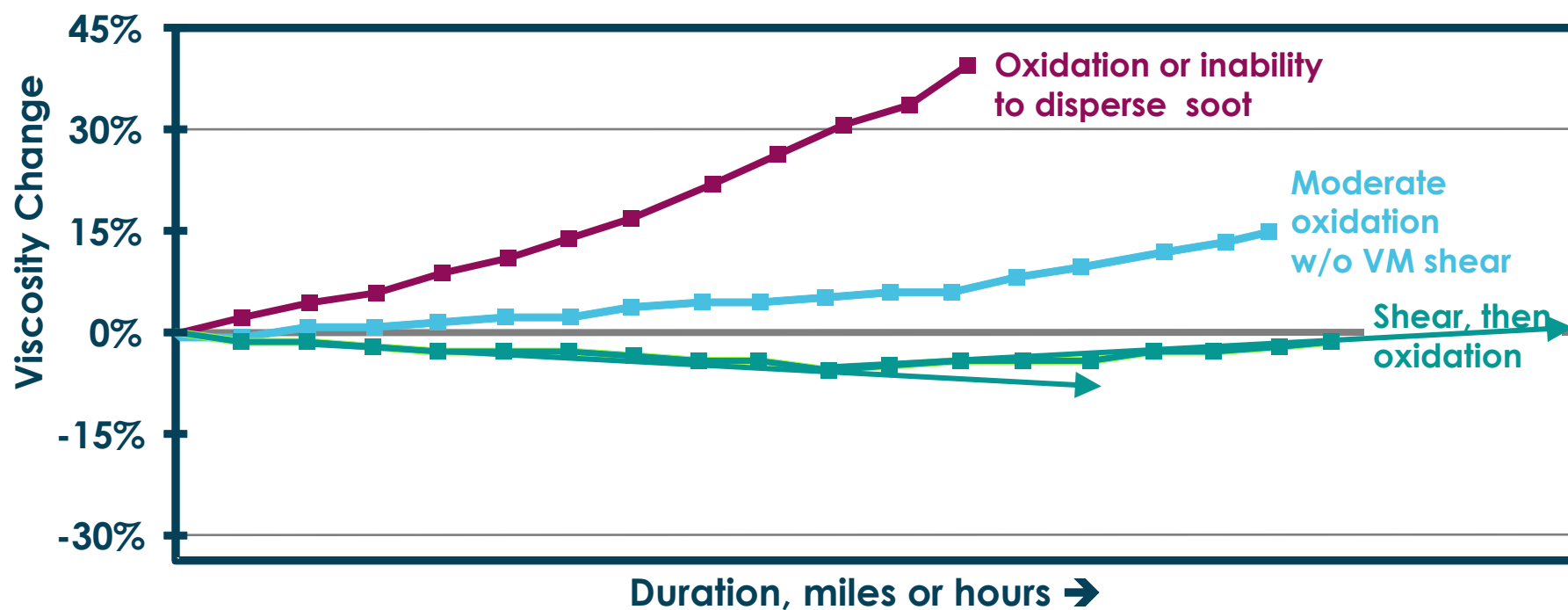
Elemental signature

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



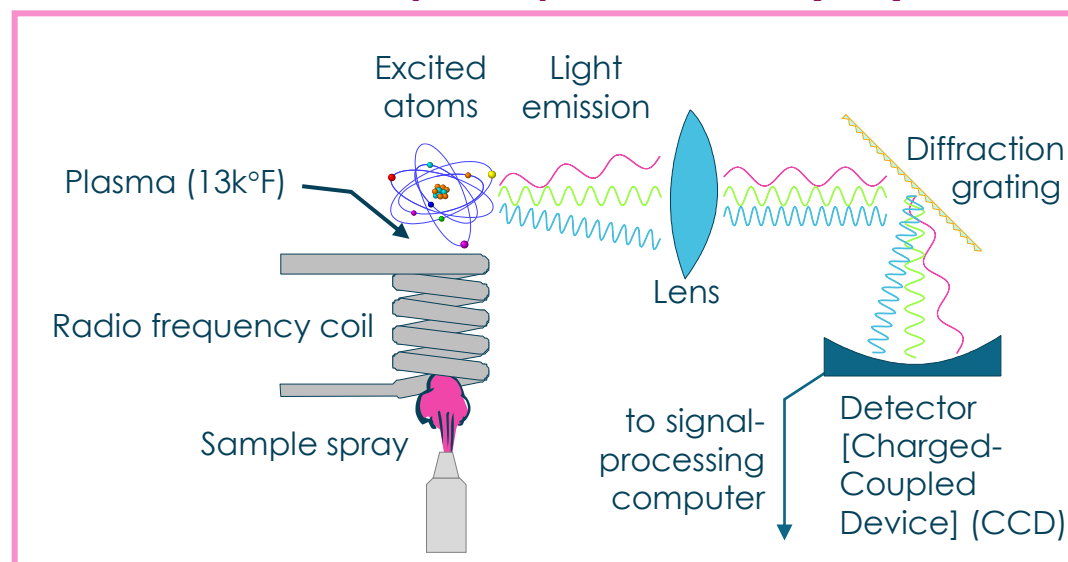
Elemental analysis

Two common methods of Atomic Emission Spectroscopy (AES)

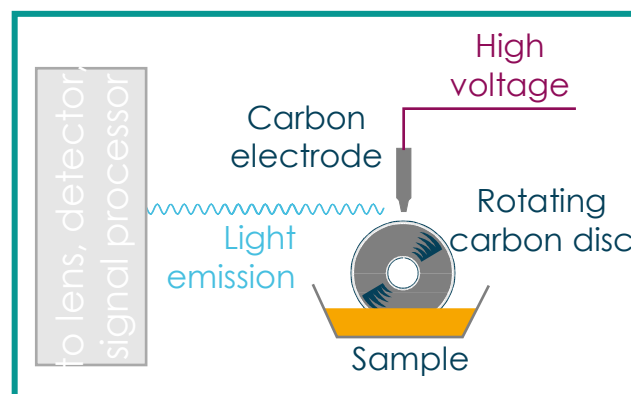
- Excite atoms at high temperature, which emit light of distinct wavelengths
- **Inductively-Coupled Plasma (ICP)**, ASTM D5185
 - Dissolved elements and particles < 3 μm (micrometers)
- **Rotating Disc Electrode (RDE)**, ASTM D6595
 - Dissolved elements and particles < 8 μm (micrometers)

X-ray AES (ASTM D4927)
only suitable for new oils

Inductively-Coupled Plasma (ICP)



Rotating Disc Electrode (RDE)



Elemental analysis

Important to know:

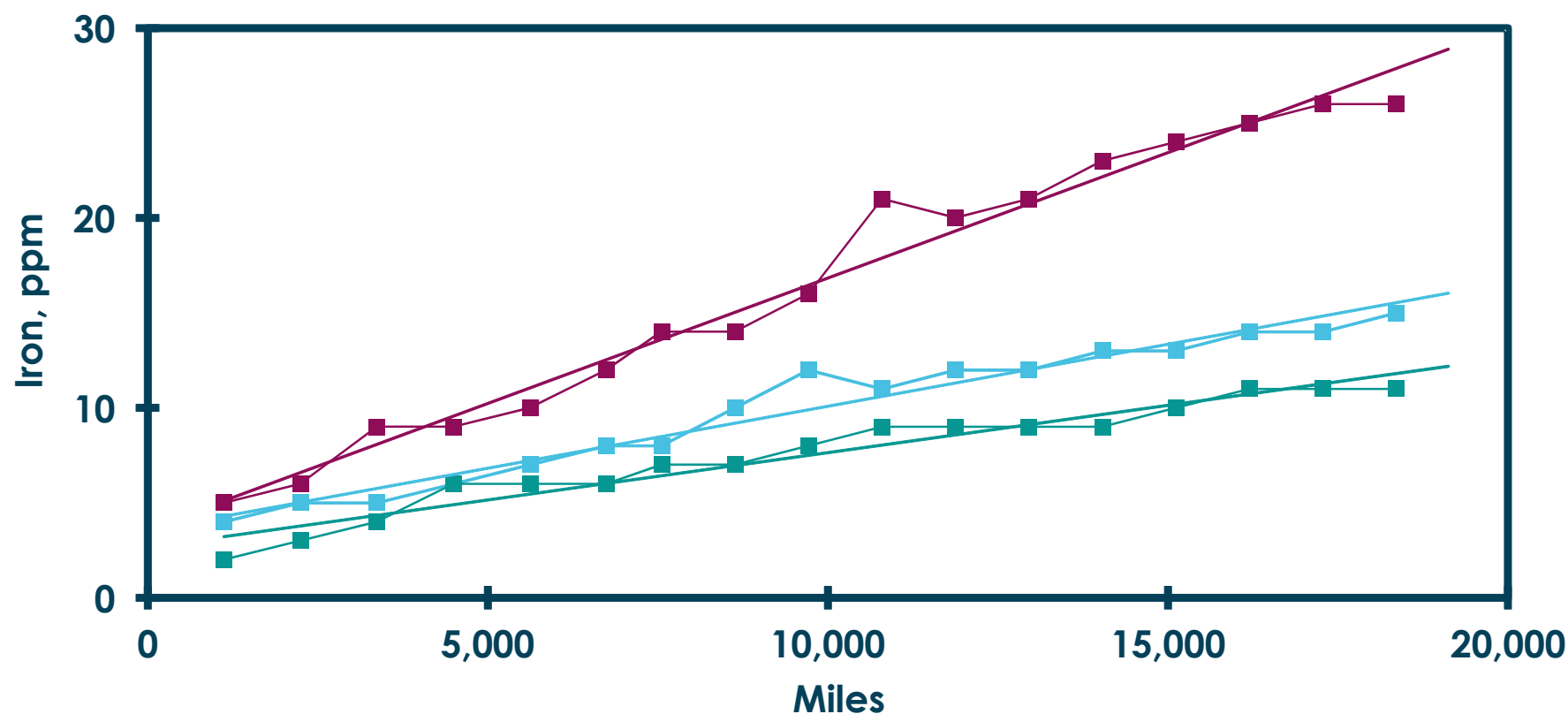
New oil, engine metallurgy, and coolant signatures

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

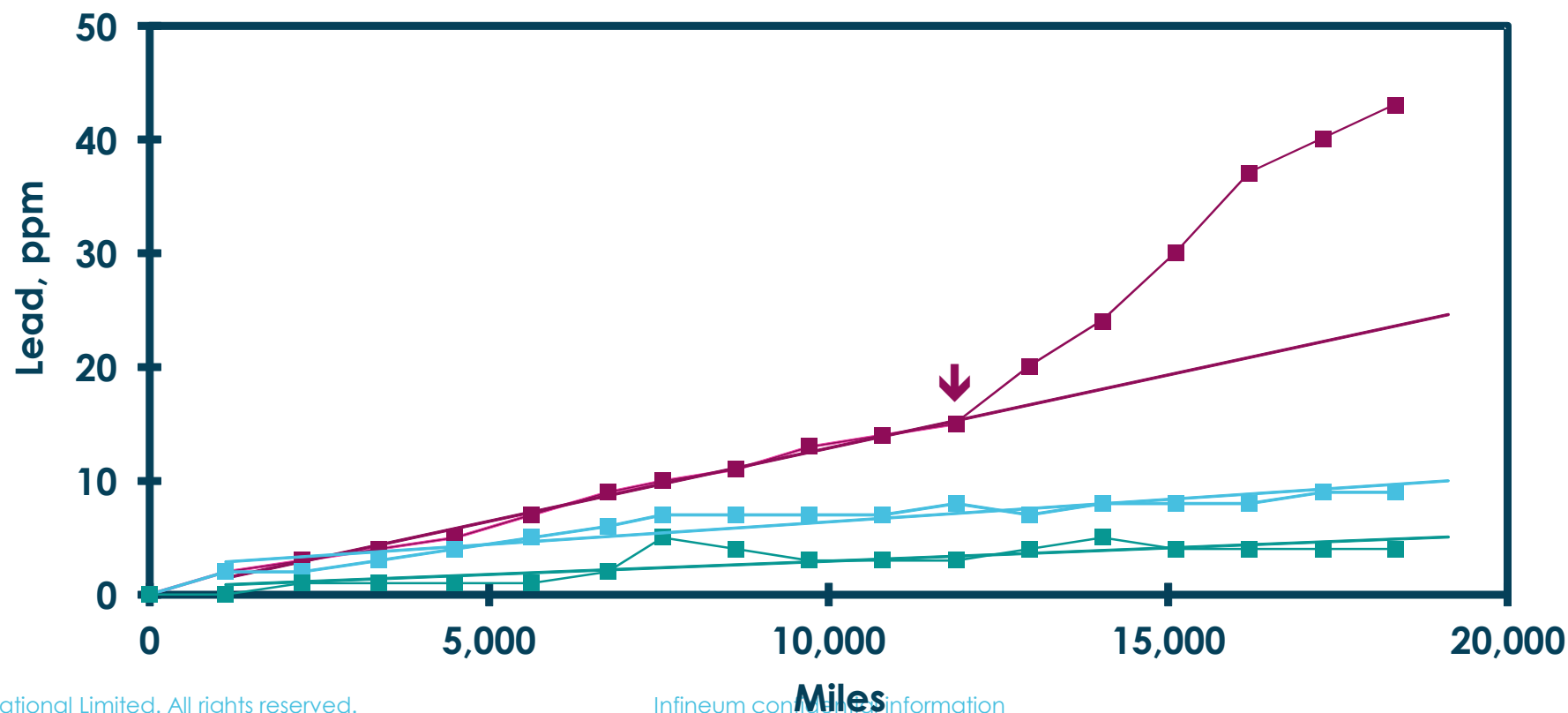
- Probable mechanism is abrasive wear of rings, liners, cams
- Wear rate can be extrapolated to predict engine life



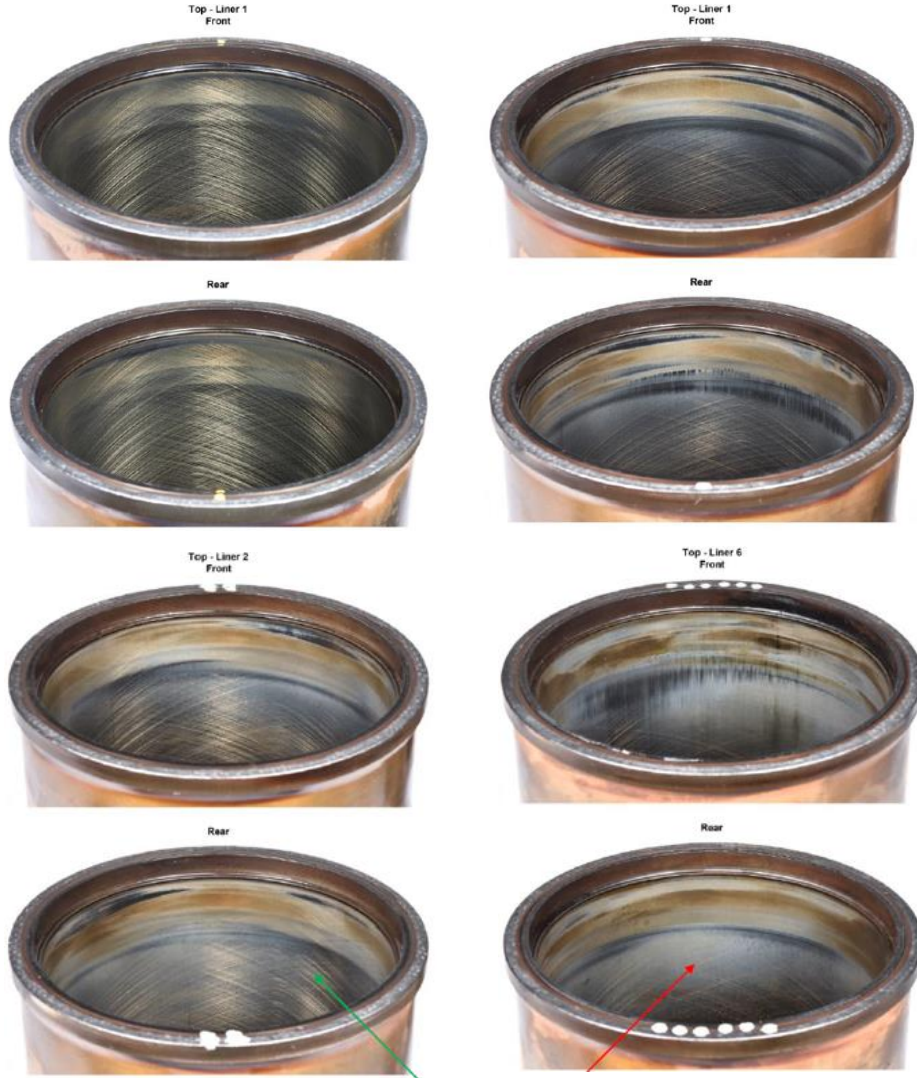
Abnormal wear example – lead

RED oil has higher lead wear rate than **BLUE** or **GREEN**

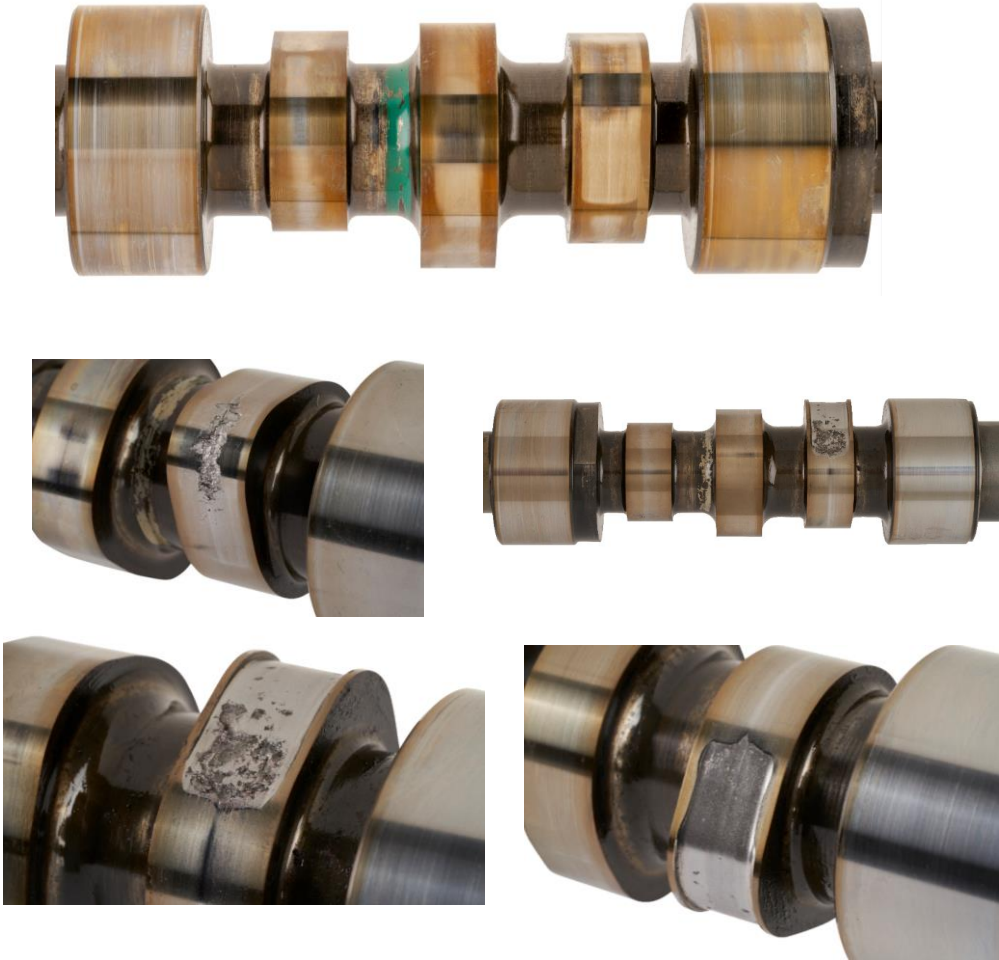
- Particularly troubling is the departure from linear trend
- Probable mechanism is corrosive bearing wear: more investigation →
- Differential diagnosis: Acid Number



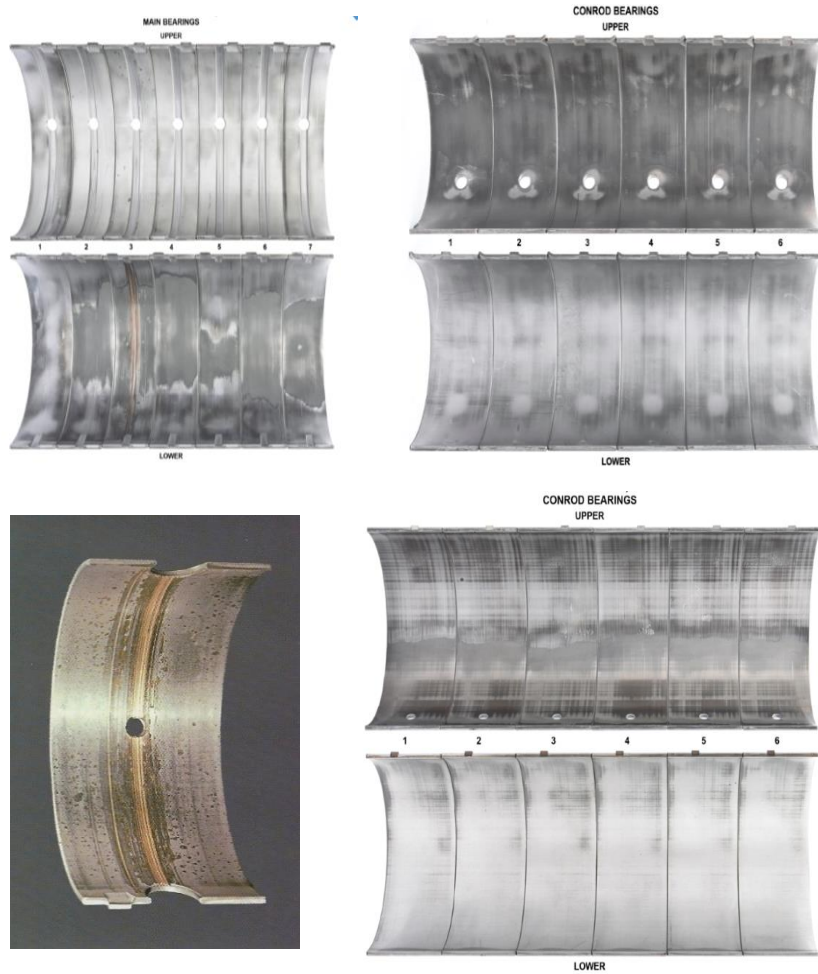
Cylinder wear



Valvetrain wear



Bearing wear



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)

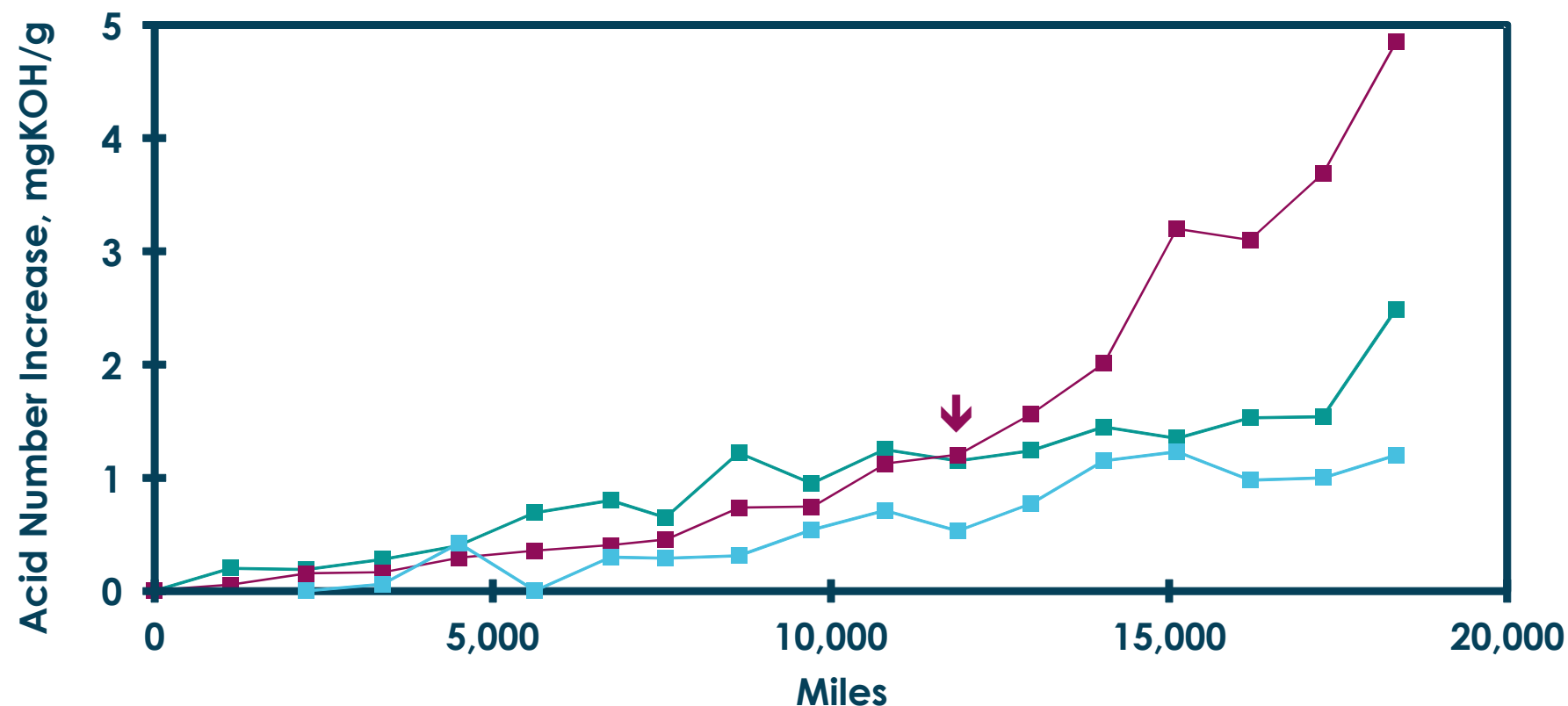
Some additives are acidic by this measurement

- 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

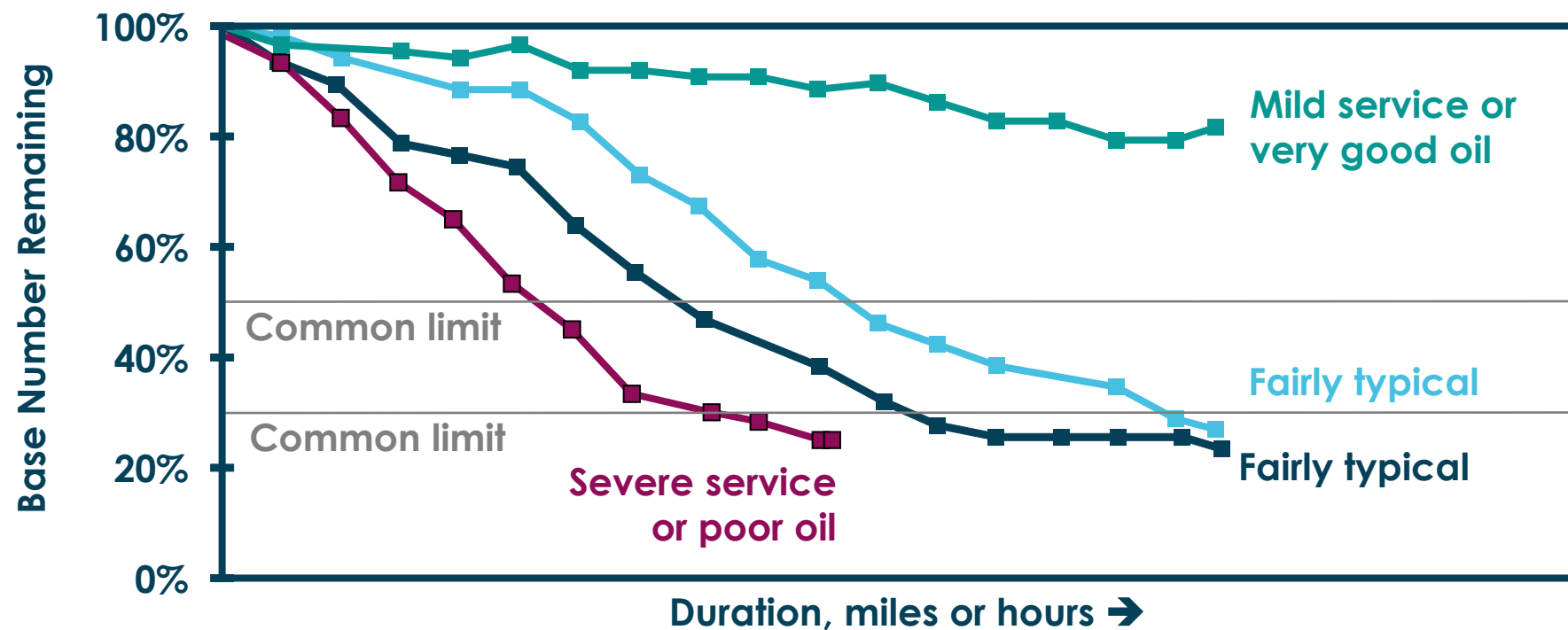
Base number example

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

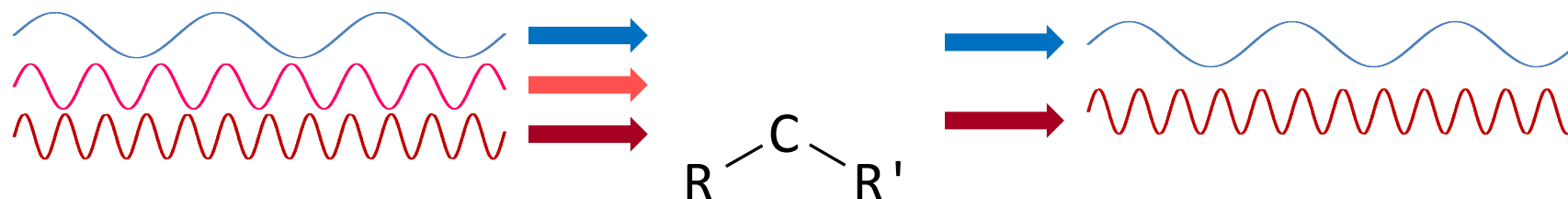
Infrared is light with longer wavelength than visible red

- 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

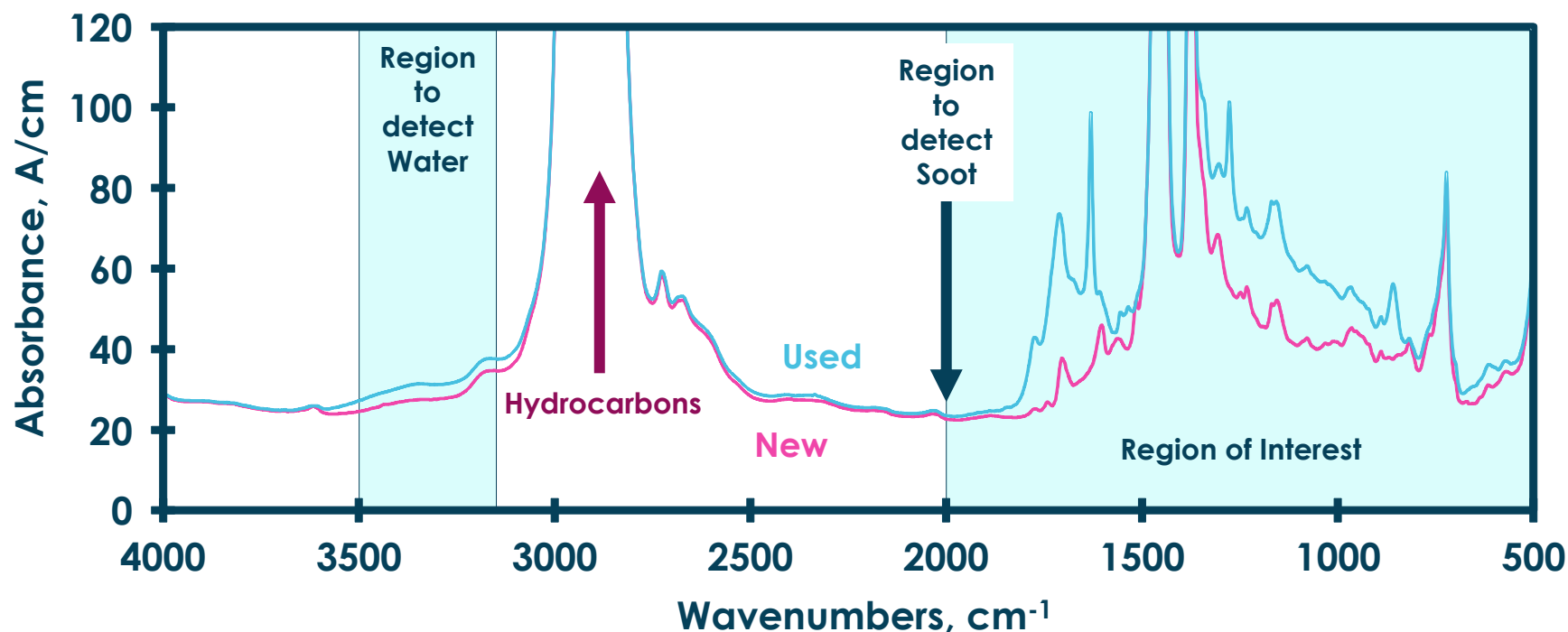
- Somewhat less reliable – but cost-effective – for water, fuel dilution, soot
- Usually good enough for routine condition monitoring



Infrared example – full spectrum

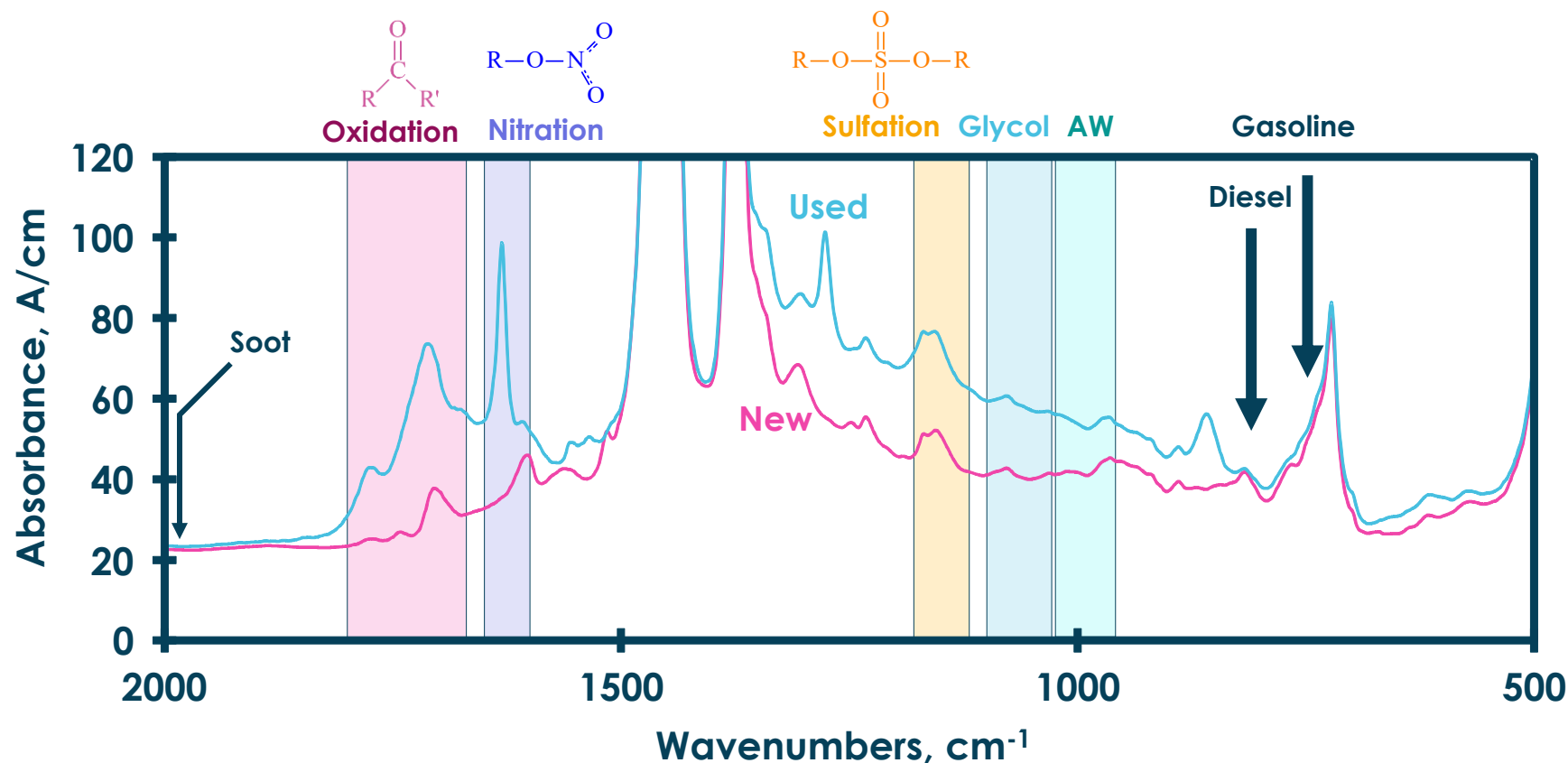
Plot of amount absorbed against wavenumbers (light frequency)

- **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



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 contributors	– Not specific: too many other
Flash Point	– Not usually included in analysis
Gas Chromatography	– Good choice, but expensive
FTIR	– Questionable accuracy Needs calibration for quantitation

Soot

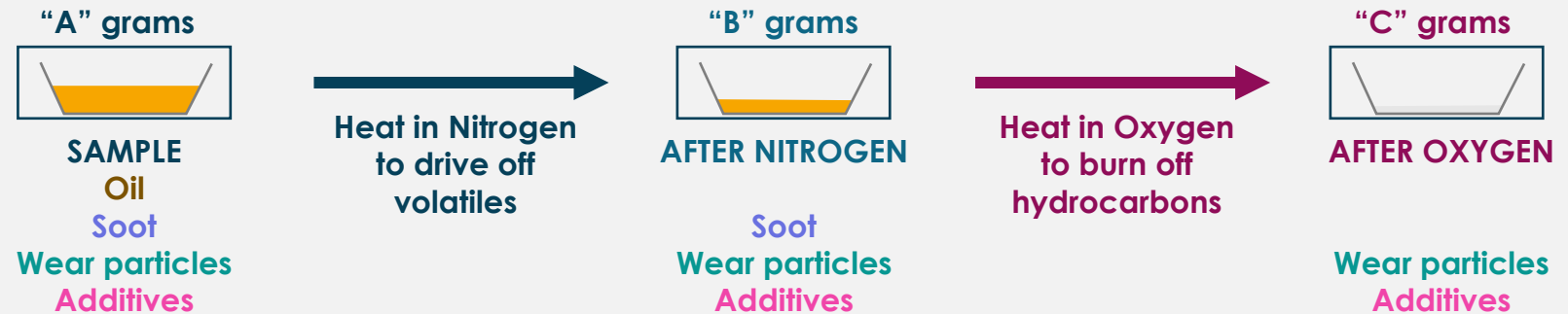
Soot is a by-product of incomplete combustion

- 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

- $\text{Soot} = B - C$
- Best choice for research



FTIR – Fast and convenient

- Adequate for condition monitoring, but requires calibration
- Sometimes called “Wilkes Soot”

Insolubles tests – Not exactly “soot”

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

Summary

1

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

2

Oil analysis must balance:

- Cost of analysis vs. risk and cost of equipment failure

3

Interpretation of oil analysis requires attention to detail:

- There are many training courses available
- Your oil analysis lab is a good place to start

Thank you



Formulating tomorrow together

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