





Greek for Beginners (Part 2) or More Tests and What They Tell Us

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n the last Technical Bulletin we looked at the four tests which every sample receives. We now take a look at tests specific to various sample test classes, and extra tests which are triggered in extraordinary circumstances.

DEBRIS ANALYSIS

In addition to the PQ test performed on every sample, two other debris measurements may be performed on a sample. These involve particle counting and a microscopic particle examination (MPE). Particle counts are only carried out as a standard test on 'Clean Oil' systems while an MPE may be carried out on any sample for a variety of reasons, the most common of which is an abnormally high PQ result.

Particle counting

Samples falling into the Hydraulic, Compressor or Transmission laboratory test classes are treated as 'Clean Oil' systems and receive the particle count test.

The instrument used in the laboratory is an optical particle counter fitted with a laser sensor. The instrument reports particle count data in eight different size ranges per one ml of oil and calculates a cleanliness code based on the international standard ISO 4406.

Cleanliness codes are often quoted as cleanliness specifications for various applications. However, particle counting technology needs to be developed further before such specifications can be rigidly applied with a high degree of certainty. At present, optical (light blockage) and filter blocking instruments dominate the automatic particle count (APC) market and generate results that are seldom in agreement for many different reasons. A new calibration material and a new calibration procedure have now been published by ISO which should go some way to reducing this variability. (A future Wearcheck Technical Bulletin will discuss the implication of these changes to the industry).













While it is difficult to put too much emphasis on cleanliness codes as an absolute measurement, under most circumstances the existing technology provides data of sufficient quality to establish reliable trends for condition monitoring purposes. The technique is also very sensitive and as such is a good early warning system for identifying situations where abnormal wear and/or contamination has started to occur.

Component	5/15 Code		
Automatic transmission	18/15		
Hydraulic system	17/14		
Servo valve	15/12		

Table 1. Examples of typical particle counts for various components.

Where an abnormal result is identified, or a particle count cannot be performed for any reason (eg. water contamination, excessive viscosity, etc.) an MPE is undertaken.

Microscopic particle examination

An MPE is a microscopic exami-

nation of a 5-micron filter patch through which a certain amount of the sample has been filtered. The patches are graded according to the size and density of contamination particles by the diagnosticians.

The grading takes the form of a four-digit code, and is used for diagnostic reference purposes. Each digit can have the value of 1, 2, 3 or 4. The value of the digit represents the density of particles: a 1 is a light or negligible concentration, and a 4 is a very heavy concentration. The first digit represents particles of very small size, the second digit represents slightly larger particles, and so on.

By way of example, an MPE of 2111 represents a light concentration of fine particles, a 4111 represents a heavy concentration of fine particles, and a 1112 would represent a low concentration of large particles. The last example is somewhat uncommon and would be reported if a large chunk of metal or a pebble were to be found in the bottle.

MPE's are performed depending on the results of other tests, or by request from the diagnostician.



Particle counting is an early warning system for abnormal wear.

Figure 1. An MPE rated 2111

Figure 2. An MPE rated 4111



Infrared analysis provides data on lubricant condition. The MPE code does not relate what type of contamination was found, i.e. whether it was wear metal, dirt or some other substance. For that information, the diagnosis on the report needs to be consulted.

Interpretation of the MPE varies according to the type and manufacture of equipment, and also according to the operating conditions of the machinery. Generally, clean oil systems such as hydraulics will be judged more harshly than non-filtered systems. A 2111 on a hydraulic system will generate an actionable report, whilst a 4211 on a gear-andchain drive might well be considered normal. The interpretation of the MPE is qualitative rather than quantitative.

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Infrared analysis is the second type of spectroscopy utilised by Wearcheck and, unlike ICP spectrometry, provides data on lubricant condition. The technique is most successfully applied to used engine oils as it measures several useful degradation parameters, most of which are related directly or indirectly to combustion by-products. The technique is also able to detect the presence of water and can also be used on occasions to identify oil base stocks.

Whilst the ICP spectroscopy measures emissions of radiation of specific wavelength in the visible and ultraviolet regions of the electromagnetic spectrum, infrared analysis involves the measurement of absorption of specific wavelengths of radiation in the infrared region. The various degradation by-products and contaminants found in the oil all cause their own characteristic absorptions in specific regions of the infrared spectrum. The higher the level of degradation or contamination in the sample, the higher the degree of absorption in the characteristic region.

A plot of absorbance versus wavelength is generated during the analysis of an oil sample and is called the 'Infrared Spectrum' of the sample. This spectrum is subsequently 'analysed' by specialised oil analysis software that yields measurements for soot, oxidation, sulphates, nitrates and water. (See Figure 3 below.)

Soot

The soot index is a linear measurement between 0 and 400 and measures the extent to which the oil has become contaminated by fuel soot, an unwanted by-product of combustion. The measurement is only really applicable to diesel engines as the soot measurement on petrol engines is expected to be zero. In diesel engines, excessive amounts of soot can be generated by overfueling, incorrect combustion temperatures, low operating revs, restricted intake and exhaust systems, and faulty turbochargers.

Dispersant additives are added to lubricating oils to keep soot in suspension, but there is a limit to how much soot a lubricant can carry. When this is exceeded, sludge deposits start building up and can cause harm to the engine. The effects of severe sludging manifest themselves as an increasing oil viscosity, usually occurring rapidly to the point where the oil can no longer be pumped and engine failure ensues.

The interpretation of the severity of the soot index measurement should take into account the soot readings on previous samples from the engine and also the magnitude of the change in the oil's viscosity. It should also be noted that high soot loading can negatively affect the accuracy of all other infrared measurements.

Oxidation

As an oil is progressively oxidised,



Figure 3. An example of an FTIR spectrum

its ability to lubricate is diminished and, in cases of severe oxidation, noticeable changes occur in the oil: it becomes darker and emits odour; varnishes, lacquers and resins are formed; and in the advanced stages viscosity increases, usually rapidly. Fortunately the chemical reaction between oxygen and lubricant molecules at room temperature is very slow and oxidative degradation is not an issue under these conditions. The situation changes however when reaction conditions are altered to favour a more rapid reaction rate.

Engine lubricants have to operate in particularly hostile environments and have to be formulated with these operational parameters in mind. Many conditions promoting accelerated oxidation co-exist in an engine, such as high temperatures, high pressures, a good air supply, agitation, the presence of metal catalysts and thin film exposure.

The most significant of these conditions is the operating temperature, as it has been determined that for every 10°C increase in temperature, the rate of oxidation doubles. Because of the sensitivity of an oil to oxidation at high temperature, the most common deduction to be made from high oxidation measurements, assuming the normal service life of the oil has not been exceeded, is overheating. Overheating is generally also accompanied by increased wear (lead, copper, tin and iron) and an increase in viscosity.

Sometimes the overheating process leads to the evaporation of volatile fractions in the oil, and regular top-up is necessitated. In this case, the sump oil will exhibit increased additive levels (concentration of non-volatile components) and an increased viscosity as a direct result of light end loss. As this 'lost' oil is replaced with fresh oil, the anti-oxidants are replaced, and often oxidation is not evident. This is illustrated in the set of results at the bottom of the page, with sample 3 being the most recent and the one in which the overheating is evident.

Sulphation

Oxides of sulphur and water are combustion by-products of diesel fuel that readily combine to form sulphurbased acids. The bulk of these corrosive acids is removed as part of the engine's exhaust, but some remain and escape into the engine cavity in blow-by gas or proceed to attack the thin oil films providing lubrication for piston rings and cylinder liners, where they are neutralised by additives in the oil.

At normal operating temperatures, acids remain in the gaseous state in the blow-by gas with minimal contact with reactive surfaces. However, when an engine experiences lower than normal operating temperatures

Sample	Mg	Ca	Zn	Р	в	S	Vis@40	Ox
1	549	3627	1136	967	5	10838	94.7	15
2	524	3295	1086	934	4	10708	92.5	13
3	667	4516	1431	1222	7	11112	129.7	17

Many conditions promoting accelerated oxidation co-exist in an engine. Trend analysis is the best indication of the oil's and the machine's wellbeing. (such as at shut down, just after startup or when a faulty cooling system results in continuous overcooling) the acids condense and come in contact with the oil in the sump and the oil films on exposed metal surfaces. This places an extra burden on the lubricant as it has to neutralise more acid than would be expected during normal operation.

The sulphate index from infrared analysis is a measurement of the amount of sulphur-based acids that have reacted with the oil and reflect the amount of sulphation that has taken place. If fuel sulphur levels remain constant the sulphate index would be expected to increase continuously with use until the oil reaches the end of its useful service life, for which the sulphation level or sulphate index is an important determinant.

Nitration

As with sulphation, nitration is the reaction of the oil with combustion by-products of nitrogen. These reactions tend to become more pronounced at higher temperatures hence increased nitration is often an indication of increased blow-by, as the hot combustion gases react with the oil. Nitration is rarely commented on because other problems, such as high top-end wear associated with blow-by, will manifest themselves first.

TOTAL ACID NUMBER (TAN)

The measurement of TAN involves a colourimetric titration where the total acid content of 2 grams of oil dissolved in a mixed solvent is completely neutralised by the gradual addition of an alcoholic solution of potassium hydroxide (KOH). The end point is detected by the use of a chemical indicator that changes colour as soon as the acid is completely neutralised.

The TAN test is at present primarily performed on compressor, turbine, and some industrial gearbox and hydraulic samples, and is used to quantify the acid build-up in these oils. An increased TAN is usually a result of overheating, or of overextended oil service.

Components within refrigeration systems are particularly susceptible to acid attack. This can occur when air containing water vapour is able to enter the system, or alternatively when the system is subjected to excessive heat, and the drier releases retained water. When this happens, acids created by the reaction of the air, water, refrigerant and oil cause iron components in the system to become plated with copper. Bearing failure due to copper plating can then occur. In refrigerant systems the acid content of the oil, moisture content and copper level need to be regularly monitored to be forewarned of incipient problems.

TAN limits vary enormously and depend both on OEM specifications and on the oil itself. In some cases, a TAN exceeding 0.05 is unacceptable; in others a TAN of up to 2.00 remains acceptable. As with all other readings, trend analysis is the best indication of the machine's and the oil's wellbeing.

TOTAL BASE NUMBER (TBN)

At present the measurement of TBN involves a complex potentiometric titration where the total alkaline reserve of 1 gram of oil dissolved in a mixed solvent is reacted with the gradual addition of a known excess of an acid solution containing perchloric acid. The reaction is monitored using a reference and a measuring electrode, and a graph of voltage (mV) vs acid added (ml) is plotted. The end point is detected from a point of inflection on the graph or, in the case of badly degraded oils, from a predetermined millivolt reading.

This test is only applicable to engine oil samples as these lubricants are deliberately formulated to have a reserve alkalinity that enables them to neutralise the corrosive acidic byproducts of the combustion process. The TBN of an oil is a direct measurement of its alkaline reserve.

Every engine oil has a starting TBN which reduces gradually during the course of use. Typical starting values for most diesel engine oils are between 10 and 14, although marine engines burning heavy fuel oil need a much higher TBN, possibly as high as 80, to deal with harsh combustion conditions from fuels containing a high concentration of sulphur. A general rule of thumb is that the time to discard the oil has arrived when the TBN drops below half its starting value.

While it may seem logical to assume that oils formulated to have a high TBN would be most desirable, this is not the case and some engines may suffer burnt valves if such an oil is used. This results from the high ash content of the oil and high valve temperatures causing fusion of the valve seats. Using a lubricant specifically formulated for diesel combustion in a petrol engine could also prove detrimental, and highlights the fact that it is important that equipment manufacturers' lubricant specifications be adhered to.

TBN measurements are only performed on samples flagged for analysis from infrared results. A TBN can be reasonably accurately predicted from the infrared data and, where this prediction is below the specified limit, a TBN test is requested automatically to confirm the degree of degradation evident in the infrared data. All samples having a predicted TBN exceeding a 'safe' limit are reported as having a TBN of '+6' while the actual result is reported for samples selected for the test.

TBN and TAN units of measurement

The units of TBN and TAN can be somewhat confusing. Although they are different tests, the results are both expressed in the same units: mg KOH/g (milligrams of potassium hydroxide per gram of oil).

The TAN of an oil is defined as the number of milligrams of KOH needed to neutralise the acid constituents in 1 gram of the oil.

The TBN of an oil is the number of milligrams of KOH needed to neutralise the acid needed to neutralise the basic constituents in 1 gram of the oil.

GAS CHROMATOGRAPHY (GC) TEST FOR FUEL DILUTION

Gas chromatography is a separation technique used to analyse used engine oils for evidence of fuel dilution. The technique as applied to fuel dilution measurements is used to separate and measure two volatile fractions of specified boiling ranges from used engine oil samples. The first volatile fraction of interest is that having a boiling range similar to that specified for petrol, while the second fraction has a boiling range similar to diesel. The instrument is calibrated, and measurements are reported as a percentage contamination by mass.

The fuel dilution test is only per-

The TBN of an oil is a direct measurement of its alkaline reserve. Wearcheck reacts early to fuel dilution.

formed where a significant drop in sample viscosity is measured, or where the test is specifically requested by a diagnostician. It is important that the oil brand and grade are correctly described if problematic samples are to be detected, and much care is needed in interpreting results as many factors can influence their interpretation. Fuels are complex mixtures of organic compounds and are classified into products based largely on distillation ranges rather than specific chemical data. There are also significant overlaps between various product specifications making it impossible to accurately separate and quantify fuel mixtures.

The presence of paraffin in an oil sample, for example, will not be detected as such by the GC but it will artificially inflate both the diesel and the petrol measurements. A light base oil with a light end fraction overlapping with the heavy end of diesel detection range will also yield an inflated result.

Apart from the limitations of the test, another issue with fuel dilution measurement is determining the severity of the problem as far as the engine is concerned. A fuel pump, slightly incorrectly calibrated, may be delivering a small amount of extra fuel to each cylinder to give, say, a fuel test result of 5%. This amount is unlikely to cause damage. But 5% fuel dilution of the oil caused by a single faulty injector could do serious damage to its cylinder. For this reason, Wearcheck tends to react early to fuel dilution, as the origin of the overfueling cannot be detected. As a norm, fuel dilution readings exceeding 4% will be commented on and in some cases even lower readings will provoke reaction, depending on past history, the vehicle involved and its type of operation.

Another commonly seen occurrence is diesel contamination of a petrol engine oil. This seems to occur more in rural areas or small towns than in the cities. The source of this is diesel contamination of the petrol. The diesel is not burnt during combustion, and it accumulates in the sump. Drivers usually become aware of the problem when the oil level in the sump rises, sometimes to the point of overflowing the dipstick.

This concludes the explanation and interpretation of the standard tests performed at Wearcheck. There are more specialised tests, but these are performed by special request.

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Produced by the Wearcheck Division of Set Point Technology

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