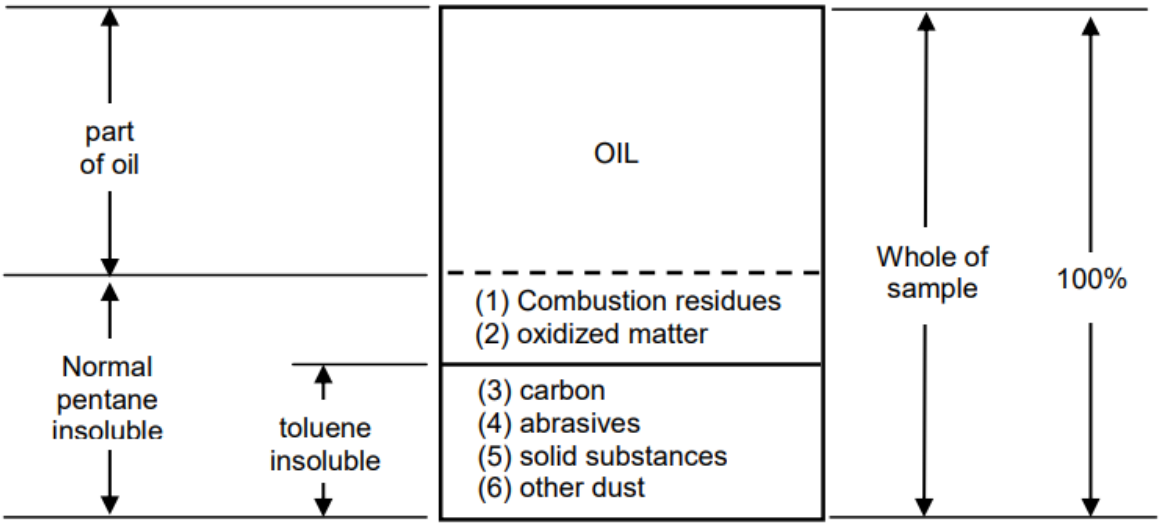
GulfSea UOA Analytica

Soot / Insolubles

Incomplete combustion of hydrocarbons such as ash, carbon and partially oxidised fuel typically results in leftovers which are generally classified as ***Soot***. Formed in fuel-rich but cool regions of the combustion chamber, soot is deposited onto cylinder walls, where it is scraped into the engine oil sump by the piston rings.

Insolubles are impurities derived from degraded additives such as sulphates from combustion of fuel oil's sulphur and reaction with BN additives and other inorganic components such as wear debris or densely carbonized matter generated from high-temperature thermal events (hard contaminants).



Insolubles % gives an indication of the efficiency of the purification or filtration of oil. Common test methods make use of solvents to lower viscosity and then subject the sample to centrifuge, thus, separating the total insolubles through a membrane filter.

Soot % indicates problems with combustion or blow-by. It is however quite difficult to quantify the amount of soot in used oil samples.

Testing for Soot:
Four tests are usually used to measure the soot of lubricants, some with direct results, some indirect. Each method has its pros and cons.

1. The blotter test allows a visual evaluation of the residual level of dispersant and soot. This type of evaluation remains subjective and is limited to qualitative results.
2. The total insoluble test is a qualitative indicator of soot. This test requires solvents, a centrifuge instrument and an oven. It is slow and complex especially when other by-products of combustion are also present in the oil.

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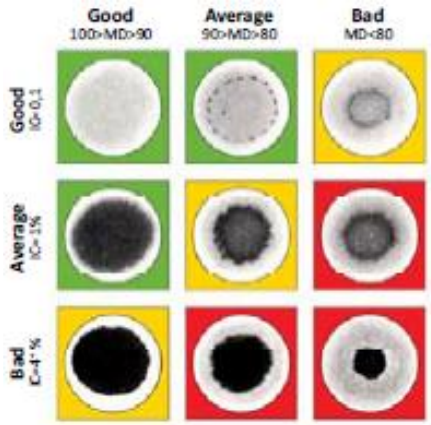
3. Thermogravimetric analysis (TGA) is a thermal analysis technique method which measures changes in mass in relation with temperature. This test is simple to perform and provides a direct reading of the percentage of soot. It is the only direct method suitable for measuring soot's carbon elements and it only requires a few drops of oil on a reading surface.
4. Infrared analysis is a simple, quick and precise testing method, especially for low soot concentrations. Results are comparable with TGA's. It is widely used by the JOINT OIL ANALYSIS PROGRAM (JOAP) developed by the US army for production and routine testing.

Understanding Each Test

Standard Test Method for Measuring the Merit of Dispersancy of In-Service Engine Oils with Blotter Spot Method - ASTM D7899 - 13



Unlike measuring soot concentration where numerous options exist, measuring dispersancy performance is a real challenge. There are two ways to approach the issue: one can attempt to measure the concentration of the dispersant additive itself, or one can measure the dispersancy performance of the oil. For routine used oil analysis, the latter offers a much more promising solution.

While the blotter test offers limited value measuring soot concentration, it provides an excellent assessment of the lubricant's dispersancy performance. An oil that is properly dispersing soot and other insolubles produces an evenly graduated blotter (see Figure 6A, next page).

A blotter indicating a high soot load, but even graduation, suggests the oil is still fit for service, but should be watched closely for degradation (see Figure 6B next page). When dispersancy begins to fail, the insolubles begin to form a dense ring on the exterior of the absorbing oil drop as seen in Figure 6C (next page).

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Figure 6D (below) indicates the characteristic dense black dot and sharp periphery that forms when the oil completely loses dispersancy performance. From a maintenance perspective, when the ring begins to form around on the exterior of the oil blotter, it is time to look at scheduling a drain. If the situation is not rectified in a timely manner, it may become problematic if the undispersed portion of soot that has deposited upon surfaces cannot be easily removed (usually by an oil change). Several changes will then need to be made at frequent intervals to effectively scour the engine clean.

If dispersancy performance degrades at an unusually rapid pace, a more extensive review of combustion and ring performance should be undertaken.

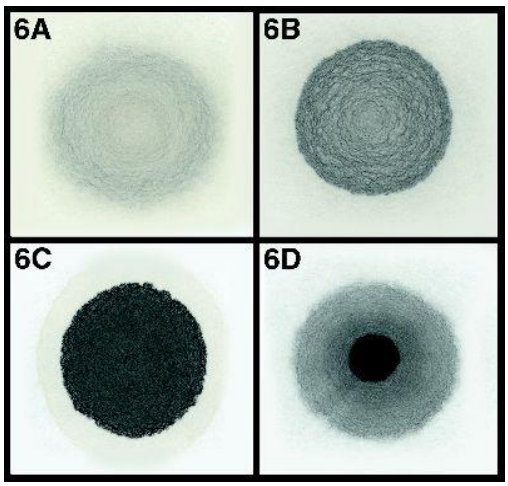

Figure 6 – Blotter A shows good dispersancy, blotter B shows high soot load with marginal dispersancy, blotter C shows high soot load and failing dispersancy, blotter D shows failed dispersancy.

Oil analysis programs will have to increasingly combine monitoring of both soot load and dispersancy. However, clear limits for soot have not been established; hence, lubricant manufacturers will need to inform operators of the acceptable condemning limit for soot load.

Pentane and Toluene Coagulate Insolubles Test - ASTM D893

Basically, this test separates insolubles from the oil after it has been mixed with solvents. First, a pentane solvent is mixed in with the oil to separate solids and oxidation products from the oil by lowering the viscosity.

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The mixture is then centrifuged to separate the insoluble material and the insoluble material is then measured to determine the percentage of insolubles. The test is then repeated using toluene instead of pentane.

The toluene dissolves organic oxides, but not soot. The mixture is centrifuged again and the insoluble material is weighed. The difference between the two weights (pentane and toluene) is the estimated soot content in the sample.

Soot Percent by Thermal Gravimetric Analysis (TGA) - ASTM D5967

The TGA method involves placing the sample in an oven, where it is weighed over specific increments of temperature. An inert nitrogen purge gas is used over the sample to enable the oil to evaporate without other reactions (oxidation) taking place.

When the weight stabilizes and remains stable for a certain duration, this signifies that all the oil has evaporated and only insolubles remain. At this point, oxygen, rather than nitrogen, is introduced, which allows all the carbonaceous material (mainly soot) to combust, leaving the other metal insolubles in the sample.

The soot percentage is calculated by measuring the difference between the weight of the sample before oxygen was introduced and the weight of the sample after it is stabilized and all the soot has been removed.

Pentane Insolubles by Membrane Filtration - ASTM D4055

Pentane insolubles above 0.8 µm in size may cause increased wear which can lead to premature equipment failure in critical applications.

This test method determines the quantity of insolubles exceeding 0.8 µm in new and used lubricating oils; it uses a pentane solvent and the oil is then passed through a submicron membrane. As the sample passes through the fine mesh patch, the insoluble material remains on the upstream surface of the patch while the remaining sample passes through.

The insolubles are then dried and weighed, and the insolubles percentage is then calculated.

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