



Exceeding Expectations

Distillate Fuel Stability

As Father Time brings on the aging process in humans, he has his affect on oil as well! Stability testing is the test which checks the aging and product degradation process in distillates

Below describes in detail the various methods used.

Polymerization and oxidation can occur as fuels age, forming gums and insoluble or non-volatile residues. These materials can lead to fuel system failures in vehicles or negatively affect combustion performance. The presence of cracked fuels compounds the problem. The addition of fuel stability improvers can slow or prevent deterioration in middle distillate fuels or diesel. These additives block the oxidation or polymerization reactions of cracked hydrocarbon molecules.

These are the most common methods that AmSpec uses to determine distillate stability:

D2274 – Oxidation Stability of Distillate Fuel Oil (Accelerated Method)

D4625 – Middle Distillate Fuel Storage Stability at 43 °C (110 °F)

D6468 – High Temperature Stability of Distillate Fuels (DuPont Stability [Octel F21])

Method	Products	Limitations
D2744	Middle distillate fuels that have specific oxidizing conditions at 95°C. (Gas oil, diesel fuel, No. 2 heating oil, etc.)	Not applicable for fuels containing residual oil and has not been validated for biodiesel or biodiesel blends
D4625	Distillate fuels that have a flash point above 38°C (100°F) and 90% distilled points below 340°C (644°F).	Not intended for quality control testing
D6468 (F21)	Middle distillate fuels under high temperature aging conditions with limited air exposure.	Not suitable for fuels whose flash point is less than 38°C or fuels containing residual oil.

D2274 – 350 mL of filtered middle distillate fuel is aged at 95°C (203°F) for 16 hours with oxygen bubbled through the sample at a continuous rate. After aging, the sample is cooled to room temperature and filtered to obtain the **filterable insolubles** quantity. The oxidation cell and associated glassware are rinsed

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with trisolvent and evaporated to obtain the quantity of **adherent insolubles**. The sum of the filterable and adherent insolubles, expressed as milligrams per 100mL, is reported as **total insolubles**.

D4625 – 400 mL volumes of filtered fuel are aged by storage in borosilicate glass containers at 43°C (110°F) for periods of 0, 4, 8, 12, 18, and 24 weeks. After aging is complete, a sample is removed from storage, cooled to room temperature, and analyzed for **filterable** and **adherent insolubles**.

D6468 – Two 50 mL volumes of filtered middle distillate fuel are aged for 90 or 180 minutes at 150°C in open tubes with air exposure. After aging and cooling, the fuel samples are filtered and the average amount of **filterable insolubles** is estimated by measuring the light reflectance of the filter pads.

Filterable Insolubles

Material that is produced in the course of stressing or storing distillate fuel that is capable of being removed by filtration.

Adherent Insolubles

Material that is produced in the course of stressing or storing distillate fuel that adheres to the glassware after fuel has been flushed from the system.

Total Insolubles

The sum of the filterable and adherent insolubles.

** Please note below, **Turnaround Time** is defined as the actual length of time, on average, it takes to perform a particular method once the sample has arrived and logged in the lab, and prepared for testing.

Method	Reporting Factors	Stability Property	Turnaround Time
D2744	Adherent Insolubles Filterable Insolubles Total Insolubles	Inherent Stability Storage Stability	20 Hours
D4625	Adherent Insolubles Filterable Insolubles Total Insolubles	Inherent Stability Storage Stability	3 Hours
D6468 (F21)	Fuel Color Reflective Rating* Visual Pad Rating	Inherent Stability Storage Stability Thermal Stability	4 Hours (For 90 minute aging) 7 Hours (For 180 minute aging)

*Reflective Rating is used to estimate the filterable insolubles content

Inherent Stability

The resistance to change when exposed to air, but in the absence of other environmental factors such as water, reactive metal surfaces, and dirt.

Storage Stability

The resistance of fuel to formation of degradation products when stored at ambient temperatures.

Thermal Stability

The resistance of fuel to formation of degradation products when thermally stressed.

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Visual Pad Rating	% Refl., W unit	% Refl., Y/Green filt. unit	Relative Stability
1-2	85-100	90-100	Excellent
3-4	71-85	80-89	Good
5-7	62-70	70-79	Fair
8-10	53-61	60-69	Marginal
11-15	32-52	50-59	Poor
16-20	0-31	0-49	V. Poor

Figure 1 - Shows an interpretation of D6468 (F21) results, which are generally accepted.

D6468 (F21) Interpretation Notes

1. The Visual Pad Rating can be affected by the color of the fuel, usually following the general rule that the darker the oil, the darker the pad rating.
2. Reflectance ratings are also affected by the color of filterable insolubles, which may not correlate to the mass of material filtered from the aged fuel sample. Therefore, no quantitative relationship exists between the pad rating and the gravimetric mass of filterable insolubles.
3. The higher the insolubles content, the lower the reflectance rating.
4. The Y search unit with the green filter has a different spectral response than the W search unit. These differences are caused by sediments on filter pads varying in color.

These methods provide stability information for middle distillate fuels and diesel. If fuel has a high insolubles content, the more likely it will have a poor relative stability. This means that exposure to heat or storing the fuel for a long period of time will cause polymerization and oxidation. In order to prevent fuel system failures or combustion problems in the future, additives are used.

Distillate Stability Additives

Distillate degradation products are heavy, sludge-like deposits and dark adherent gums. These molecules can be complex and structurally diverse. Therefore, distillate stability additives need to be “multi-functional” blends of different chemical components to provide good performance across a range of potential degradation reaction pathways. These components would include: antioxidants, dispersants, acid neutralizers and metal deactivator.

Antioxidant additives used for gasoline stability (traditional hindered phenol and PDA) typically do not usually provide good stability performance in distillate fuels.

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Distillate stabilizers can be used to control color darkening and sludge formation during storage; allowing a fuel to be held longer. They can also be used to offset stability impacts when blending increased levels of cycle oil and cracked fractions; allowing increased use of lower-value streams.

Distillate stabilizers, however, cannot be used to change fuel color from a dark ASTM color to a lighter ASTM color. Therefore, it is important to address distillate instability issues prior to color darkening and sludge formation.

AmSpec provides a first class additive service which will aid you in all your additive needs. For more information regarding this service, please contact Scott Hagstrom scott.hagstrom@amspecllc.com

For more general information regarding stability, please contact Jennifer Nesci jnesci@amspecllc.com

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