MARINE FUELS COMPATIBILITY TESTING BY AUTOMATED INSTRUMENTATION ACCORDING TO ASTM D4740

Since 2016, when the International Maritime Organisation (IMO) confirmed a global cap to 0.5% sulfur level in fuel oil used on board of ships, the marine fuel market has undergone complex and far-reaching changes. Issues surrounding fuel stability and compatibility have never been more important.

Stability relates primarily to the fuel's capacity to retain asphaltenes in solution preventing its precipitation and the formation of sludge. However, a bulk fuel, stable and homogeneous at the point of delivery, can become unstable when stored for long periods. Once a fuel has chemically broken down there is no way to satisfactorily reverse the process. Precipitated asphaltene cannot be re-dissolved.

Talking about the compatibility, it is important to remember that a stable fuel oil can become unstable when mixed with another stable fuel, although generally fuels of the same viscosity grade and similar densities will be compatible.

Depending on the manufacturing route and blending components availability, the blended fuels can be predominantly aromatic or paraffinic in nature, or somewhere in between. While these fuels will be stable at their own, the variation in solvency when blended may lead to increased risk of incompatibility creating serious problems such as filters plugging and fuel supply systems affected by flocculated asphaltenes.

In the case of bunkering, a complete segregation of fuels is not possible. There is no guarantee that a vessel bunkering a 0.50%S fuel at Rotterdam will then be able to pick up a compatible fuel in Fujairah.

Carrying out of compatibility tests between the existing and proposed bunker fuel delivery is the only way to provide a realistic evaluation of the potential issues that might result.

A simple and efficient test that can be carried out on board a vessel is the ASTM D4740 spot test.

This quick test helps the ship's crew during subsequent operations such as fuel switching, or when there is a need to bulk fuels for new bunker preparation and/or tank transfers for ship stability purposes.

A drop of the heated and homogenized sample is put on a test paper and heated to 100°C. After 1 hour, the test paper is removed from the oven and the resultant spot is examined for evidence of precipitation and rated for compatibility against ASTM D4740 Adjunct Reference Spots (see Figure 1).

The test contains two procedures: cleanliness and compatibility.

- The cleanliness test is applied to the sample as received to assess the degree to which asphaltenes are already precipitated.
- For the compatibility assessment, a cleanliness test is performed with a blend composed of new bunker and the bunkers remaining on board. It is recommended to test blends at different mixture ratios because some fuel blends may be stable at a certain ratio but unstable at another. Typically, tests are performed at the expected ratios at which fuels will be used or transferred, 10/90; 90/10 and the 50/50 as the worst-case scenario.

The efficacy and accuracy of this simple and affordable test depend heavily on the proficiency and experience of the ship's crew in the interpretation of the spots.

Rating ASTM D4740	Reference Spot	Spot Description	Fuel status
1		Homogeneous spot, no inner ring	Compatible / Stable
2		Faint or poorly defined inner ring	Will deposit some sludge Handle carefully Consider chemical additives Do not overheat Increase purifier Blow down frequency
3		Well-defined inner ring, only slightly darker than the background	As for 2 but increased sludge potential. Future onshore test verification recommended
4		Well-defined inner ring, thicker than the ring in reference spot no. 3 and somewhat darker than the background	Incompatible / Unstable
5		Very dark solid or nearly solid area in the center, the central area is much darker than the background	Incompatible / Unstable

Figure 1. D4740 Adjunct Reference Spots (source ASTM [1])

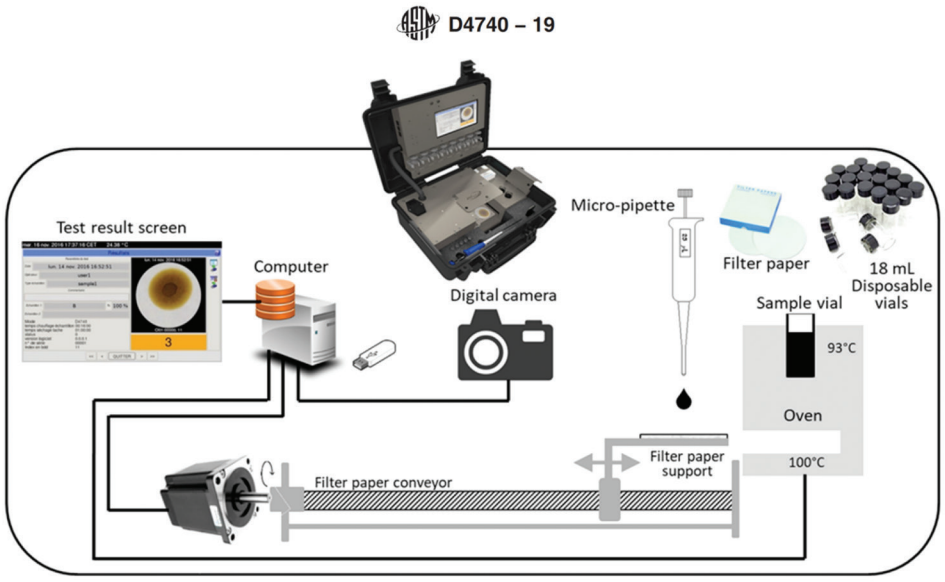


Figure 2. Schematics of the automated apparatus (Source : ASTM [1])

To overcome these limitations, AD Systems, France has developed a portable, fully automated instrument for cleanliness and compatibility testing of heavy fuel oils on-site or on-board. The Model ST10 completely automates both procedures. All accessories and devices required to perform a test are grouped in a robust case. It consists of an oven for sample conditioning, and preparation/drying of the spot, as well as a digital camera and associated software used for automatic spot rating. The only phases carried out by the operator are the sampling and the deposition of a drop of fuel on to a filter paper using a micropipette provided with the equipment. The visual, and therefore subjective, interpretation of the fuel spot has been replaced by a powerful image analysis algorithm.

Since 2019 the automated procedure has been part of the ASTM D4740 test method.

10ml of fuel (cleanliness) or a mix of fuels (compatibility) is poured into a disposable vial and then placed into the built-in oven for 15 minutes. The filter paper is positioned on the conveyor. The ST10 automatically places the filter paper in the oven for drying. In 15 minutes, when the sample is hot, the operator is prompted and the filter paper is pre-positioned. With the micropipette, the operator pours one drop of fuel on the filter paper. The filter paper is automatically moved back to the 100°C oven. After 60 min drying time, the spot is automatically moved under the camera to be photographed.

The picture is binarised, analysed, and the ST10 reports a 1 to 5 rating depending on the cleanliness / compatibility of the fuel. The real image of the spot is displayed, stamped with date/time, and memorised in built-in database with its rating. The ST10 ensures perfect traceability of the test. The standard model of the ST10 includes an Ethernet connection: with central data management, the result can be immediately transmitted to the Cloud via a satellite connection.

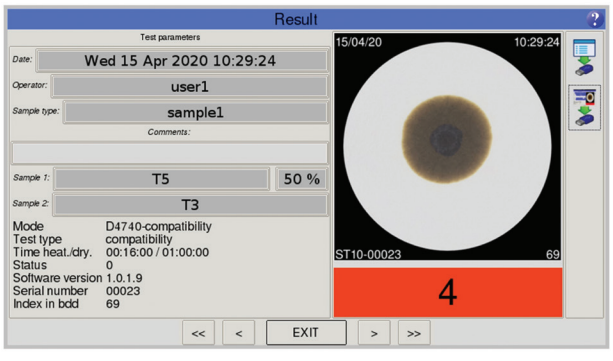


Figure 3. Example of ST10 report D4740 compatibility mode

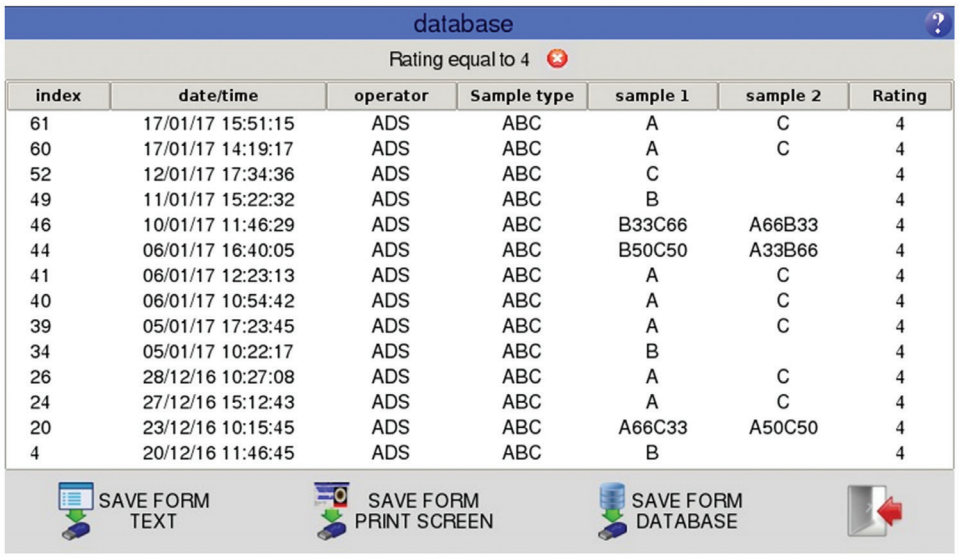
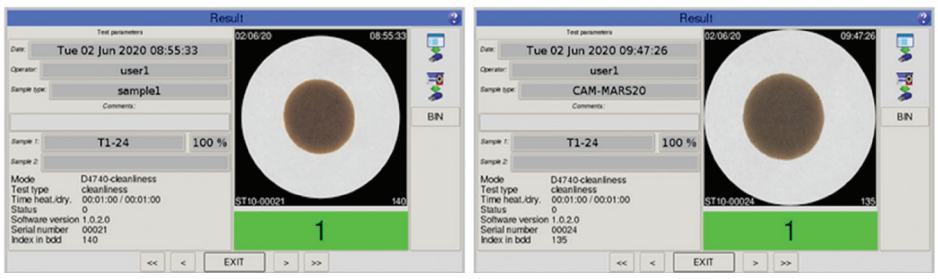
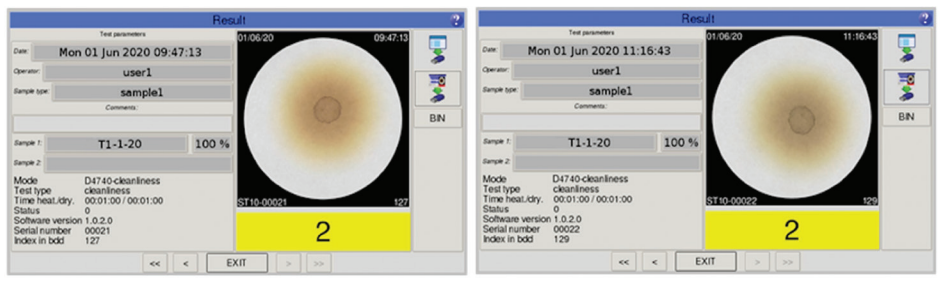


Figure 4. Example of the ST10 database

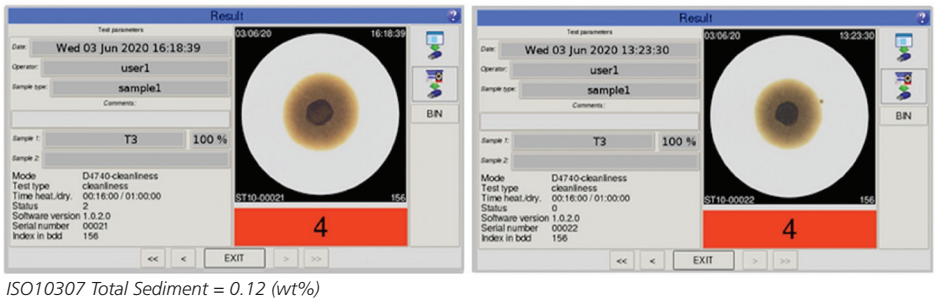


ISO10307 Total Sediment = 0 (wt%)

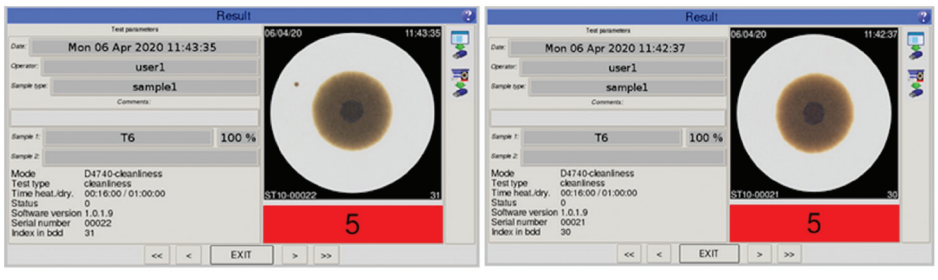


ISO10307 Total Sediment = 0 (wt%)

ISO10307 Total Sediment = 0,01 (wt%)



ISO10307 Total Sediment = 0.12 (wt%)



ISO10307 Total Sediment = 0.4 (wt%)

Figure 5 : Examples of the test reports by the automated analyzer and corresponding sediment level.

However, it needs to be noted, that while it remains a valid and easy test undertaken onboard, the ASTM D4740 spot test can return 'false positives' when testing waxy/paraffinic nature fuels.

Typically containing very low asphaltenes content, such fuels may form an inner ring due to wax separation and not asphaltenes precipitation.

If a spot result requires validation, the stability and compatibility of fuel can be future tested in an onsite laboratory using robust tests such as the Total Sediment test by ISO 10307-2 [3] or Asphaltene Stability test, for example, Intrinsic Stability of Asphaltene (S-value) by ASTM D7157 [4].

Figure 5 shows test reports, generated by the automated analyser, of residual fuels samples and corresponding total sediment value. To evaluate a level of conformity in spot rating, the test was performed in parallel with several ST10 instruments (due to the space limitations of this article only 2 spots are shown).

The spots of samples T3 and T6 were rated 4 and 5 accordingly. These fuels reported as unstable by D4740 have been confirmed by ISO10307-2 as having significant level of sediments. The sample T6-1 generated a warning by spot test with rating 3. In this case the verification by onshore testing reported a low level of sediment, and the fuel can be considered as stable. Thus, additional laboratory tests help to clarify borderline cases.

More comprehensive stability assessment can be performed in an onshore laboratory by evaluating asphaltenes stability and solvency power of the fuel or fuels blend by, for example, S-value analysis described in ASTM D7157. This test method determines stability reserve, i.e. the ability of the fuel to keep the asphaltenes in colloidal suspension .

Performed by the SV10 analyser, manufactured by AD Systems, France, the test procedure is fully automated and involves the dilution of fuel samples by the addition of an aromatic solvent (toluene) followed by titration with a paraffinic solvent (heptane). Asphaltene precipitation is detected by an optical detector monitoring a change in the light transmission properties of the solution. To calculate fuel stability parameters, 3 samples with different dilution ratio are tested and the regression line is plotted. The automatic SV10 analyser can run all 3 samples simultaneously in parallel, thus saving time. The S-value, Sa and So calculated values reported by the analyser will help better evaluate the stability and optimise blending operations.

Figure 6 displays the corresponding reports on D4740 and D7157 tests of residual fuel samples performed by the automated analyser for ST10 (spot test) and SV10 (S-value).

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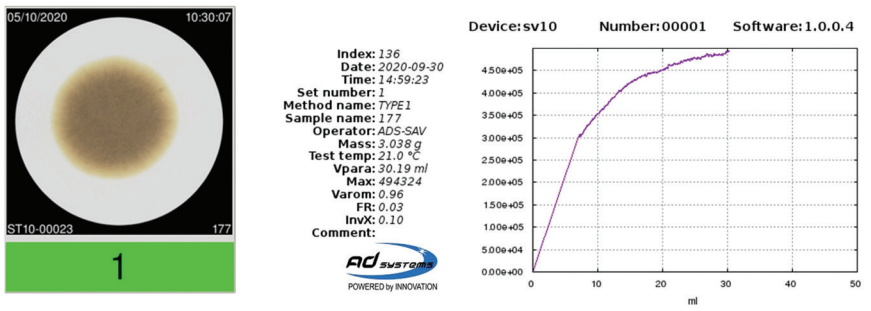

Figure 6-1. Stable fuel sample

The spot is rated 1 and the S-value analysis has shown no floculation, confirming a stable distillate fuel containing no asphaltenes.

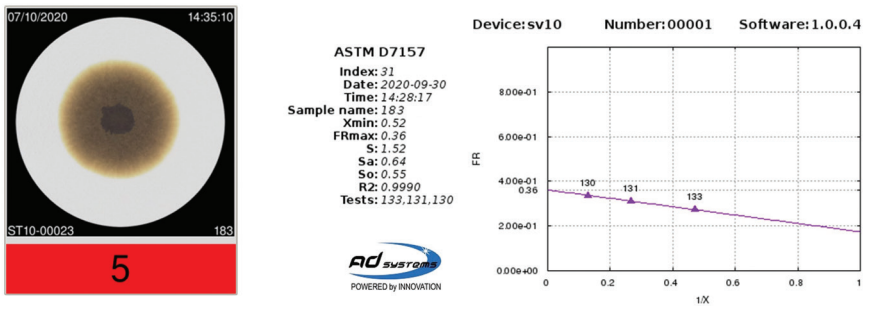

Figure 6-2. Unstable fuel.

The spot test rated 5 of this HFO sample demonstrates that being thermally stressed this fuel makes asphaltenes precipitate. The S-value test confirms the low capacity of this fuel (S value 1.5) to keep the asphaltenes in a colloidal dispersion (Sa value < 0.8) and, thus, has low stability reserve.

Conclusion

The automated D4740 ST10 tester provides vessel operators with a quick and reliable enough determination of the compatibility of onboard fuels without the worry of a potential errors caused by an inexperienced ship's crew. As a result, the apparatus can be used anywhere by untrained personnel wherever and whenever a power source is available.

A video of ST10 in operation is available: https://www.youtube.com/Kl7trsQYCsU

Routinely used by numerous customers, the ST10's proven high level of reliability and precision is helping ships operator in this challenging time.

In cases when a spot result requires validation, the stability and compatibility of fuel can be future evaluated in onsite laboratory using Total Sediment ISO 10307-2, Asphaltene Intrinsic Stability (S-value) by ASTM D7157 or other test methods.

References:

[1] ASTM D4740 "Standard Test Method for Cleanliness and Compatibility of Residual Fuels by Spot Test"

[2] CIMAC Guideline Marine fuel handling in connection to stability and compatibility. (2019)

[3] ISO 10307-2 "Petroleum products — Total sediment in residual fuel oils — Part 2: Determination using standard procedures for ageing"

[4] ASTM D7157 "Standard Test Method for Determination of Intrinsic Stability of Asphaltene-Containing Residues, Heavy Fuel Oils, and Crude Oils (n-Heptane Phase Separation; Optical Detection)"

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Author Contact Details

Igor Borissov, AD Systems • Saint-André-sur-Orne, 14320, France • Email: iborissov@adsystems-sa.com • **Web: www.adsystems-sa.com**