03 | 2024

CIMAC Guideline

Overview and interpretation of total sediment test results in the context of ISO 8217:2024

By CIMAC WG7 Fuels

The first edition of this CIMAC Guideline was approved by the members of the CIMAC WG7 Fuels at its meeting in April 2024.

Content

1 Scope.

This CIMAC document provides an overview of test methodologies for assessing stability and cleanliness of residual marine fuels through the interpretation of the accelerated total sediment (TSA), potential total sediment (TSP) and existent total sediment (TSE) test results.

2 Background.

Experience from the marine fuels market, since the introduction of the 0.50 % sulfur fuels, has shown that the blends making up the very low sulfur fuel oils have various sediment anomalies that are best assessed by applying all three sediment test methods. These results, when viewed together, can provide a more comprehensive understanding of fuel behaviour in order to optimise its storage, handling and use onboard.

Two new tables have been added to the International marine fuel standard ISO 8217:2024 [1] bringing the total number of tables to 4:

Table 1: Distillate and bio-distillate marine fuels

Table 2: Residual marine fuels with sulfur content below or at 0.50 % by mass

Table 3: Bio-residual marine fuels

Table 4: Residual marine fuels with sulfur content above 0,50 % by mass

The new tables 2 and 3 include additional requirements on total sediment, where in addition to the maximum limit for potential total sediment content (TSP), there is a requirement to report the accelerated total sediment content (TSA) and existent total sediment content (TSE).

Clarity is therefore given, in this document, on the potential consequences of the different sediment analysis results with guidance provided on how to interpret the results with regards to the storage and handling of the fuel.

3 Definition and glossary.

VLSFO (Source: ISO 8217:2024 [1]): Very low sulfur fuel oil.

Marine fuel with a maximum sulfur content of 0.50 % by mass.

ULSFO (Source: ISO 8217:2024 [1]): Ultra-low sulfur fuel oil.

Marine fuel with a maximum sulfur content of 0.10 % by mass.

HSFO (Source: ISO 8217:2024 [1]): High sulfur fuel oil.

Marine fuel with a sulfur content exceeding 0.50 % by mass.

TSE (Source: ISO 10307-1 [2]): Existent total sediment.

Sum of insoluble organic and inorganic material, separated from the bulk of the sample by filtration through a specified filter, and insoluble in a predominantly paraffinic solvent.

TSP (Source: ISO 10307-2 Procedure A [3]): Potential total sediment.

Total sediment, determined by ISO 10307-1, after ageing a sample of residual fuel for 24 h at 100 °C under prescribed conditions.

TSA (Source: ISO 10307-2 Procedure B [3]): Accelerated total sediment.

Total sediment, determined by ISO 10307-1, after dilution of a sample of residual fuel with hexadecane in the ratio of 1 ml per 10 g of sample under carefully controlled conditions, followed by storage for 1 h at 100 °C.

Compatibility (Source: ISO 8217:2024 [1])

Ability of two or more fuels to be commingled at a defined ratio without evidence of material separation, which can result in the formation of multiple phases. An example of this is flocculation, where dispersed particles of asphaltenes form larger clusters which can lead to asphaltenic sludge formation.

Stability (Source: ISO 8217:2024 [1])

Stability of a residual fuel is defined by its resistance to the breakdown and precipitation of asphaltenic sludge despite being subjected to forces, such as thermal and ageing stresses, while stored, handled, and treated under normal operating conditions.

Stability reserve (Source: ISO/PAS 23263:2019 [4])

The ability of an oil to maintain asphaltenes in a peptized (colloidally dispersed) state and prevent flocculation and subsequent deposition of the asphaltenes.

FAME (Fatty acid methyl ester) (source: ISO 8217:2024 [1])

Ester derived by (trans-) esterification of fats and oils of vegetal or animal origin.

4 Factors influencing stability.

4.1 Fuel formulation.

The formulation of the fuel blend is the main factor in ensuring fuel stability. It is the responsibility of the supplier to formulate the blend to make sure that the fuel is stable, and that the fuel's stability reserve is sufficient to withstand the normal storage, handling, and treatment onboard a ship.

ISO 8217:2024 includes the use of FAME as a blend component in both distillate and residual fuels without limitation on the blend ratio. The presence of FAME, being an ester, can influence the total sediment result. FAME has good solvency power to keep the asphaltenes solubilised in the oil matrix.

The chemical structure of asphaltene can vary significantly, resulting in different types of asphaltenes with different characteristics and behaviour. To date, the effect of FAME on different asphaltene types and at different FAME blend ratios has not been fully studied, nor fully understood, hence in some cases FAME may potentially have a negative impact on TSP.

Due to the solvency characteristics of FAME, deposits in storage tanks and fuel lines may be dispersed, resulting in a potential increase of deposits in the separator and/or filter clogging issues.

4.2 Thermal and mechanical stresses

The main influences affecting the fuel's stability onboard a ship are the direct thermal and mechanical stresses which may occur during storage, handling and treatment. For marine fuels, temperature management is critical. For further guidance on the use and handling of marine fuels please see CIMAC guideline 02/2024 on design and operation of fuel cleaning systems for diesel engines [5].

4.3 Storage time

Extended storage time can degrade the fuel causing it to become unstable and deposit asphaltenic sludge, along with any inorganic materials present that may settle over time. Since there is no definitive way of assessing how long a fuel will remain homogenous and stable during storage, handling, or treatment onboard, it is recommended to apply "first-in first-out" principle on the use of fuel batches (see CIMAC Recommendation 25 [6]).

Although in the past, the general recommendation has been to use fuels within 6 months, VLSFOs may have reduced shelf-life and hence storage time should be kept to a minimum. If the fuel is expected to be stored for an extended period, samples of the fuel may be drawn periodically to determine any signs of degradation.

4.4 Mixing fuels

Every attempt should be made to avoid mixing fuels in the storage tank.

Mixing individual fuels that are deemed to be stable, does not guarantee that the resulting mixture will also be stable. Instead, the deposition of sediments and asphaltenic sludge may result. Such fuels are described as being incompatible.

The compatibility should be assessed before mixing fuels of different origins where at least one of the fuels is of a residual nature. It is challenging to keep fuels totally segregated onboard ships, as remaining onboard (ROB) quantity, available tanks, tank space, lack of flexibility in storage and cleaning systems do not always allow complete segregation between products.

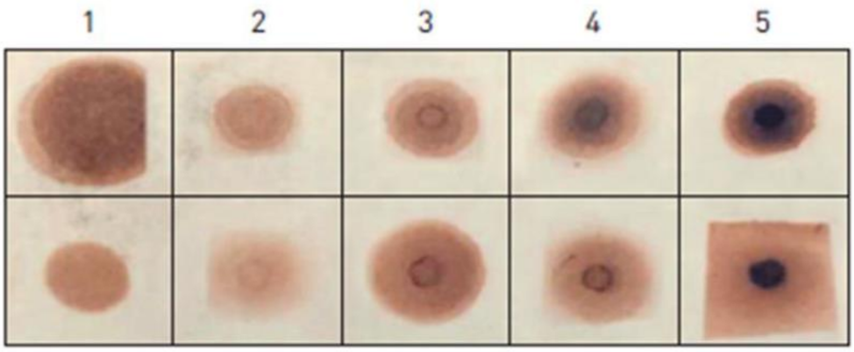
Generally, two fuels with a similar composition are more likely to be compatible. i.e. fuels with similar density, viscosity, carbon residue and total sediment will normally mix without consequence. However, there is no guarantee and as such compatibility testing should always be carried out before mixing fuels.

As per IACS 151 (Recommendation for fuel oil treatment systems [7]) commingling in the different settling and service tanks must be minimized. Ships' engineers should be provided with the most relevant fuel information to enable them to apply best practices in order to prevent fuel mixing as far as reasonably possible.

There are already several documents available to advise on best practices in handling marine fuels. This includes 01/2019 CIMAC Guideline "Marine fuel handling in connection to stability and compatibility" [8] providing in-depth guidance on how to best mitigate onboard issues related to stability and compatibility. The guideline addresses the onboard procedures and routines available to the ships' crew to test for compatibility between different batches of fuels received.

The only available standardized test method, suitable for onboard application, is ASTM D4740 - standard test method for cleanliness and compatibility of residual fuels by spot test [9]. The automated apparatus can be used for onboard screening for compatibility between different fuels, where at least one fuel is residual in nature. It is also noted that this method can give false negative or higher results where waxy, more paraffinic fuels are involved.

Figure 1 demonstrates the variability of results when using ASTM D4740. A spot with a rating of 1 or 2 are defined as "compatible", a rating of 3 as "borderline/caution" and a rating 4 and 5 are "incompatible".



Spot rating description:

1 : Homogeneous spot (no inner ring)
2 : Faint or poorly defined inner ring
3 : Well-defined thin inner ring, only slightly darker than the background
4 : Well-defined inner ring, thicker than the ring in reference spot No. 3 and somewhat darker than the background
5 : Very dark solid or nearly solid area in the center. The central area is much darker than the background

Figure 1: ASTM D4740 spot rating examples

Detailed advice on fuel mixing is available in "Joint Industry Guidance on the supply and use of 0.50 %-sulfur marine fuel" [10] released in late 2019 to facilitate the arrival of more paraffinic, lower sulfur fuels introduced to meet the MARPOL Annex VI [11], 2020 regulations.

5 Test methods

5.1 ISO 10307-1 (existent total sediment, TSE)

TSE measures the amount of sediment present in a fuel at a particular moment by filtration and weighing the amount of sediment on the filter.

TSE is the sum of organic and inorganic materials.

5.2 ISO 10307-2 (total sediment using standard procedures for ageing)

Total sediment aged testing is divided into two ageing procedures:

5.2.1 Potential total sediment, TSP – Procedure A

TSP is the total sediment formed after ageing a sample of residual fuel for 24 h at 100 °C under prescribed conditions, e.g. the amount of sediment after stressing the fuel through heating. This method refers to the thermal ageing of the fuel.

This method is expected to show the total amount of sediment that is already present in the fuel (TSE), combined with the sediment likely to be formed when stressing the fuel thermally.

5.2.2 Accelerated total sediment, TSA – Procedure B

TSA is the total sediment formed after diluting a residual fuel sample with a paraffinic solvent (hexadecane) under carefully controlled conditions, followed by storage for 1h at 100 °C. This method refers to the chemical ageing of the fuel.

This method is expected to show the total amount of sediment that is already present in the fuel (TSE), combined with the sediment likely to be formed after stressing the fuel chemically and during storage at 100 °C for 1 h.

5.3 Precision data

The precision statement detailed in ISO 10307-1 is accurate for distillate fuels with results ranging from 0.01 to 0.40 % by mass and for residual fuels ranging from 0.01 to 0.50 % by mass.

The same precision statement is extended to ISO 10307-2 for both TSA and TSP, as it has been determined that the ageing procedures do not affect the precision of the method.

CIMAC Guideline 01/2024 "The Interpretation of Marine Fuel Analysis Test Results" [12] guides the industry on how to apply ISO 4259-2 [13] (as referenced in ISO 8217) in the interpretation of the analysis test results in respect of those marine fuel characteristics given in ISO 8217, from both recipient and supplier perspectives.

As ISO 8217 defines maximum limits for the sediment test results, the following ISO 4259-2 statements apply:

<u>Recipient</u>

If the recipient has a single test result that is less than or equal to recipient's limit, Y (i.e. the specification limit + 95% confidence level), then the recipient cannot claim that the specification limit has not been met and consequently has to accept that the fuel, as supplied, met the specification limit.

If the recipient has a single test result that is greater than the recipient's limit, Y, then the recipient can claim that the specification limit has not been met. Consequently, the fuel, as supplied, has potentially failed to meet the limit and the supplier is required to test their retained sample.

Note. The tables also contain the values for Y when the laboratory has determined two valid test results and provided the Recipient with the average of these results. In this situation the Reproducibility is modified to R1, in accordance with the relevant equation given in ISO 4259-2.

<u>Supplier</u>

To claim that the fuel has met the specification limit or 'supplier's limit', X, a supplier must determine a single test result at or below that limit. If the single test result is above the specification limit, the supplier cannot claim with any level of confidence that the fuel supplied has met that limit and therefore has to accept that the fuel has failed to meet the specification limit.

Table 1and Table 2 assess the application of ISO 4259-2 for TSA, TSP and TSE.

The reproducibility, R, (as defined in ISO 4259-2) is the closeness of agreement, usually found, between individual results obtained in the normal and correct operation of the same method on identical test material but under different test conditions (e.g., different operators, different apparatus, different calibration standards and different laboratories).

Table 1: Assessment of quality conformance in accordance with ISO 4259-2 for TSE.

Existent total sediment (% by mass) for distillate fuels containing residual components using ISO 10307-1:2009		
Supplier's limit	Recipient's limit Single test result	Recipient's limit Average of two test results
X = Specification Limit	**Y = X + 0.59R**	**Y = X + 0.59R_1**
0.10	0.13	0.13

Table 2: Assessment of quality conformance in accordance with ISO 4259-2 for TSA and TSP.

Total Sediment – Aged (% by mass) for residual fuel using ISO 10307-2:2009 (TSA or TSP)		
Supplier's limit	Recipient's limit Single test result	Recipient's limit Average of two test results
X = Specification Limit	**Y = X + 0.59R**	**Y = X + 0.59R_1**
0.10	0.15	0.15

It should be noted that this precision data was established in the context of HSFO based on data generated in the 1980s. A study is currently being undertaken by ISO/TC 28/WG27 on fuels currently available in the market to evaluate the potential need for amendments to the existing methodology (see Chapter 8). At the time of this publication, there is no precision data for blends containing FAME.

6 Application of the test methods and interpretation of the results.

6.1 Relationship between TSP and TSA

Experience collected over the past decades has shown that for high sulfur fuels, TSA typically exceeds TSP. For this reason, TSA has been used as an alternative to TSP when evaluating if a fuel meets the specification.

However, data and experience since the introduction of VLSFO have shown that some of these lower sulfur fuels exhibit a different relationship between TSA and TSP. As such, TSA is no longer a reliable alternative to TSP even if TSA is within the specification limit.

The reason for the different relationship between TSA and TSP is the difference in composition between ULSFO/VLSFO and HSFO as evidenced often by the lower viscosity and increased paraffinic nature of ULSFO/VLSFO.

Data shows that VLSFOs with a viscosity at 50 °C greater than 200 cSt behave similarly to HSFO, i.e. TSA exceeds TSP. Table 3 summarizes the relationship.

Table 3: Relationship between TSA and TSP depending on fuel type.

Fuel type	% of tested samples*	Viscosity at 50°C	Typical TSA - TSP relationship
HSFO	-	All	TSA > TSP
VLSFO and ULSFO	16%	> 200 cSt	TSA > TSP
VLSFO and ULSFO	84%	≤ 200 cSt	No relationship

* - *percent of tested samples of VLSFO and ULSFO with viscosities above and below 200 cSt.*

Irrespective of viscosity, the relationship between TSA and TSP for HSFO and VLSFO is illustrated in Figure 2 and Figure 3. Figure 3 is a magnification of Figure 2 for a sediment content up to 0.14 mass%, which better illustrates the relationship between TSA and TSP for HSFO, and the absence of relationship for VLSFO, around the specification limit.

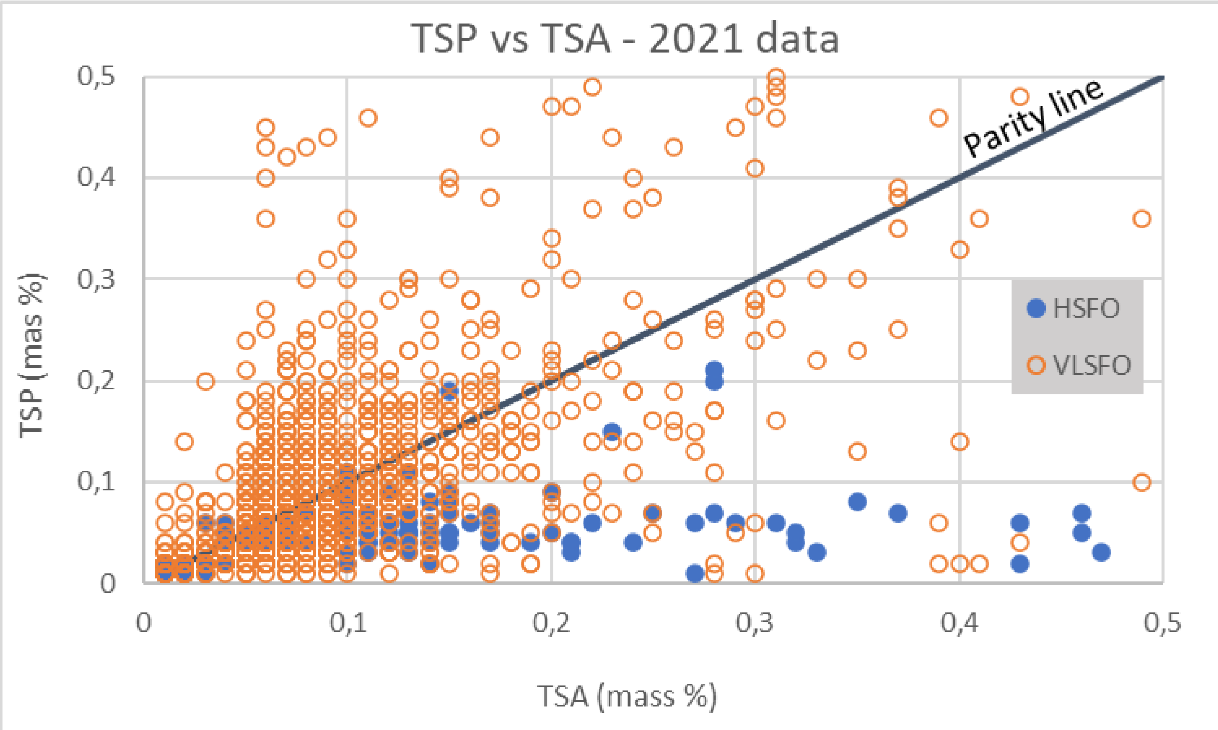


Figure 2: Relationship between TSP and TSA for HSFO and VLSFO

<u>HSFO</u>

- TSA generally exceeds TSP with very few outliers. TSA can therefore be used as an alternative to TSP.
- In case of dispute, TSP remains the reference test method.
- HSFO is sensitive to chemical ageing.

<u>VLSFO</u>

- The large dispersion around the parity line shows no general trends in the relationship between TSA and TSP. As such, TSA cannot be used as an alternative to TSP.
- VLSFO is sensitive to both chemical and thermal ageing.

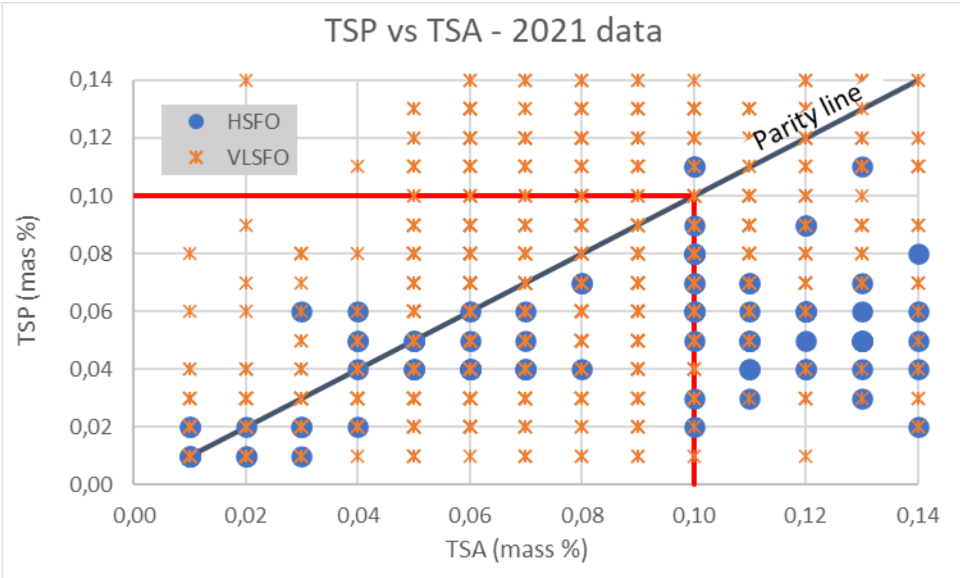


Figure 3: Relationship between TSP and TSA for test results below 0.14 % by mass

6.2 Interpretation of the test results

For HSFO, ISO 8217:2024 limits the total sediment after ageing to max 0.10% by mass. Either TSA or TSP may be applied but in case of a dispute, the reference test method is TSP.

For ULSFO and VLSFO and the bio-residual marine fuels, ISO 8217:2024 limits TSP to max 0.10% by mass and requires that TSE and TSA to be reported. Interpreting the results together will provide a more comprehensive understanding of the fuel's current condition and the possible behavior of the fuel when subjected to storage, treatment, and thermal conditioning prior to use.

Annex 1 provides examples of some combinations of TSE, TSA and TSP results and the interpretation thereof.

7 Summary of the changes in ISO 8217:2024 related to the sediment testing.

Being faster to run to completion, the TSA test method has traditionally been an attractive alternative to TSP, however experience has shown that TSA cannot reliably replace TSP for all fuel types. ISO 8217:2024 takes the following approach basis fuel type (note that TSP is the reference test method in case of dispute for all fuel types):

- ISO 8217:2024, Table 2 (VLSFO): TSP is to be tested while TSA and TSE are to be reported.
- ISO 8217:2024, Table 3 (bio-residual fuel): TSP is to be tested while TSA and TSE are to be reported.
- ISO 8217:2024, Table 4 (HSFO): Both TSA and TSP can be used.

Figure 4 illustrates the approach of ISO 8217:2024 by fuel type.

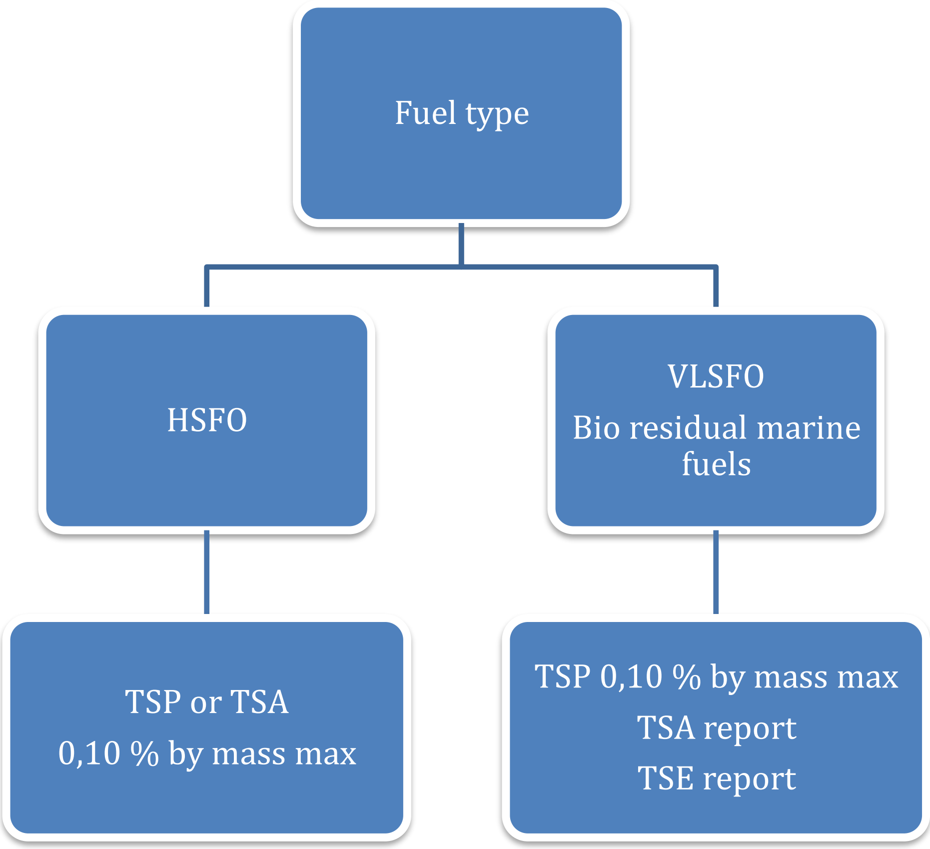


Figure 4: Flowchart for total sediment testing requirements of ISO 8217:2024

8 Test method improvement and performance testing.

Continuous efforts are being made to improve existing test methods and to better anticipate the behaviour of fuel onboard a ship.

As part of this initiative, actions were agreed within ISO/TC 28/WG 27 to review ISO 10307-1 and ISO 10307-2. In parallel new tests are being developed to simulate how a fuel responds to the stress applied during fuel handling and treatment before being burned.

The precision data of ISO 10307-1 test method was established on marine fuels available on the market at the time of the development of the test method, i.e. HSFO.

Due to the variety of fuel blends being used in the market today, including VLSFO and bio-residual marine fuels, both the test methods (ISO 10307, part 1 and 2) are under review to accommodate these fuel types.

Further to the above, new performance tests are being researched to better determine how a fuel might respond to stresses from handling, treatment, and high pressure/velocity conditions onboard a ship.

9 References

[1] ISO 8217:2024, Products from petroleum, synthetic and renewable sources— Fuels (class F) — Specifications of marine fuels

[2] ISO 10307-1:2009, Petroleum products — Total sediment in residual fuel oils — Part 1:

Determination by hot filtration

[3] ISO 10307-2:2009, Petroleum products — Total sediment in residual fuel oils — Part 2:

Determination using standard procedures for ageing.

[4] ISO/PAS 23263:2019, Petroleum products — Fuels (class F) — Considerations for fuel suppliers and users regarding marine fuel quality in view of the implementation of maximum 0.50 % sulfur in 2020

[5] Internal Council on Combustion Engines (CIMAC) – CIMAC guideline on design and operation of fuel cleaning systems for diesel engines (05 | 2024)

[6] Internal Council on Combustion Engines (CIMAC) – Recommendation No. 25, Recommendations concerning the design of heavy fuel treatment plants for diesel engines. (May 2006)

[7] IACS 151 Recommendation for petroleum fuel treatment systems for marine diesel engines

[8] Internal Council on Combustion Engines (CIMAC) – CIMAC Guideline. General guidance in marine fuel handling in connection to stability and compatibility (01 | 2019)

[9] ASTM D4740-20, Standard Test Method for Cleanliness and Compatibility of Residual Fuels by Spot Test

[10] Joint Industry Guidance, The supply and use of 0.50 %-sulphur marine fuels

[11] International Convention for the Prevention of Pollution from Ships (MARPOL), Annex VI — Regulations for the Prevention of Air Pollution from Ships, as amended

[12] Internal Council on Combustion Engines (CIMAC) – CIMAC guideline. The interpretation of marine fuels analysis test results (01 | 2024)

[13] ISO 4259-2:2017, Petroleum and related products — Precision of measurement methods and results — Part 2: Interpretation and application of precision data in relation to methods of test

ANNEX 1: Examples of combinations of TSE, TSA and TSP

Below are some examples of various combinations of TSE, TSA and TSP along with initial guidance which may vary depending on individual ship's circumstances and the interpretation by the testing agencies at that time.

Fuel testing agencies constantly monitor current trends and have experience providing operational recommendations on the basis of the fuel type, the specific combination of TSE, TSA and TSP and the ship installation.

Case N°	Sediment test results on residual fuel blends	TSE	TSA	TSP	Responding guidance
1	All results are low	0.02	0.03	0.03	All three-sediment results show a low propensity for sludging and filter accumulation and, as such, would indicate the fuel to be clean and stable.
2	TSA is exceeding the limit of 0.10 % by mass however TSE and TSP are low	0.02	0.12	0.03	TSP indicates the fuel to be thermally stable – whilst TSA exceeds the 0,10% by mass limit and TSE is low. The high TSA result indicates more risk in case of commingling with a paraffinic fuel. The low TSE and TSP values indicate the fuel to be clean and stable.
3	TSE and TSA are low however TSP is elevated but still within the prescribed ISO 8217 limit	0.02	0.03	0.08	The TSP whilst within limits is elevated and may indicate a limited thermal stability characteristic. Avoid unnecessary overheating of the fuel during storage and avoid prolonged storage. (A reminder to maintain the fuel temperature at least 10 °C above the pour point temperature). The low TSE indicates the fuel to have low content of extraneous dirt, however the slightly elevated TSP may indicate increased asphaltenic sludge deposition at the separators.
4	TSP exceeds the specification limit; TSA and TSE are low	0.02	0.03	0.21	TSP is high with the TSE and TSA being low indicates the fuel to be thermally unstable. Although TSP exceeds the specification limit, the fuel may not necessary be unsuitable for use as experience has shown that the use of fuel with elevated TSP may not result in excessive sludging. It would be expected for the TSA to be higher however the nature of some VLSFO's has shown this to be a specific chemical anomaly for VLSFO blends possibly due to the high paraffinic nature of the fuel. To avoid prolonged excessive heating, it is recommended to put this fuel into use as soon as possible, optimizing the separator plant set up whilst other fuels remain onboard – so that if the problems, as described above, are severe and unmanageable, an alternative fuel is available which has already been previously used without issues.

Case N°	Sediment test results on residual fuel blends	TSE	TSA	TSP	Responding guidance
5	TSE is elevated, TSA is high, and TSP exceeds the specification limit of 0.10 % by mass	0.09	0.17	0.17	All sediment results are high, indicating a possible combination of extraneous dirt and asphaltenic deposit on the test filter. Further laboratory testing, using toluene washing of the high sediments on the TSE filter should be able to differentiate the degree of asphaltenes or extraneous deposits present. It is recommended to optimise the separator plant set up whilst other fuels remain onboard – so that if the level of sludge deposits is severe and unmanageable, an alternative fuel is available, which has already been previously used without issues.
6	Fuel is meeting the spec (TSP is < 0.10 % by mass), although TSA is > 0.10 % by mass	0.02	0.12	0.08	The low TSE indicates the fuel to have low levels of extraneous dirt however the slightly elevated TSP may result in increased asphaltenic sludge deposition at the separators. The TSP, whilst within limits, is still elevated and along with the high TSA may indicate more risk in case of commingling with a paraffinic fuel, for example gasoil. Avoid unnecessary overheating of the fuel during storage and avoid prolonged storage. (A reminder to maintain the fuel temperature at least 10 °C above the pour point temperature).
7	Fuel is slightly exceeding the specification (TSP > 0.10 % by mass) whilst both TSA and TSE are <0.10 % by mass	0.02	0.06	0.12	The higher TSP indicates a possible reduction in thermal stability of the fuel. The low TSE indicates the fuel to have low levels of extraneous dirt, however the elevated TSP may result in increased asphaltenic sludge deposition at the separators and filters. Minimize flow rate and adjust the-sludge cycle as required. To avoid prolonged excessive heating, it is recommended to optimise the separator plant set up whilst other fuels remain onboard – so that an alternative fuel is available which has already been previously used without issues.
8	Fuel is meeting the specification	0.09	0.09	0.09	All the sediment results are elevated and of the same order of magnitude which indicates the presence of inorganic sediment rather than instability or a combination of the two.
9	According to the TSP result, the fuel is meeting the specification, but the high TSA indicates potential compatibility issues	0.02	0.21	0.09	The TSP whilst within limits is still elevated and along with the high TSA may indicate more risk in case of commingling with a paraffinic fuel and possible limitation in the stability reserve of the fuel. The low TSE indicates the fuel to have a low content of inorganic sediment, however the elevated TSP may result in increased asphaltenic sludge deposition at the separators. This should be monitored and time between sludge discharge may need to be reduced.

Case N°	Sediment test results on residual fuel blends	TSE	TSA	TSP	Responding guidance
10	Fuel is not meeting the specification	0.20	0.22	0.25	All the sediment test results are significantly high and of the same order of magnitude which indicate the presence of inorganic sediment rather than instability or a combination of the two. In addition, the TSP result exceeds the ISO 8217 specification limit. These results indicate that operational problems may be experienced with any degree of increased sludge deposition at purifier and filters. Further analytical investigation is recommended. If the use of this fuel is necessary, it is recommended to use the fuel as soon as possible, optimising the separator plant set up whilst other fuels remain onboard – so that an alternative fuel which has already been previously used without issues is still available.

Imprint

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