NIPPON KAIJI KYOKAI

Statement of Fact

No.: KC19YH00027

Date: 29 March 2019

THIS IS TO CERTIFY that the undersigned surveyor attended the test in accordance with the attached test procedure and confirmed the followings at the request of Messrs. Nippon Yuka Kogyo Co., Ltd., Japan on 28-29 March 2019, at the Yokohama factory of Nippon Yuka Kogyo Co., Ltd.;

Fuel oil additive for VLSFO (sludge dispersion type)
Product name: Yunic 800VLS
Manufacturer: Nippon Yuka Kogyo Co., Ltd.

1. Requested contents attendance for sludge dispersion performance

2. General property
   Density : Approx.0.92g/cm^3 (15 °C)
   Kinematic viscosity: Approx. 20cSt (50 °C)
   Flash point : Higher than 70 °C (Tag closed cup)
   Main component : Surfactant, Petroleum solvent

3. Test methods
   (1) The Japanese Shipowners' Association (JSA) improved method
   (2) Centrifugal separation method

4. Test results
   Inspection test results were confirmed as described in Item 4.< Test results> of the attached test procedure and it was confirmed that the amount of precipitated sludge was changed as the dosing rate of Yunic800VLS was increased

(S.KUSUHARA)
Surveyor to Nippon Kaiji Kyokai

Attach: Test procedure for verification test of fuel oil additive "Yunic800VLS"

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2019/3/29

燃料油添加剤 Yunic800VLS の検証試験方案

Test procedure for verification test of fuel oil additive “Yunic800VLS”

日本油化工業株式会社　開発技術課
Development section, Nippon Yuka Kogyo Co., Ltd.

1. ＜試験目的, Purpose＞

本試験の目的は、燃料油添加剤“Yunic800VLS”（日本油化工業（株）製）が、VLSFO の混合による発生スラッジの分散効果について[船主協会－改良法]および[遠心分離法]の 2 種の方法により検証を行う。

実際の検証試験は、日本油化工業（株）横浜工場 技術研究所にて日本海事協会検査員立ち会いのもと実施する。

The purpose of this test is to verify the dispersion performances of the fuel additive “Yunic800VLS” (manufacturer Nippon Yuka Kogyo Co., Ltd.) by sludge formation caused by mixing of VLSFO by 2 types of method ([JSA improved method] and [Centrifugal separation method]).

The actual verification test is carried out with attendance of Nippon Kaiji Kyokai surveyor, and conducted at Technical Research Center, Yokohama Factory, Nippon Yuka Kogyo Co., Ltd.

2. ＜試験方法, Test method＞

1) [船主協会－改良法] [JSA improved method]

※日本船主協会の船舶通報 E・25「燃料添加剤試験方法」Ⅱ・3 項に定める分散試験方法（船主協会法と記す）を一部改良して実施する。表 1 にその詳細を[船主協会－改良法]と記して、参考として船主協会法の概要を付記する。

※Carried out the “Dispersion test method (described as The Japanese Shipowners' Association method, original method) specified in II・3 in the report E・25 "Fuel additive test method" by the Japanese Shipowners' Association. Details are given in Table 1 and together with the outline of JSA original method as a reference.

表 1　試験方法[船主協会－改良法]

項目＼方法		[船主協会－改良法]	参考: 船主協会法
評価方法		定量的評価	定性的評価
		沈殿物有無による A, B, C 評価と沈殿量を正確に読み、経時変化を表と図に示して評価する。	沈殿物有無による A, B, C 評価
供試油調製	供試溶剤	n-ヘプタン:　30mL	n-ヘプタン:　20mL
	燃料油	2 種類の VLSFO の混合油: 0.15g	重油:　0.1g
	攪拌方法	超音波照射とプロペラ攪拌を併用。供試燃料油を微細化するために超音波照射攪拌を行う。	比色管転倒を繰り返す。
評価時間		30 分～6 時間後＋24 時間後 ※24 時間後の状態を写真撮影。	8 時間後
静置温度		約 25±5 °C（室温）	35℃恒温浴

供試燃料油として選定した 2 件の混合油燃料油を用い、下記手順にしたがってスラッジ分散性能試験を実施する。

Table1 Test method [JSA improved method]

Items＼Methods		[JSA improved method]	Reference: JSA original method
Evaluation method		Quantitative evaluation	Qualitative evaluation
		Evaluate of A, B, C the presence or absence of sediment. And accurately read the amount of precipitation. Evaluate the results by elapsed time.	Evaluate of A, B, C the presence or absence of sediment.
Preparation of test oil	Solvent	n-Heptane: 30mL	n-Heptane: 20mL
	Fuel oil	Mixed oil of 2 kinds of VLSFO : 0.15g	Heavy fuel oil: 0.1g
	Stirring method	A combination of ultrasonic applying and the propeller stirrer is performed to refine the heavy components in the test fuel oil.	Repeat up and down of the color comparison tubes.
Evaluation time		30min. ~ 6hours after + 24hours after starting the test. ※Take photograph after 24 hours.	8 hours after starting the test
Temperature		25±5 °C (Room temperature)	35 °C

2 types of mixed 2 kinds of VLSFOs are selected as test fuel oils, perform the dispersion performance test according to the following procedure.

＜[船主協会－改良法]試験手順, Test procedure of [JSA improved method]＞

① 予め表 2 に示す所定量の Yunic800VLS をマイクロシリンジにて比色管に注入し、準備しておく。
A predetermined amount of Yunic800VLS shown in Table2 is injected into the color comparison tube with a microsyringe and prepared in advance.

表 2 比色管 No.と Yunic800VLS 添加量
Table2 Color comparison tube and dosing volume of Yunic800VLS

比色管 No., Color comparison tube No.		1	2	3
Yunic800VLS	添加量, dosing rate	(Blank)	1/1000	1/2000
	添加量, dosing volume, μL		30	15

② トールビーカに供試燃料油 0.50g を採取し、次いで n-ヘプタン 100mL を計量し注ぎ入れる。
Take 0.50g of test fuel oil into a tall beaker, and then pour 100mL of n-Heptane.

③ このトールビーカを超音波洗浄機の浴槽内中央部にセットし、さらに小型撹拌機の攪拌羽根をセットする。
Set this tall beaker in the center of the ultrasonic washing equipment and set a propeller of the small mixer.

④ 撹拌機のスイッチを同時にスタートさせ少なくとも 5 分の連続超音波攪拌を行い、供試燃料油の分散液を調製する。
※この分散液を以後“供試油”と記す。
Start the switch of the stirrer at the same time and perform continuous ultrasonic stirring at least 5 minutes to prepare “the dispersion solution” of the test fuel oil.
※This “dispersion solution” is hereinafter referred to as “test oil”.

⑤ 攪拌終了後、直ちに供試油を各々の比色管に 30mL ずつ注入し、共栓をした後比色管立てごと十回以上繰り返し転倒させ、予め添加してある添加剤との混合を十分行い、室内に静置する。
After stirring, 30mL of test oil is immediately poured into each color comparison tube. After plugged, invert the color comparison tube for 10 or more times and mix well inside and leave it in the room.

⑥ 経時毎に比色管を観察し、次の基準 A～C により分散度を判定すると共に、スラッジの沈殿量を比色管目盛りで読み取り、記録して評価する。
Observe the color comparison tube at each elapsed time, determine the degree of dispersion according to the following criteria A to C, read the sedimentation amount of sludge by the scale on the color comparison tube, record and evaluate the results.

結果, Results A, B and C:

A:	完全に分散し沈殿物のない	Completely dispersed and having no precipitate
B:	分散はしているが沈殿物のある	With dispersion but with precipitation
C:	分散しない	Not dispersed

※試験に供した機器具および試薬　Equipment and reagents used for testing as follows:

○小型撹拌機: Small lab mixer	スリーワンモータ, Three One Motor (HEIDON 1200BL) 回転数, rpm: 500rpm. 攪拌羽根, Propeller: φ50mm with 4 blades
○超音波洗浄機: Ultrasonic washing equipment	AU-50C （アイワ医科工業, Aiwa） 出力, Output: 120W (AC100V). 周波数, Frequency: 28 kHz
○マイクロシリンジ: Micro syringe	10，50μL
○n-ヘプタン: n-Heptane	一級グレード Reagent grade 1

2) [遠心分離法] [Centrifugal separation method]

供試燃料油として選定した 2 件の VLSFO 混合油を用いて、下記手順①～⑤を実施しスラッジ分散性能試験を実施する。この定量的実験は、燃料油添加剤“Yunic555D”との比較も併せて行う。

Using the 2 types of mixed 2 kinds of VLSFOs selected as the test fuel oil, repeat the following procedure twice to carry out the dispersion performance test. This quantitative experiment also compares with fuel oil additive "Yunic555D".

①予め沈殿管に所定量の Yunic800VLS または Yunic555D を注入する
A predetermined amount of Yunic800VLS or Yunic555D was injected into the test tube beforehand

⇩

②n-パラフィン油と供試燃料油を混合調製する
Mix n-Paraffin oil and test fuel oil

⇩

③沈殿管にゴム栓をし、十分中身を混合攪拌する（1 分）。その後直ちに遠心分離器で 6 分間分離する
Plug the test tube and stir thoroughly for 1min. Immediately after separate for 6 minutes with the centrifuge separator

⇩

④沈殿管の上澄みを捨て沈殿量（重量）を計測する
Discard the solvent in the test tube and measure only the amount of sediment

⇩

⑤重量計測後、写真撮影および沈殿量を評価
After measurement, photography and evaluation of sediment quantity

＜[遠心分離法]試験手順, Test procedure of [Centrifugal separation method]＞

① 予め重量を計量した 100mL 沈殿管に、無添加および供試油に対して Yunic800VLS または Yunic555D を 1/500、1/1000、1/2000、1/8000 を各々注入しておく（表 3）。
Inject Yunic800VLS or Yunic555D of 1/500, 1/1000, 1/2000 and 1/8000 respectively of the test oil into a previously weighed glass tube (Table3).

表 3　沈殿管と Yunic800VLS、Yunic555D 添加量
Table3　Test tube and dosing rate and volume (μL) of Yunic800VLS, Yunic555D

沈殿管, Tube No.	1	2	3	4	5	6	7	8
添加率, Dosing rate	1/500	1/1000		1/2000		1/8000		(Blank)
添加量, Dosing volume, μL	200	100		50		12.5		0
添加剤, Fuel additive	555D	800VLS	555D	800VLS	555D	800VLS	555D	No additive

② 供試燃料油 100mL（10vol%）をパラフィン油 900mL（90vol%）に加え、超音波を少なくとも 20 分間照射しながら、十分に攪拌して供試燃料油の分散液（以後、供試油）を調製する。
100 mL (10 vol%) of the test fuel oil is added to 900 mL (90 vol%) of n-Paraffin oil and sufficiently stirred while applying ultrasonic waves at least 20 minutes to prepare the dispersion solution (test oil) of the fuel oil.

③ 次に、②で調製した供試油 100ml を各々の沈殿管に注入し、ゴム栓をし、十分撹拌した後、遠心分離器（1800rpm、6 分間）分離する。
Next, 100 mL of the test oil prepared in ② is poured into each test tube, plugged and thoroughly stirred. Thereafter, it is separated by the centrifuge separator for 6 minutes at 1800 rpm.

④ 遠心分離した後、沈殿管を傾斜して供試油の上澄みだけを排出し、さらに逆さまにして少なくとも 5 分放置後、遠沈管の口を拭き取り、全体重量を計測する。
After centrifugation, tilt the test tube, drain only the liquid of the test oil, leave it still upside down at least 5 minutes, wipe off the oily part on the test tube and measure the weight.

⑤ Yunic800VLS または Yunic555D 添加量に対するスラッジの沈殿量（g）を図示すると共に、沈殿管の外観写真も参考に分散性能を評価する。
Also, evaluate the dispersion performance with reference to the appearance picture of the test tube (Plotting precipitation of sludge (g) relative to the dosing rate of Yunic800VLS and Yunic555D).

※試験に供した機器具類および試薬　Equipment and reagents used for testing as follows:

○小型撹拌機: Small lab mixer	スリーワンモータ, Three One Motor （HEIDON BL1200） 回転数, rpm: 500rpm. 攪拌羽根, Propeller: φ50mm with 4 blades
○超音波洗浄機: Ultrasonic washing equipment	AU-50C（アイワ医科工業, Aiwa） 出力, Output: 120W (AC100V). 周波数, Frequency: 28 kHz
○遠心分離器 Centrifuge separator	油試験用遠心機 H-215-8（コクサン, KOKUSAN） 最大回転数, maximum rpm: 1800rpm
○マイクロピペット: Micro pipette	～200μL, ～20μL
○パラフィン油: Paraffin oil	芳香族, Aroma = 0 vol %

3. ＜供試燃料油, Test fuel oils＞

以下の VLSFO は、オイルサプライヤ（オイルブレンダ）から入手した 2020 年以降の適合油（表 4）。
The following VLSFOs are a compliant fuel oil used after 2020 obtained from oil supplier or oil blender（Table4）.

表 4　2 件の供試燃料油（VLSFO の混合），
Table4　2 types of mixed 2 kinds of VLSFOs（mixed 2 kinds of VLSFO）

	供試燃料油 1,　Test fuel oil 1		
Mixing ratio vol%	50	50	Mixed
Fuel name	Test fuel oil CC	Test fuel oil CP	
Supplier	********	********	
Production area	Asia	Europa	
Density@15 ℃ g/cm^3	0.9289	0.9805	
Viscosity@50 ℃ cSt	190	383	
Flash point ℃	202	118	
CCAI	797	841	
Pour point ℃	Less than 15	Less than 15	
Sulfur %m/m	0.30	0.48	
Asphaltene %m/m	1.81	5.09	
Spot test スポットテスト (※)	3	1	**3**
Al+Si mg/kg (ppm)	1	16	

	供試燃料油 2,　Test fuel oil 2		
Mixing ratio vol%	50	50	Mixed
Fuel name	Test fuel oil CT	Test fuel oil B2	
Supplier	********	********	
Production area	Europa	Asia	
Density@15 ℃ g/cm^3	0.8998	0.9724	
Viscosity@50 ℃ cSt	30	141	
Flash point ℃	106	98	
CCAI	794	844	
Pour point ℃	Greater than 15	Greater than 15	
Sulfur %m/m	0.49	0.48	
Asphaltene %m/m	0.55	3.58	
Spot test スポットテスト (※)	5	3	**6**
Al+Si mg/kg (ppm)	1	22	

※ スポットテストは VLSFO（供試燃料油）を混合した混合安定性を示す。
※ Spot test number indicates compatibility of test fuel oil (mixed 2 kinds of VLSFOs).

4. ＜試験結果, Test results>

1) [船主協会－改良法]による結果　Test results by [JSA improved method]

供試油①, ②　　（燃料油 ＝ 0.15g、溶剤：n-ヘプタン ＝ 30mL）
Test fuel oil ①, ②　　（Test fuel oil = 0.15g and n-Heptane (solvent) =30 mL）

供試油①　Test fuel oil ①

表 5-①　[船主協会－改良法]による結果

Table5-① Test results by [JSA improved method]

比色管 No., Color comparison tube No.		1	2	3
Yunic800VLS	添加量, dosing rate	(Blank)	1/1000	1/2000
	添加量, dosing volume, μL	－	30	15
経過時間毎の評価結果 Evaluation results for each elapsed time	After 30min.	**A**	**A**	**A**
	After 1 hour	**A**	**A**	**A**
	After 2 hours	**A**	**A**	**A**
	After 3 hours	**B / <0.1**	**A**	**A**
	After 4 hours	**B / 0.3**	**A**	**A**
	After 6 hours	**B / 0.8**	**A**	**A**
	After approx. 24 hours	**B / 1.0**	**B / <0.1**	**B / <0.1**



写真 1-①　[船主協会-改良法]による試験結果　(ユニック　800VLS, 約 24 時間後)

Picture1 Test results by [JSA improved method] (Yunic 800VLS, after approx. 24 hours)

供試油②　Test fuel oil ②

表 5-②　[船主協会－改良法]による結果

Table5-②　Test results by [JSA improved method]

比色管 No., Color comparison tube No.		1	2	3
Yunic800VLS	添加量, dosing rate	(Blank)	1/1000	1/2000
	添加量, dosing volume, μL	－	30	15
経過時間毎の評価結果 Evaluation results for each elapsed time	After 30min.	**B / 1.4**	**A**	**A**
	After 1 hour	**C / 1.3**	**A**	**A**
	After 2 hours	**C / 1.1**	**A**	**A**
	After 3 hours	**C / 0.9**	**A**	**A**
	After 4 hours	**C / 0.8**	**A**	**A**
	After 6 hours	**C / 0.8**	**A**	**B / <0.1**
	After approx. 24 hours	**C / 0.8**	**B / <0.1**	**B / <0.1**



写真 1-②　[船主協会-改良法]による試験結果　(ユニック　800VLS, 約 24 時間後)

Picture1 Test results by [JSA improved method] (Yunic 800VLS, after approx. 24 hours)

※1)　表中の“経時毎の評価結果”の A,B,C は船主協会法に基づく分散度の判定を示し、“/”の右側に各比色缶底の沈殿量（mL）を記入する。
例えば、比色管の評価結果 [B/0.3] は、分散度＝B、沈殿量＝0.3mL を表す。
The results A, B, and C of "Evaluation results with elapsed time" in the table indicate the degree of dispersion according to the [JSA improved method]. The amount of precipitation (mL) of each color comparison tube bottom was written on the right side of “ / ”.
For example,[B/0.3] is dispersion degree = B, and precipitation amount is 0.3 mL.

※2)　沈殿量を示す数値が経時により増減しているが、数値が増えている場合はスラッジの沈降が進行中であることを示し、分散度は [B] とする。また、数値が減少している場合、スラッジ沈降は終了と判断し、その時点で分散度判定を [C] とする。
Although the numerical value indicating the amount of precipitation increased or decreased with the elapsed time, when the numerical value was increased, it showed that sedimentation of sludge was in progress and the degree of dispersion is [B]. On the other hand, when the numerical value is decreasing, it showed that sedimentation of sludge was finished, and at that point the degree of dispersion is [C].

2) [遠心分離法]による結果, Test results by [Centrifugal separation method]

供試油①　Test fuel oil ①

表 6-①　[遠心分離法]による結果
Table6 Test results by [Centrifugal separation method] (Upper: Yunic800VLS, Lower: Yunic 555D)

Yunic800VLS 沈殿管, Tube No.	—	2	4	6	8
添加率, Dosing rate	1/500	1/1000	1/2000	1/8000	—
添加量, Dosing volume, μL		100	50	12.5	0
沈殿管重量, Tube weight, g	—	81.3296	81.1912	81.0350	81.6749
全体残量, Total remaining amount, g	—	81.6574	81.5456	81.3971	82.1537
沈殿量, Amount of precipitated sludge, g	—	**0.3278**	**0.3544**	**0.3621**	**0.4788**

Yunic555D 沈殿管, Tube No.	1	3	5	7	8
添加率, Dosing rate	1/500	1/1000	1/2000	1/8000	—
添加量, Dosing volume, μL	200	100	50	12.5	0
沈殿管重量, Tube weight, g	81.6184	81.4258	81.2625	81.2853	81.6749
全体残量, Total remaining amount, g	81.9653	81.7994	81.6434	81.7604	82.1537
沈殿量, Amount of precipitated sludge, g	**0.3469**	**0.3736**	**0.3809**	**0.4751**	**0.4788**

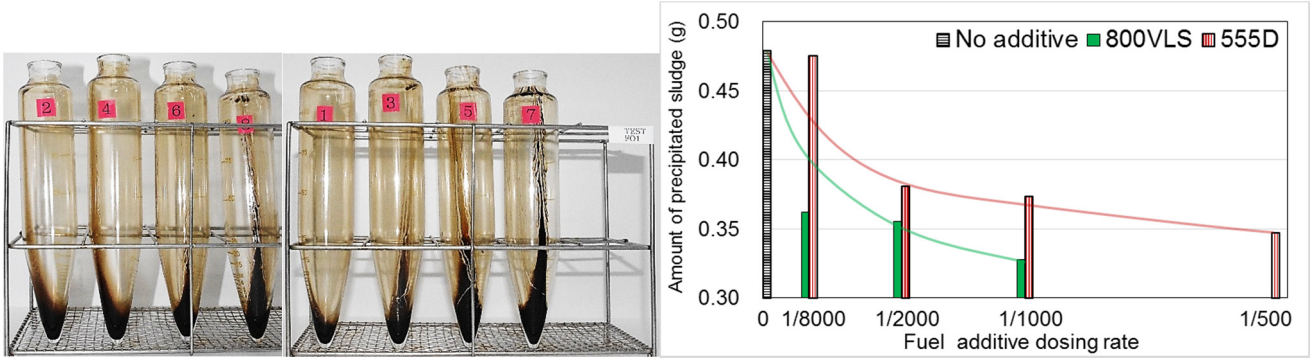


図 1-①　[遠心分離法]による試験結果
Figure1-① Test results by [Centrifugal separation method].

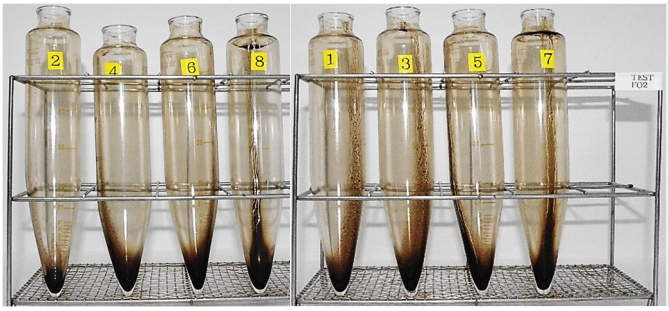
供試油②　Test fuel oil ②

表 6-②　[遠心分離法]による結果

Table6-② Test results by [Centrifugal separation method] (Upper: Yunic800VLS, Lower: Yunic 555D)

Yunic800VLS 沈殿管, Tube No.	―	2	4	6	8
添加率, Dosing rate	1/500	1/1000	1/2000	1/8000	―
添加量, Dosing volume, μL		100	50	12.5	0
沈殿管重量, Tube weight, g	―	81.5521	81.3075	81.4673	81.4507
全体残量, Total remaining amount, g	―	81.7675	81.5472	81.6951	81.7508
沈殿量, Amount of precipitated sludge, g	―	**0.2154**	**0.2397**	**0.2278**	**0.3001**

Yunic555D 沈殿管, Tube No.	1	3	5	7	8
添加率, Dosing rate	1/500	1/1000	1/2000	1/8000	―
添加量, Dosing volume, μL	200	100	50	12.5	0
沈殿管重量, Tube weight, g	81.0926	81.4939	81.2975	81.6215	81.4507
全体残量, Total remaining amount, g	81.3163	81.7272	81.5442	81.8978	81.7508
沈殿量, Amount of precipitated sludge, g	**0.2237**	**0.2333**	**0.2467**	**0.2763**	**0.3001**



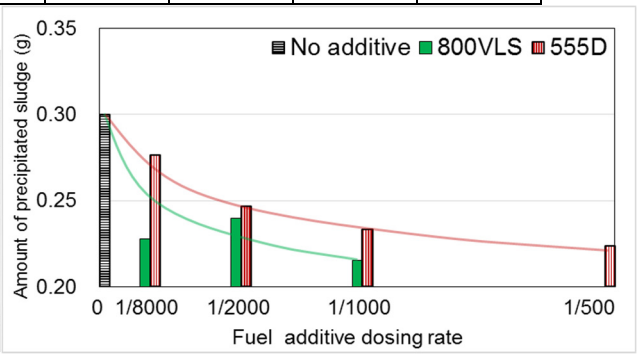


図 2-②　[遠心分離法]による試験結果

Figure2 Test results by [Centrifugal separation method].

以上