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(54) **"A" fuel oil composition and method of manufacture thereof**

(57) An "A" fuel oil composition characterised in that it contains a residual carbon-imparting base material satisfying the following properties (1) to (4):

- (1) a density of not more than 1.0 g/cm³;
(2) a dielectric constant of not more than 3.0;
(3) an asphaltene content of not more than 10% by mass;
and
(4) a resin content of not more than 10% by mass.

In addition to the aforementioned properties (1) to (4), it is preferable if the metal content in the residual carbon-imparting base material is not more than 5 ppm by mass.

A method of manufacture of said "A" fuel oil composition adds said residual carbon-imparting base material to a middle distillate base material.

EP 2 177 591 A1

Description

[0001] The present invention relates to the "A" fuel oil compositions widely used as a fuel for combustion devices such as boilers and diesel engines, other than for use in land transportation, used in agriculture and fishing, fixed electricity generators and construction machinery. More specifically, it relates to an "A" fuel oil composition which has superior filter permeability properties and so does not give rise to blocking of fuel filters by sludge blockages created by separation and mixing of water.

[0002] An "A" fuel oil is a fuel oil type classified as Type 1, No. 1 or No. 2 of the "fuel oils" of JIS K 2205. Its constituents mostly comprise straight-run gas oil or desulphurised gas oil, or fractions such as cracked gas oil. In order to exempt the "A" fuel oils after production from the gas oils which are subject to the provisions of the Local Tax Law, Article 700 (2), No. 1-1, a small amount of a heavy fraction such as a residual oil is, therefore, added as a residual carbon-imparting base material to the "A" fuel oil to make the 10% carbon residue thereof (referred to below as 10% carbon residue) more than 0.2% by mass.

[0003] In general, impurities such as trace metals as well as asphaltene and resins associated with the residual carbon are contained in the heavy fraction constituted by the residual carbon-imparting base material. They subsist as micro-particles in the "A" fuel oil composition in the form of a dry sludge. It is generally known that sludge is a factor in the clogging of filters. Methods of improving the stability of the sludge or of improving permeability have been proposed in order to reduce such sludge. For example, JP-A-2004-091676 discloses an "A" fuel oil composition with improved oil permeability owing to the use of specific petroleum resins as the residual carbon-imparting base material, whilst JP-A-2007-231121 discloses an "A" fuel oil composition with reduced dry sludge owing to the incorporation of specific fatty acid alkyl esters.

[0004] However, the use of specific petroleum resins for the residual carbon-imparting base material or the use of specific fatty acid alkyl esters for the base material require the use of newly installed special manufacturing plant or the purchase of commercial products, and as a result manufacturing costs are increased, which is not desirable. Also, although the amount of dry sludge is reduced, problems remain in that clogging or blockage of filters can still occur in connection with separated water, as described below.

[0005] In other words, the microparticles of the dry sludge present in "A" fuel oil, unless they are too numerous, will easily pass through the mesh of a combustion filter, and so it does not often happen that they cause problems such as blocking filters. On the other hand, in cases where the amount of saturated water is reduced because, for example, of temperature changes in the "A" fuel oil composition during storage, and where the dissolved water separates out, or in cases where there is ingress of water because of dew inside the storage tanks, it is possible that much of the free water will be easily separated and removed by means of a coalescer, but part of the separated water will bond with the particulate dry sludge and be dispersed within the composition and so will not be separated and removed by the coalescer. Also, in the case of the particle size becoming larger than the mesh of the combustion filter, it may often happen that the combustion filter ends up being blocked.

[0006] Therefore, in order to resolve such problems, not only does it go without saying that the dry sludge in "A" fuel oil compositions should be reduced, but it is also necessary to inhibit water bonding with the dry sludge. However, in the prior art, including the references quoted above, it has not proved possible to resolve the problem of filter blockage caused by free water bonding with the dry sludge.

[0007] The aim of the present invention, therefore, is to provide an "A" fuel oil composition which uses a residual carbon-imparting base material obtained by normal petroleum refining processes and which does not give rise to problems of filter blockage caused by dry sludge binding with free water, and also a method of manufacturing same.

[0008] As the result of intensive research into the factors which promote the bonding of water and dry sludge in an "A" fuel oil composition while using a residual carbon-imparting base material obtained by normal petroleum refining processes, it has been discovered that in the case of using a residual carbon-imparting base material having specific properties there is a trend which makes it easier to inhibit the bonding of water and dry sludge.

[0009] Thus, in accordance with the present invention there is provided an "A" fuel oil composition characterised in containing a residual carbon-imparting base material which satisfies the following properties (1) to (4):

- (1) a density of not more than 1.0 g/cm³.
- (2) a dielectric constant of not more than 3.0;
- (3) an asphaltene content of not more than 10% by mass; and
- (4) a resin content of not more than 10% by mass.

[0010] Preferably, in said fuel oil composition the residual carbon-imparting base material is present in the amount of 0.2 to 0.5% by volume.

[0011] What is meant in the present invention by a residual carbon-imparting base material is anything which imparts residual carbon. In general, residual oils in which crude oil has been distilled at atmospheric pressure or under vacuum,

asphalt fractions, or the residual oils of cracking apparatus such as catalytic cracking or hydrocracking, are used as residual carbon-imparting base materials, but in the present invention it is necessary that they satisfy the aforementioned properties (1) to (4). Suitable such base materials are, for example, deasphalted oil obtained by using solvent extraction to remove asphalt from atmospheric distillation residual oils or vacuum distillation residual oils obtained from light crudes of Middle East origin.

[0012] The "A" fuel oil composition of the present invention may also contain a residual carbon-imparting base material which not only satisfies the aforementioned properties (1) to (4) but which also has a metal content of not more than 5 ppm by mass. The main metals which form the total metal content may be assumed to be sodium, potassium, vanadium, nickel, iron, aluminium and silicon, but for other metals, in the same way as for these, it is preferable if they are not contained by the residual carbon-imparting base material.

[0013] In accordance with the present invention there is also provided a method of manufacture of an "A" fuel oil composition **characterised in that** a residual carbon-imparting base material satisfying the following properties (1) to (4):

- (1) a density of not more than 1.0 g/cm³;
- (2) a dielectric constant of not more than 3.0;
- (3) an asphaltene content of not more than 10% by mass; and
- (4) a resin content of not more than 10% by mass is added to a middle distillate base material.

[0014] The residual carbon-imparting base material so added may, in addition to the aforementioned properties (1) to (4), also satisfy the property that the metal content should be not more than 5 ppm by mass.

[0015] Preferably, the residual carbon-imparting base material is added in the amount of 0.2 to 0.5% by volume.

[0016] What is meant by a middle distillate base material in the present invention is a base material having the requisite properties for an "A" fuel oil composition, for example density, distillation characteristics and viscosity, and which incorporates sulphur as appropriate, and mainly refers to fractions obtained by atmospheric distillation of crude oils or by cracking heavy oils in which the boiling point range is approximately 150 to 400°C. In specific terms, it may be a straight-run kerosene fraction or cracked kerosene fraction with a boiling point range of the order of 150 to 280°C, or a straight-run gas oil fraction or cracked gas oil fraction with a boiling point range of the order of 200 to 400°C, or hydrodesulphurised forms thereof.

[0017] The "A" fuel oil composition of the present invention, including the "A" fuel oil composition due to the method of manufacture of the present invention, uses a residual carbon-imparting base material obtained by normal petroleum refining processes, and compared with "A" fuel oil compositions of the prior art it has less bonding of dry sludge with water that has separated out or ingressed during storage or transport, and is less prone to give rise to blockage of combustion filters.

[0018] If the density of the residual carbon-imparting base material incorporated in the "A" fuel oil composition of the present invention is not more than 1.0 g/cm³, it is possible to obtain the effect of the present invention so long as the other aforementioned properties (2) to (4) are satisfied. If the density is greater than 1.0 g/cm³, even if the other properties are satisfied this is detrimental to the separability of the water and the dry sludge, which is not desirable. Moreover, in order to enhance the effect further, the density should preferably be not more than 0.95 g/cm³, and more preferably not more than 0.94 g/cm³.

[0019] If the dielectric constant of the residual carbon-imparting base material incorporated in the "A" fuel oil composition of the present invention is not more than 3.0, it is possible to obtain the effect of the present invention so long as the other aforementioned properties (1), (3) and (4) are satisfied. If the dielectric constant is greater than 3.0, the effect will not be obtained even if the other properties are satisfied, which is not desirable. Moreover, in order to enhance the effect further, the dielectric constant should preferably be not more than 2.9.

[0020] If the asphaltene content of the residual carbon-imparting material incorporated in the "A" fuel oil composition of the present invention is not more than 10% by mass, it is possible to obtain the effect of the present invention so long as the other aforementioned properties (1), (2) and (4) are satisfied, but in order to enhance the effect it should preferably be not more than 5% by mass, more preferably not more than 3% by mass, and most preferably not more than 1% by mass.

[0021] If the resin content of the residual carbon-imparting material incorporated in the "A" fuel oil composition of the present invention is not more than 10% by mass, it is possible to obtain the effect of the present invention so long as the other aforementioned properties (1) to (3) are satisfied, but in order to enhance the effect it should more preferably be not more than 6% by mass.

[0022] Also, if, in addition to the residual carbon-imparting base material satisfying the aforementioned properties (1) to (4), the metal content is not more than 5 ppm by mass, mixtures of water and dry sludge will be less likely to occur. Further, in order to enhance the effect, the metal content should more preferably be not more than 3 ppm by mass and most preferably not more than 1 ppm by mass.

[0023] A detailed description is given below of the "A" fuel oil composition and the method of manufacture thereof.

[0024] The "A" fuel oil composition of the present invention may be obtained by adding a residual carbon-imparting

base material having specific properties to one kind or more of known light oil base materials selected from, for example, straight-run kerosene fractions, straight-run light oil fractions, cracked light oil fractions, cracked kerosene fractions and hydrodesulphurised forms thereof (corresponding to the middle distillate base material of the present invention). What is meant here by straight-run kerosene fractions and straight-run gas oil fractions is that they can be obtained by atmospheric distillation. Cracked gas oil fractions and cracked kerosene fractions can be obtained by catalytic cracking, thermal cracking or hydrocracking of heavy oils. Also, with a view to reducing in advance the sulphur content of the above mentioned cracked gas oil fractions and cracked kerosene fractions, it is possible to carry out a hydrodesulphurisation treatment using indirect or direct desulphurisation processes before the catalytic cracking, thermal cracking or hydrocracking of the heavy oil. In addition, it is possible also to use light hydrocarbon fractions produced in association with the above mentioned desulphurisation reactions for the cracked gas oil fractions and cracked kerosene fractions. Normally, so that these fractions will have the desired properties, they are used in blended forms so that, for example, the density, distillation characteristics, sulphur content and kinematic viscosity are within the desired ranges.

[0025] The residual carbon-imparting base material is a base material added, as mentioned above, so that the 10% carbon residue of the "A" fuel oil becomes greater than 0.2% by mass in order to satisfy the provisions of the tax laws in respect of combustion oils classified as "A" fuel oils, and it is necessary for it to satisfy the properties (1) density of not more than 1.0 g/cm³, (2) dielectric constant of not more than 3.0, (3) asphaltene content of not more than 10% by mass, and (4) resin content of not more than 10% by mass. A deasphalted oil obtained by removal of asphalt by means such as solvent extraction from an atmospheric distillation residual oil or a vacuum distillation residual oil obtained from a Middle East type light oil is typically suitable as an example of such a base material.

Examples

[0026] The present invention will now be described further by reference to the following Examples:

Example 1

[0027] 69.6% by volume of a straight-run gas oil fraction (GO), 30.0% by volume of light cracked oil (LCO) and 0.4% by volume of an atmospheric distillation residual oil obtained by atmospheric distillation of a Middle East type light crude oil (LSLR) as the residual carbon-imparting base material were blended together to prepare an "A" fuel oil composition that satisfied JIS K 2205.

Example 2

[0028] 69.7% by volume of a straight-run gas oil fraction (GO), 30.0% by volume of light cracked oil (LCO) and 0.3% by volume of a deasphalted oil (DAO) obtained by subsequent solvent extraction of a vacuum distillation residual oil as the residual carbon-imparting base material were blended together to prepare an "A" fuel oil composition that satisfied JIS K 2205.

Comparative Example 1

[0029] 69.8% by volume of a straight-run gas oil fraction (GO), 30.0% by volume of light cracked oil (LCO) and 0.2% by volume of a vacuum distillation residual oil obtained by atmospheric distillation and vacuum distillation of a Middle East type medium crude oil (MSSR) as the residual carbon-imparting base material were blended together to prepare an "A" fuel oil composition that satisfied JIS K 2205.

Comparative Example 2

[0030] 69.7% by volume of a straight-run gas oil fraction (GO), 30.0% by volume of light cracked oil (LCO) and 0.3% by volume of a mixture of heavy cycle oil and slurry oil (HCO/SL) with a boiling point in the range of approximately 300 to 550°C and obtained by catalytic cracking of vacuum gas oil, which was the raw material, as the residual carbon-imparting base material were blended together to prepare an "A" fuel oil composition that satisfied JIS K 2205.

Comparative Example 3

[0031] 69.7% by volume of a straight-run gas oil fraction (GO), 30.0% by volume of light cracked oil (LCO) and 0.3% by volume of an atmospheric distillation residual oil (HSLR) obtained by atmospheric distillation of a Middle East type heavy crude oil as the residual carbon-imparting base material were blended together to prepare an "A" fuel oil composition that satisfied JIS K 2205.

EP 2 177 591 A1

Reference Example

[0032] A composition of 70.0% by volume of straight-run gas oil (GO) and 30.0% by volume of light cracked oil (LCO) blended together without incorporating a residual carbon-imparting base material was prepared as a reference example.

[0033] Table 1 shows the properties of the residual carbon-imparting base material incorporated in Examples 1 and 2 and Comparative Examples 1 to 3. Various measurements were carried out as follows.

Dielectric Constant

[0034] Measured by the internal dielectric constant test mutual inductance bridge method of JIS K 6911 "Testing methods for thermosetting plastics".

Density

[0035] Measured by the oscillating type density test method of JIS K 2249 "Crude petroleum and petroleum products - Determination of density and petroleum measurement tables".

Sulphur Content

[0036] Measured by the X-ray fluorescence method of JIS K 2541-4 "Crude oil and petroleum products - Determination of sulphur content".

Carbon Residue

[0037] Measured by the micro method JIS K 2270 "Crude petroleum and petroleum products - Determination of carbon residue".

Ash

[0038] Measured by the ash testing method of JIS K 2272 "Crude petroleum and petroleum products - Determination of ash and sulphated ash".

Nitrogen

[0039] Measured by the chemiluminescence method of JIS K 2609 "Crude petroleum and petroleum products - Determination of nitrogen content".

Metal Content

[0040] Measured in accordance with Japan Petroleum Institute standard JPI-5S-62-2000 "Petroleum products - Determination of metal content". The testing method uses the ICP luminescence method for the determination of aluminium (Al), silicon (Si), iron (Fe), nickel (Ni) and vanadium (Va), and the atomic absorption method for the determination of sodium (Na) and potassium (K).

Saturates, Aromatics, Resins, Asphaltenes

[0041] Measured by the thin-layer chromatography/hydrogen flame ionisation detector method using an 'Iatroscan MK-5' (trade name, made by Iatron Corporation).

Table 1

		LSLR (Ex. 1)	DAO (Ex. 2)	MSSR (Comp. Ex. 1)	HCO (Comp. Ex. 2)	HSLR (Comp. Ex. 3)
Dielectric constant		2.89	2.87	3.19	3.29	3.3
Density	g/cm ³	0.9434	0.9229	1.0170	1.0333	0.9780
Sulphur	wt. %	2.43	1.75	3.68	0.79	3.57

EP 2 177 591 A1

(continued)

		LSLR (Ex. 1)	DAO (Ex. 2)	MSSR (Comp. Ex. 1)	HCO (Comp. Ex. 2)	HSLR (Comp. Ex. 3)
Carbon residue	wt. %	6.71	1.55	19.25	5.82	11.87
Ash	wt. %	0.007	<0.001	0.027	0.003	0.034
Nitrogen	wt. %	0.12	0.08	0.41	0.15	0.26
Sodium	mass ppm	<1	<1	6	<1	36
Potassium	mass ppm	<1	<1	<1	<1	<1
Vanadium	mass ppm	14	<1	150	<1	110
Nickel	mass ppm	5	<1	45	<1	35
Iron	mass ppm	3	<1	10	<1	4
Aluminium	mass ppm	<1	<1	4	3	2
Silicon	mass ppm	<1	<1	1	3	<1
Total metals	mass ppm	22	<1	216	6	187
Saturates	mass %	34.6	46.6	11.4	14.9	21.2
Aromatics	mass %	55.9	46.8	53.6	76.5	54.9
Resins	mass %	5.9	5.8	22	7.8	13.3
Asphaltenes	mass %	3.6	0.8	13	0.8	10.6

[0042] The following measurements were carried out in respect of the Examples 1 and 2, Comparative Examples 1 to 3 and the Reference Example. Table 2 shows the results.

10% Carbon Residue

[0043] After preparation of the 10% carbon residue was carried out, measurements were taken using the micro method of JIS K 2270 "Crude petroleum and petroleum products - Determination of carbon residue".

Water Plus Sludge Content (Centrifugal Separation Test)

[0044] 100 ml of the "A" fuel oil compositions of the Examples and Comparative Examples, and of the reference example, was drawn off into a separating funnel, and was mixed there with 3 ml of water (volume ratio with the "A" fuel oil composition or reference example composition 100 : 3). After shaking for 30 minutes, the mixture was transferred to a graduated test tube used for centrifugal separation and centrifugal separation was carried out for 120 minutes in accordance with JPI-5S-18-80 "Test method for separation of insolubles in used lubricating oils". The volume of the layer of mixed water and sludge which formed immediately on top of the water layer which formed at the bottom of the graduated test tube was measured, and a calculation was made in respect of the total volume of the "A" fuel oil composition and water after centrifugal separation of the volumetric ratio of this mixture layer in the form of a "water + sludge mixture ratio". A 'Shaker SA31' (trade name, made by Iatron Corporation) was used for the shaking, with a stroke of 40 mm and shaking speed of 225 times per minute.

Table 2

		Ex. 1	Ex. 2	Comp. Ex. 1	Comp. Ex. 2	Comp. Ex. 3	Ref. Ex.
GO	Volume %	69.60	69.70	69.80	69.70	69.70	70.0
LCO	Volume %	30.00	30.00	30.00	30.00	30.00	30.0
Residual carbon-imparting base	(Abbreviation)	LSLR	DAO	MSSR	HCO	HSLR	--
	Volume %	0.40	0.30	0.20	0.30	0.30	0.00
10% residual carbon	Mass %	0.25	0.26	0.25	0.30	0.27	0.00

(continued)

		Ex. 1	Ex. 2	Comp. Ex. 1	Comp. Ex. 2	Comp. Ex. 3	Ref. Ex.
5 Mater plus sludge ratio	Volume %	0.4	0.0	3.5	1.5	3.0	0.0

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[0045] As is clear from the results in Table 2, the water plus sludge mixing ratio for the "A" fuel oil compositions of Comparative Examples 1 to 3 is high, whereas it is low for the "A" fuel oil compositions of Examples 1 and 2, and it is evident that there is a trend for a layer of a water and sludge mixture to be less prone to occur. From Table 1 it can be seen that the residual carbon-imparting base materials used in Examples 1 and 2 satisfy in each case the properties (1) density of not more than 1.0 g/cm³, (2) dielectric constant of not more than 3.0, (3) asphaltene content of not more than 10% by mass, and (4) resin content of not more than 10% by mass. In contrast, the residual carbon-imparting base materials used in Comparative Examples 1 to 3 do not satisfy all these conditions, and it can be confirmed that the layer of mixed water and sludge has a trend of being more likely to occur.

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[0046] Also, from the results in Table 2 it can be seen that in Example 2, despite the impartation of a carbon residue, a layer of mixed water and sludge does not occur. From Table 1 it can be seen that the residual carbon-imparting material used in Example 2 has a very low metal content (less than 1 ppm by mass). From these results, it can be confirmed that if, in addition to satisfying the aforementioned properties (1) to (4), the residual carbon-imparting material has a metal content of not more than 1 ppm by mass, a layer of mixed water and sludge is not likely to occur.

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[0047] What is meant by the aforementioned water plus sludge content is different from the water content. Given that, in the reference example, a layer of mixed water and sludge does not occur, it can be confirmed that the influence of the residual carbon-imparting base materials which contain, in addition to water, the important element of the dry sludge, is reflected in the centrifugal separation tests in order to obtain the aforementioned water plus sludge content. Therefore, the aforementioned centrifugal separation tests may be considered an effective test in the measurement of the bonding properties of water and dry sludge.

Claims

- 30
1. An "A" fuel oil composition **characterised in that** it contains a residual carbon-imparting base material satisfying the following properties (1) to (4):
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(1) a density of not more than 1.0 g/cm³;
(2) a dielectric constant of not more than 3.0;
(3) an asphaltene content of not more than 10% by mass; and
(4) a resin content of not more than 10% by mass.
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2. An "A" fuel oil composition according to claim 1 **characterised in that** the metal content in the residual carbon-imparting base material is not more than 5 ppm by mass, preferably not more than 3 ppm by mass.
3. An "A" fuel oil composition according to claim 1 or 2 **characterised in that** the residual carbon-imparting base material is present in the amount of 0.2 to 0.5% by volume.
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4. An "A" fuel oil composition according to claim 1, 2 or 3 wherein the density of the residual carbon-imparting base material is not more than 0.95 g/cm³.
5. An "A" fuel oil composition according to any one of the preceding claims wherein the dielectric constant of the residual carbon-imparting base material is not more than 2.9.
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6. An "A" fuel oil composition according to any one of the preceding claims wherein the asphaltene content of the residual carbon-imparting base material is not more than 5% by mass.
7. An "A" fuel oil composition according to any one of the preceding claims wherein the resin content of the residual carbon-imparting base material is not more than 6% by mass.
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8. A method of manufacture of an "A" fuel oil composition **characterised in that** a residual carbon-imparting base material satisfying the following properties (1) to (4):

- (1) a density of not more than 1.0 g/cm³;
- (2) a dielectric constant of not more than 3.0;
- (3) an asphaltene content of not more than 10% by mass; and
- (4) a resin content of not more than 10% by mass is added to a middle distillate base material.

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9. A method according to claim 8 **characterised in that** the metal content in the residual carbon-imparting base material is not more than 5 ppm by mass, preferably not more than 3 ppm by mass.

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10. A method according to claim 8 or 9 **characterised in that** the residual carbon-imparting base material is present in the amount of 0.2 to 0.5% by volume.

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EUROPEAN SEARCH REPORT

Application Number
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The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 7 January 2010	Examiner Pöllmann, Klaus
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7

EPO FORM 1503 03 82 (P04C01)



EUROPEAN SEARCH REPORT

Application Number
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7

EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 09 17 2762

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The members are as contained in the European Patent Office EDP file on
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