

(19)



(11)

EP 2 042 584 A2

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:

01.04.2009 Bulletin 2009/14

(51) Int Cl.:

C10L 1/222 (2006.01) **C10L 1/238** (2006.01)
C10L 1/2383 (2006.01) **C10L 1/2387** (2006.01)
C10L 1/24 (2006.01) **C10L 10/00** (2006.01)

(21) Application number: **08160792.1**

(22) Date of filing: **21.07.2008**

(84) Designated Contracting States:

**AT BE BG CH CY CZ DE DK EE ES FI FR GB GR
HR HU IE IS IT LI LT LU LV MC MT NL NO PL PT
RO SE SI SK TR**

Designated Extension States:

AL BA MK RS

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(30) Priority: **25.09.2007 US 860664**

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(54) **Diesel fuel additive compositions for prolonged antistatic performance**

(57) An additive composition comprising at least one antistatic agent; and a strong acid, wherein the at least one antistatic agent and the strong acid are present in the additive composition in a ratio of from about 1:0.05 to about 1:1 is disclosed. There is also disclosed a fuel composition comprising a middle distillate fuel and the

additive composition. Methods of making and using the compositions are also disclosed. Further, methods of improving and/or prolonging the conductivity of a fuel are disclosed.

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Description**DESCRIPTION OF THE DISCLOSURE**Field of the Disclosure

[0001] This disclosure relates to an additive composition comprising at least one antistatic agent and at least one strong acid. The additive composition can be combined/blended/admixed with a middle distillate fuel to form a fuel composition. Methods of making and using the additive and fuel compositions are also disclosed. Also disclosed are methods of improving and/or prolonging the ability of an additive composition to impart effective conductivity to a fuel.

Background of the Disclosure

[0002] Certain middle distillate fuel compositions, particularly diesel fuels, are capable of generating static electricity, particularly when moving rapidly, such as when the fuel is being dispensed into a tanker or other bulk container or vessel. While diesel fuels are not very volatile, the tankers used to transport diesel fuels are also used to transport gasoline, kerosene and other more volatile and flammable liquids. Even after the more volatile fuel is dispensed from the tanker, the vapors may still be present and pose a risk of fire or explosion from a spark generated by the discharge of static electricity from the fuel composition.

[0003] These risks have become more acute in recent years with the increased popularity and use of low sulfur fuels and even more acute in recent months with the introduction of ultra-low sulfur diesel fuels. The process used to remove the sulfur from the fuels also decreases the concentration of other polar compounds in the fuel, which in turn reduces the ability of the fuel to dissipate a static charge.

[0004] To mitigate the risks of fire or explosion with low and ultra-low sulfur fuels, it has become common to add a conductivity improver to the fuel at or prior to the point of dispensing the fuel into a bulk container. The conductivity improver, as the name suggests, improves the conductivity of the fuel, thus permitting any static charge built up during high volume transport of the fuel to safely dissipate without generating a spark. Conductivity improvers are also known as antistatic agents.

[0005] The most common type of conductivity improver or antistatic agent used in fuels, particularly diesel fuels, has been the Stadis® brand of antistatic agents sold by Innospec Fuel Specialties, LLC, Newark, Delaware. The Stadis® brand of antistatic agents contain sulfur. Sulfur-containing antistatic agents present a problem when used with additive concentrates that contain basic nitrogen. Specifically, the ability to deliver conductivity improvement by a sulfur-containing antistatic agent dissipates very rapidly when used in additive concentrates containing basic nitrogen because of a reaction between the two materials. This is disadvantageous because it prevents pre-blending of these antistatic agents into additive concentrates that contain basic nitrogen. Many components of a typical fuel additive concentrate may include nitrogen-containing compounds, such as dispersants, detergents, cold flow improvers, lubricity improvers, corrosion inhibitors, stabilizers, and the like. As a result, it is often necessary to add the sulfur-containing antistatic agents separately from the other components of the additive concentrate. Thus, these types of antistatic agents must be kept in a separate tank at the depot and added separately to the fuel. Accordingly, these types of antistatic agents, apart from their inherent additional cost, require additional costs and complexity in terms of storage, handling and dispensing.

[0006] Therefore, there is a need for compositions and methods to address the build-up and discharge of static electricity in middle distillate fuel compositions. Moreover, there is a need for a multifunctional diesel fuel additive package that when stored does not lose the ability to deliver conductivity to the fuel.

SUMMARY OF THE DISCLOSURE

[0007] In accordance with the disclosure, there is disclosed an additive composition comprising at least one antistatic agent; and a strong acid, wherein the at least one antistatic agent and the strong acid are present in the additive composition in a weight ratio of from about 1:0.05 to about 1:1.

[0008] Moreover, there is also disclosed a pre-blended additive composition comprising at least one antistatic agent; and a strong acid, wherein the at least one antistatic agent and the strong acid are present in the additive composition in a weight ratio of from about 1:0.05 to about 1:1.

[0009] In one embodiment is provided herein a method of improving the conductivity in a fuel comprising combining, with the fuel, a strong acid with a composition comprising an antistatic agent and a basic, nitrogen-containing component; wherein the strong acid is combined in an amount sufficient to neutralize at least a portion of the basic nitrogen in the component. In another embodiment, less than all of the basic nitrogen needs to be neutralized to stabilize deliverable conductivity. Thus, in some examples here only about 40%-70% of the basic nitrogen needs to be neutralized by the use of additional strong acid. The amount of basic nitrogen to be neutralized can vary and may be impacted by both

steric availability and basicity leading in one embodiment to less than 40% of the basic nitrogen reacting with the antistatic agent.

[0010] Additional objects and advantages of the disclosure will be set forth in part in the description which follows, and can be learned by practice of the disclosure. The objects and advantages of the disclosure will be realized and attained by means of the elements and combinations particularly pointed out in the appended claims.

[0011] It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the disclosure, as claimed.

DESCRIPTION OF THE EMBODIMENTS

[0012] The present disclosure relates to an additive composition comprising at least one antistatic agent; and a strong acid, wherein the at least one antistatic agent and the strong acid are present in a weight ratio of from about 1:0.05 to about 1:1. The additive composition can be combined/mixed/blended with a middle distillate fuel to form a fuel composition. In order to improve and/or maintain the conductivity of the fuel, the strong acid can, in one embodiment, be combined/mixed/blended with the antistatic agent in an amount sufficient to neutralize at least a portion (40-70%) or all of the basic nitrogen.

[0013] The phrase "improved conductivity" is used to indicate that the ability of the additive package to provide conductivity to the fuel does not decline by more than about 50% from an initial measure with the conductivity additive alone. This measure is taken within 3 hours of storing the additive composition at 90°C. For example, in one embodiment the conductivity effect does not decline by more than about 30% within that 3 hour period at 90°C. As a further example, the conductivity measure declines by no more than about 30% for a period of at least 7 hours at 90°C. In some aspects, the conductivity measure can still be at least about 50% of its initial measure after 10 hours 90°C following the preparation of the additive composition. Thus another embodiment herein provides prolonged conductivity performance of the additive package. In one embodiment, there is disclosed a method of prolonging the conductivity performance of an additive composition comprising combining a strong acid with a composition comprising an antistatic agent, wherein the composition comprises a basic, nitrogen-containing component; and wherein the antistatic agent and the strong acid are combined in a weight ratio of about 1:0.05 to 1:1.0 Useful basic, nitrogen-containing components, such as dispersants, are known to those skilled in the art, but can include for example hydrocarbyl-substituted succinimides, polyetheramines, and Mannich base reaction products.

[0014] The term "conductivity benefit" is used to indicate that the conductivity imparted by the additive composition is sufficient to provide a conductivity of the fuel of at least about 25 pS/m at the time and temperature of delivery of the fuel. All examples herein are well above this value, when measured at room temperature.

[0015] The present compositions can be particularly suited for middle distillate fuel compositions. Middle distillate fuel compositions include, but are not limited to, jet fuels, diesel fuels, and kerosene. In an aspect, the fuel is a low-sulfur fuel having less than about 500 ppm sulfur, for example less than about 350 ppm of sulfur. In an aspect, the fuel can be an ultra-low sulfur diesel fuel or ultra-low sulfur kerosene.

Ultra-low sulfur fuels can be generally considered to have no more than about 30 ppm of sulfur, for example no more than about 15 ppm of sulfur, and as a further example no more than about 10 ppm of sulfur. The term "diesel fuel" is generally considered to be a generic term encompassing diesel, biodiesel, biodiesel-derived fuel, synthetic diesel and mixtures thereof. All disclosures herein to parts per million "ppm" are by mass unless otherwise indicated.

[0016] The present disclosure encompasses jet fuels, although these are conventionally not regarded as "low-sulfur" or "ultra-low sulfur" fuels because their sulfur levels can be comparatively quite high. Nevertheless, jet fuels may also benefit from the conductivity improvement of the present embodiments regardless of their sulfur content.

[0017] The antistatic agent for use in the disclosed compositions can in one embodiment comprise, based on total weight, from about 5 to 25 percent of a polysulfone, from about 5 to 25 percent of a polymeric polyamine, from about 5 to 30 percent of oil-soluble sulfonic acid, and from about 20 to 85 percent of solvent.

[0018] The polysulfone copolymers often designated as olefin-sulfur dioxide copolymer, olefin polysulfones, or poly(olefin sulfone) can be or comprise, for example, linear polymers wherein the structure is considered to be that of alternating copolymers of the olefins and sulfur dioxide, having a one-to-one molar ratio of the comonomers with the olefins in head to tail arrangement. The polysulfones used herein are readily prepared by the methods known in the art (cf. Encyclopedia of Polymer Science and Technology Vol. 9, Interscience Publishers, page 460 et seq.).

[0019] The polyamine component of the antistatic agent disclosed herein can be or comprise, for example, a polymeric reaction product of epichlorohydrin with an aliphatic primary monoamine or N-aliphatic hydrocarbyl alkylene diamine. The polymeric reaction products can be prepared by heating an amine with epichlorohydrin in the molar proportions of from 1:1-1.5 in the temperature range of about 50°C to about 100° C. Generally, with aliphatic monoamines the molar ratio can be about 1:1. The initial reaction product is believed to be an addition product. The aminochlorohydrin upon reaction with an inorganic base can then form an aminoepoxide. The aminoepoxide, which can contain a reactive epoxide group and a reactive amino-hydrogen, can undergo polymerization to provide a polymeric material containing several

amino groups. The ratio of epichlorohydrin to amine and the reaction temperature used are such that the polymeric reaction product can contain from 2 to 20 recurring units derived from the aminoepoxide.

[0020] Generally, the amount of strong acid incorporated in the additive composition can be an equivalent amount, that is, a sufficient amount of strong acid to, in one embodiment, neutralize at least a portion of or all the basic nitrogen, such as a dispersant, although lesser or greater than the equivalent amount can be used. In another embodiment, an amount of the strong acid sufficient to neutralize a portion of the basic nitrogen can be used effectively to achieve the improved antistatic performance of the additive package. Thus, the antistatic agent and the strong acid can, in one embodiment, be present in a weight ratio of about 1:0.05 to about 1:1 depending upon the amount of basic nitrogen present in the additive composition.

[0021] The compositions and methods of the present embodiments can provide conductivity to a fuel of at least 25 pS/m at the time and temperature of delivery. This conductivity is sufficient to meet the proposed new ASTM standard for conductivity in diesel fuels (ASTM D975 and amendments and appendices thereto) measured according to any appropriate test procedure, including but not limited to ASTM D2622 and ASTM D4951. This level of conductivity can be obtained and sustained for extended periods of time by the present embodiments. The disclosed fuel composition can exhibit improved conductivity as compared to a fuel composition devoid of the additive composition. Moreover, the disclosed fuel composition can exhibit prolonged conductivity as compared to a fuel composition devoid of the additive composition.

[0022] The fuel compositions of the present disclosure can contain supplemental additives in addition to the antistatic agent described above. The supplemental additives include, but are not limited to, dispersants/detergents, antioxidants, carrier fluids, metal deactivators, dyes, markers, corrosion inhibitors, biocides, additional antistatic agents, drag reducing agents, demulsifiers, dehazers, anti-icing additives, antiknock additives, cold flow improver, anti-valve-seat recession additives, lubricity additives and combustion improvers.

[0023] The additives used in formulating the fuels of the present disclosure can be blended into the base fuel individually or in various sub-combinations. However, it is recommended to blend all of the components concurrently using an additive concentrate as this takes advantage of the mutual compatibility afforded by the combination of ingredients when in the form of an additive concentrate. Also use of a concentrate reduces blending time and lessens the possibility of blending errors. In an aspect, at least two components, such as the antistatic agent and the strong acid, can be pre-blended.

[0024] Thus there is also provided herein a method of prolonging the conductivity effect of an additive package in a fuel comprising: combining, with the fuel, an additive package comprising a strong acid and an antistatic agent, wherein the additive package comprises a basic, nitrogen-containing component; and wherein the antistatic agent and the strong acid are combined in a weight ratio of from about 1:0.05 to about 1:1.

[0025] Examples 1-4 were prepared as blends of HiTEC® 4130WM, Stadis® 425 and Witconic™ 1298 Hard Acid. HiTEC® 4130WM is a multifunctional diesel fuel additive that contains a polyisobutylene succinimide, a cold flow improver, an ester lubricity additive, a demulsifier, a corrosion inhibitor and solvents and is available from Afton Chemical Corporation. HiTEC® 4130WM typically contains 0.032% nitrogen. Stadis® 425 is a sulfur-containing antistatic additive and is available from Innospec Fuel Specialties, LLC. Witconic™ 1298 Hard Acid is predominantly dodecylbenzene sulfonic acid (DBSA) and is available from Akzo Nobel Surface Chemistry, LLC. The sample formulations were sealed in glass vials and placed in an oven set to 90°C for 16 hours. No fuel was present for the aging. This accelerated aging period is thought to be equivalent to about three (3) months storage at 20°C. After allowing the samples to cool at the end of the aging period, each was evaluated for its effect on improving the conductivity of an ultra-low sulfur diesel (ULSD) fuel (obtained from the Citgo pipeline terminal in Richmond, VA) at 22°C. The results are shown in Table 1. The test ULSD fuel had a measured conductivity of 0 picosiemens per meter (pS/m) without antistatic additive and 504 pS/m when treated with 5 ppm of Stadis® 425 by itself. All 4 example blends were added to the fuel such that the treat rate of Stadis® 425 was 5 ppm. Table 1.

Example #	Mass % in Blend			Blend Treat Rate (ppm)	Measured fuel conductivity @ 22°C after blend aged 16 hr @ 90°C (pS/m)	% loss in conductivity performance of blend
	HiTEC® 4130WM	Stadis® 425	Witconic™ 1298 Hard Acid			
1	99.42	0.29	0.29	1720	506	0.0
2	99.49	0.29	0.22	1720	314	37.7
3	99.54	0.29	0.17	1720	143	71.6

(continued)

Example #	Mass % in Blend			Blend Treat Rate (ppm)	Measured fuel conductivity @ 22°C after blend aged 16 hr @ 90°C (pS/m)	% loss in conductivity performance of blend
	HiTEC® 4130WM	Stadis® 425	Witconic™ 1298 Hard Acid			
4	99.71	0.29	0.00	1720	55	89.1

[0026] A base multifunctional diesel fuel additive ("MFDA") composition is shown in Table 2. The ester lubricity additive is an ethylene glycol diester of dimer acid. The cold flow improver is HiTEC® 4566 fuel additive (available from Afton Chemical Corporation), containing ethylene vinyl acetate copolymer and a nitrogen-containing wax anti-settling component to improve the low temperature filterability of the finished fuel. The cold flow improver contains 0.026 % nitrogen. The conductivity improver (antistatic agent) is Stadis® 425 and the solvent is Aromatic 100 fluid supplied by ExxonMobil Chemical.

Table 2. Base MFDA formulation

Function	Weight %
Ester lubricity additive	22.00
Cold flow improver	25.33
Conductivity improver	1.33
Aromatic solvent	51.33

[0027] This base formulation was then mixed with varying amounts of Witconic™ 1298 (DBSA) and the resulting blends (Examples 5-8) were added to an ULSD fuel (obtained from ExxonMobil Corporation) at 450 ppm. For each of the Examples 5-8, the initial conductivity imparted to the fuel was measured immediately after blending the components (Column A). Additional blends were also stored at 90°C for 16 hours as described above. After aging, these blends were added to the ExxonMobil ULSD fuel and the fuel's conductivity was again measured (Column B). The results are shown in Table 3.

Table 3.

Example #	Column A		Column B		
	Mass % in Blend		Initial measured fuel conductivity (pS/m)	Measured fuel conductivity @ 22°C after blend aged 16 hr @ 90°C (pS/m)	% loss in conductivity performance
	Base MFDA	Witconic™ 1298 Hard Acid			
5	99.89	0.11	778	769	1.2%
6	99.91	0.09	760	600	21.1 %
7	99.98	0.02	750	240	68.0%
8	100.0	0.00	745	135	81.9%

[0028] The results in Table 3 indicate that lesser amounts of the strong acid (Witconic™ 1298 Hard Acid) were needed to achieve the prolonged imparted conductivity because of the reduced amounts of a basic, nitrogen-containing component in the base multifunctional diesel fuel additive composition. Comparative Example 8, having no exogenous strong acid, presented a radical loss in conductivity (81.9%) whereas the Inventive Examples 5-7 illustrate the ability of the aged additive packages provided herein to deliver effective conductivity to fuels containing an antistatic agent.

[0029] For the purposes of this specification and appended claims, unless otherwise indicated, all numbers expressing quantities, percentages or proportions, and other numerical values used in the specification and claims, are to be understood as being modified in all instances by the term "about." Accordingly, unless indicated to the contrary, the

numerical parameters set forth in the following specification and attached claims are approximations that can vary depending upon the desired properties sought to be obtained by the present disclosure. At the very least, and not as an attempt to limit the application of the doctrine of equivalents to the scope of the claims, each numerical parameter should at least be construed in light of the number of reported significant digits and by applying ordinary rounding techniques.

[0030] It is noted that, as used in this specification and the appended claims, the singular forms "a," "an," and "the," include plural referents unless expressly and unequivocally limited to one referent. Thus, for example, reference to "an antioxidant" includes two or more different antioxidants. As used herein, the term "include" and its grammatical variants are intended to be non-limiting, such that recitation of items in a list is not to the exclusion of other like items that can be substituted or added to the listed items.

[0031] While particular embodiments have been described, alternatives, modifications, variations, improvements, and substantial equivalents that are or can be presently unforeseen can arise to applicants or others skilled in the art. Accordingly, the appended claims as filed and as they can be amended are intended to embrace all such alternatives, modifications variations, improvements, and substantial equivalents.

[0032] The application also relates to the following numbered embodiments:

1. An additive composition comprising:

at least one antistatic agent; and
a strong acid,
wherein the at least one antistatic agent and the strong acid are present in the additive composition in a weight ratio of from about 1:0.05 to about 1:1.

2. The additive composition of embodiment 1, wherein the antistatic agent comprises a mixture comprising a polysulfone, a polymeric polyamine, a sulfonic acid, and a solvent.

3. The additive composition of embodiment 1, wherein the antistatic agent comprises, based on total weight, about 5 to about 25% of polysulfone, about 5 to about 25% polymeric polyamine, and about 5 to about 30% oil-soluble sulfonic acid.

4. The additive composition of embodiment 3, wherein the oil-soluble sulfonic acid is or comprises an acid selected from the group consisting of benzenesulfonic acids and alkylnaphthyl sulfonic acids.

5. The additive composition of embodiment 1, wherein the strong acid is or comprises an oil-soluble acid.

6. The additive composition of embodiment 1, wherein the strong acid is dodecylbenzene sulfonic acid.

7. The additive composition of embodiment 1, further comprising a basic, nitrogen-containing component.

8. The additive composition of embodiment 7, wherein at least two components of the additive composition are pre-blended.

9. A pre-blended additive composition comprising:

at least one antistatic agent; and
a strong acid,
wherein the at least one antistatic agent and the strong acid are present in the additive composition in a ratio of from about 1:0.05 to about 1:1.

10. A fuel composition comprising:

the additive composition of claim 1; and
a middle distillate fuel.

11. The fuel composition of embodiment 10, wherein the middle distillate fuel is an ultra-low sulfur diesel fuel.

12. The fuel composition of embodiment 10, wherein the fuel composition exhibits improved conductivity as compared to a fuel composition devoid of the additive composition.

13. The fuel composition of embodiment 10, wherein the fuel composition exhibits prolonged conductivity as compared to a fuel composition devoid of the additive composition.

14. A method of improving the conductivity in a fuel comprising:

combining, with the fuel, a strong acid with a composition comprising an antistatic agent and a basic, nitrogen-containing component;
wherein the strong acid is combined in an amount sufficient to neutralize at least a portion of the basic nitrogen in the component.

15. The method of embodiment 14, wherein the antistatic agent is or comprises a mixture comprising a polysulfone, a polymeric polyamine, a sulfonic acid, and a solvent.

16. The method of embodiment 14, wherein the basic, nitrogen-containing component is a hydrocarbyl-substituted succinimide derived from a hydrocarbyl-substituted succinic anhydride and a polyamine.

17. The method of embodiment 14, wherein the strong acid is dodecylbenzene sulfonic acid.

18. The method of embodiment 14, wherein the antistatic agent and the strong acid are combined in a weight ratio of from about 1:0.05 to about 1:1.

19. A method of improving the conductivity in a fuel comprising:

combining, with the fuel, a strong acid with a composition comprising an antistatic agent, wherein there the composition comprises a basic, nitrogen-containing component; wherein the antistatic agent and the strong acid are combined in a weight ratio of from about 1:0.05 to 1:1.

20. A method of prolonging the conductivity effect of an additive package in a fuel comprising:

combining, with the fuel, an additive package comprising a strong acid and an antistatic agent, wherein the additive package comprises a basic, nitrogen-containing component; and wherein the antistatic agent and the strong acid are combined in a weight ratio of from about 1:0.05 to about 1:1.

Claims

1. An additive composition comprising:

at least one antistatic agent; and
a strong acid,
wherein the at least one antistatic agent and the strong acid are present in the additive composition in a weight ratio of from about 1:0.05 to about 1:1.

2. The additive composition of claim 1 or 2, wherein the antistatic agent comprises a mixture comprising a polysulfone, a polymeric polyamine, a sulfonic acid, and a solvent.

3. The additive composition of claim 1 or 2, wherein the antistatic agent comprises, based on total weight, about 5 to about 25% of polysulfone, about 5 to about 25% polymeric polyamine, and about 5 to about 30% oil-soluble sulfonic acid.

4. The additive composition of claim 3, wherein the oil-soluble sulfonic acid is or comprises an acid selected from the group consisting of benzenesulfonic acids and alkylnaphthyl sulfonic acids.

5. The additive composition of any one of claims 1 to 4, wherein the strong acid is or comprises an oil-soluble acid, in particular dodecylbenzene sulfonic acid.

6. The additive composition of any one of claims 1 to 5, further comprising a basic, nitrogen-containing component, in particular wherein the basic, nitrogen-containing component is a hydrocarbyl-substituted succinimide derived from a hydrocarbyl-substituted succinic anhydride and a polyamine.

7. The additive composition of claim 6, wherein at least two components of the additive composition are pre-blended.

8. A pre-blended additive composition according to any one of claims 1 to 7.

9. A fuel composition comprising:

the additive composition of any one of claims 1 to 8; and
a middle distillate fuel.

10. The fuel composition of claim 9, wherein the middle distillate fuel is an ultra-low sulfur diesel fuel.

11. The fuel composition of claim 9 or 10, wherein the fuel composition exhibits improved and/or prolonged conductivity

as compared to a fuel composition devoid of the additive composition.

12. A method of improving and/or prolonging the conductivity in a fuel comprising:

5 combining, with the fuel, a strong acid with a composition comprising an antistatic agent and a basic, nitrogen-containing component;
wherein the strong acid is combined in an amount sufficient to neutralize at least a portion of the basic nitrogen in the component.

10 **13.** The method of claim 12, wherein the antistatic agent and the strong acid are combined in a weight ratio of from about 1:0.05 to about 1:1 or wherein the fuel is combined with an additive composition according to any one of claims 1 to 8.

14. A method of improving and/or prolonging the conductivity in a fuel comprising:

15 combining, with the fuel, a strong acid with a composition comprising an antistatic agent, wherein there the composition comprises a basic, nitrogen-containing component;
wherein the antistatic agent and the strong acid are combined in a weight ratio of from about 1:0.05 to 1:1 or
20 wherein the fuel is combined with an additive composition according to any one of claims 1 to 8.

15. A method of improving and/or prolonging the conductivity effect of an additive package in a fuel comprising:

25 combining, with the fuel, an additive package comprising an additive composition according to any one of claims 1 to 8.

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REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

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