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2,807,525

ADDITIVE FOR MOTOR FUELS

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4 Claims. (Cl. 44—53)

This invention relates to additives for use in connection with internal combustion engine fuels derived from petroleum to reduce engine gum deposits, and more particularly to additives comprising oil-soluble polyoxyalkylene compounds.

Fuels derived from petroleum, and particularly cracked gasoline fuels, normally contain from 10 to 60% olefins which tend to polymerize upon standing to form "potential" gum. This gum is readily decomposed during combustion of the fuel in the engine to form adhering deposits on the valves and in the intake system.

Gum deposits may be the cause of serious operational difficulties, for the accumulation of deposits in the intake passageway diminishes its size and therefore the maximum air flow which the passage is capable of delivering to the engine. The effects of such deposits on engine performance are, therefore, manifested whenever the engine is operated at or near full throttle as a reduction in power output due directly to the reduction in air flow.

The art has been aware that if there were incorporated in the fuel an additive which has a high solvent power for the gummy residue and which also is a non-volatile liquid at combustion temperatures, it would remain with the residue on the metal parts, such as valve stems, dissolve the gum, and then drain from the parts. Thus it would remove and prevent deposition of residue on such parts. Numerous materials of this type have therefore previously been blended with motor fuels, such as torch oil. In addition to having a high solvent power for the gummy residue and substantial nonvolatility, however, such substances must also be relatively stable to oxidation and decompose into volatile decomposition products. Those materials used heretofore have been deficient in one or more of these properties.

Accordingly, it is an object of this invention to provide an additive for use with motor fuels derived from petroleum which has a high solvent power for gummy potential engine deposits, is a substantially nonvolatile liquid at engine temperatures, and also is stable to oxidation, nonviscous and decomposable into volatile decomposition products.

It is a further object of the invention to provide an additive composition for use with fuels which composition will not only serve to prevent intake system and valve gum deposits, but also lower the freezing point of water

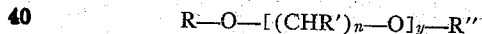
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present in the fuel and therefore prevent water from freezing in the fuel systems, and will also lubricate the valves and keep the valves and manifold clean during use.

In accordance with the invention, these objects are achieved through an additive comprising an oil-soluble polyoxyalkylene compound having a viscosity of from 200 to 1000 SUS at 100° F. These compounds are non-volatile liquids at the temperatures ordinarily encountered in internal combustion engines and are sufficiently nonviscous to drain off from metal surfaces such as valve stems. Moreover, they have a sufficiently high solvency for fuel gum deposits not only to prevent deposition of gummy potential residues present in the fuel but also in many cases to dissolve gummy residues already deposited in the engine. Moreover, they are stable to oxidation and do not themselves deposit residues upon decomposition.

These compounds have a weak but unusual odor, and may also be colored and/or cloudy in appearance. They also have a relatively high viscosity. Desirably, therefore, in order to form a more marketable product these additives are blended with a water-miscible volatile organic aliphatic alcohol, preferably an alcohol miscible with water in all proportions, to form a solution containing from 20% to 80% alcohol by volume. Such an additive composition has a better odor, color, clearness and viscosity, and is easier to blend with the fuel than the polyoxyalkylene compound alone. Also, in addition to preventing the formation of induction system and valve gum deposits, these solutions will lower the freezing point of water present in the fuel. Dilute solutions have the additional advantage of facilitating accurate addition of small amounts of the polyoxyalkylene compound.

The oil-soluble polyoxyalkylene compounds employed as the additives of the invention are old compounds, well known to those skilled in the art, and therefore form no part per se of the present invention. They are characterized by having the following general formula:



in which R and R'' are selected from the group consisting of hydrogen, alkyl and acyl groups having from four to about twenty carbon atoms, R' is hydrogen or an alkyl group having from one to about five carbon atoms, n is a variable small whole number from one to about four, preferably two or three and may vary in this range even in a single long chain molecule, and y represents the number of repeating units in the chain, and may be a whole number from about five to several hundred, depending on the molecular weight.

For the purposes of the invention the character of R' and R'', i. e., of the end groups of the chain, is not critical. Gum solvency properties of these compounds are attributed primarily to the oxyalkylene units which form the chain, and not to the end groups. However, although quite a wide variety of these compounds have gum solvency properties, only those with viscosities in the range of 200 to 1000 SUS at 100° F. have the necessary involatility and gasoline solubility characteristics;

These compounds are prepared by condensation of the corresponding oxides, or oxide mixtures, such as ethylene or 1,2-propylene oxide, or mixtures thereof, as is set forth more fully in U. S. Patents Nos. 2,425,755 to Roberts et al., issued August 19, 1947; 2,425,845 to Toussaint et al., issued August 19, 1947; 2,448,664 to Fife et al., issued September 7, 1948; and 2,457,139 to Fife et al., issued December 28, 1948. If R or R' is alkyl, an aliphatic alcohol is incorporated in the reaction mixture, and if R or R' is acyl, an aliphatic carboxylic acid is incorporated in the reaction mixture.

Compounds in accordance with the above general formula are available commercially under the trade-name "Ucon" from the Carbide and Carbon Chemicals Corporation. The compounds available commercially may have a hydroxyl group at one end of the chain and an alkyl ether or ester group at the other end of the chain, such as those of Patent Nos. 2,425,755, 2,448,664 and 2,457,139 but it will be understood that for the purpose of the instant invention compounds having two terminal hydroxyl groups at each end of the chain, such as those of Patent No. 2,425,845, or having two terminal alkyl ether or ester groups at each end of the chain are suitable as well. All three types of these compounds are intended to be covered by the generic term "polyoxyalkylene compound" as employed in the specification and claims.

It is essential that the polyoxyalkylene compound be oil-soluble, and have an SUS viscosity at 100° F. of from 200 to 1000. Compounds having a viscosity of less than 200 SUS are too volatile to serve as gum solvents, while those having a viscosity of over 1000 SUS are too insoluble in gasoline fuels to be suitable.

When the polyoxyalkylene compound is to be blended with an alcohol to produce an additive composition in accordance with the invention it is preferable that the composition contain alcohol in an amount in the range of 20% to 80% by volume of the composition. It is desirable to employ an organic aliphatic alcohol which is miscible with water in all proportions, such as methyl or ethyl alcohol, since such alcohols produce compositions capable of yielding a greater freezing point lowering upon combination with the water present in the gasoline. However, other less soluble aliphatic alcohols, such as isopropanol and propanol, may also be used for this purpose. Preferably, an alcohol having a lower viscosity than the polyoxyalkylene compound is used as a viscosity reducer in order to facilitate blending of the latter compound with the fuel.

If desired, an inhibitor may also be incorporated in the additive composition in order to inhibit the oxidation of the unsaturated compounds in the fuel to form "potential" gum therein, but inhibitors are not essential, inasmuch as the additive itself will prevent gum present from being deposited in the engine.

In practice, one or a mixture of two or more polyoxyalkylene compounds in accordance with the invention, with the alcohol or an oxidation inhibitor, if these are used, may be blended into a fuel at the refinery. Alternatively the polyoxyalkylene compound may be mixed with the alcohol and an amount of this mixture added to a tank of gasoline at a service station. Since the compounds are liquids and soluble in hydrocarbon fuels derived from petroleum, incorporation is easily accomplished. It has been found that less than 0.01% by volume of the fuel of polyoxyalkylene compound is ineffective, and preferably at least 0.1% is employed. The minimum amount required will of course depend to some extent upon the gum content of the fuel, so that precise limits cannot be set. For economic reasons, however, it is obviously undesirable to employ more additive than is necessary to achieve the desired result. Past experience indicates that amounts of additive in excess of 2% by volume of the fuel will be wasteful,

and give no added improvement with respect to gum solvency. The alcohol will be within the range of 0.025 to 8% by volume of the fuel.

The following are examples of gasoline blends which may be employed:

REGULAR GRADE GASOLINE BLENDS

Mixed catalytically and thermally cracked petroleum distillate	percent by volume	60 to 75
Straight run gasoline	do.	25 to 40
Tetraethyl lead	cc. per gallon	0.5 to 3.0
Polyoxyethylene compound	percent by volume	0.1 to 2.0
Butane in sufficient amount to obtain a Reid vapor pressure of 8-15 pounds per square inch.		

PREMIUM GRADE GASOLINE BLENDS

Mixed catalytically and thermally cracked petroleum distillate	percent by volume	75 to 90
Straight run gasoline	do.	5 to 20
Catalytic polymer gasoline	do.	3 to 10
Tetraethyl lead	cc. per gallon	0.5 to 3.0
Polyoxyethylene compound	percent by volume	0.1 to 2.0
Butane in sufficient amount to obtain a Reid vapor pressure of 8-15 pounds per square inch.		

The following examples give data showing the improvement to be expected by incorporating additive compositions of the invention in motor fuels derived from petroleum.

Example 1

A laboratory test was designed to evaluate the following additive properties:

1. Whether the additive is sufficiently nonvolatile and non-viscous to remain with the gum deposit during or after combustion of the fuel to allow the deposit to drain from metal surfaces, such as valve stems.
2. Whether the additive has a high solvent power for gum deposits.
3. Whether the additive is relatively stable to oxidation.
4. Whether the additive decomposed into volatile decomposition products.

The test procedure is similar to ASTM Gum Determination D381-46 and is as follows:

100 cc. portions of a high gum content fuel (aged thermal debutanizer bottoms, ASTM gum 90 mg. per 100 cc.) containing the additive to be evaluated were heated in weighed ASTM beakers at 320° F. for 1½ hours. A one pound per square inch air jet was maintained throughout. At ½ hour intervals the beakers were examined visually and the appearance of the residue recorded. At the end of 1½ hours the beakers were removed, the residue examined and the weight of residue determined. A rough check of viscosity was made by tilting the beakers and noting the rate of flow of the residue. A sample of each residue was also examined under a microscope.

If the additive tested was too volatile or unstable to oxidation, the residue became very viscous or even solid, and lacquer-like deposits appeared on the sides of the beaker. If the additive showed poor solvent power for the gum deposit, a heavy suspension of granular particles was easily detectable under a microscope. The weight of residue gives some indication of the degree of oxidation and loss by volatilization.

The additives tested were torch oil, zone paraffin (a high paraffin petroleum fraction) and two commercial gum additives, listed A and B in the table below, as well as the additive of the invention, a polyoxyalkylene compound having a viscosity of 300 SUS at 100° F., a viscosity index of 142, a pour point of -40° F., a refractive index n_D^{20} of 1.452 and a density of .0979 g./cc. at 100° F. (Ucon LB-300-X, an oil soluble monobutoxy poly-1,2-oxypropylene glycol having a viscosity of about 300 SUS at 100° F. and containing an inhibitor, available from Carbide and Carbon Chemicals Corporation). The results of the laboratory tests on these additives are summarized in Table I:

TABLE I

Solvent oil	Volume concentration, percent	Viscosity characteristics	Microscope examination	Residue weight, grams	Remarks
Torch oil.....	2	Gradual increase until heavy almost solid residue remained at end of test.	Heavy granular suspension in residue.	-----	This appeared to be poorest of all tested.
Zone paraffin.....	2	Gradual increase throughout test but still fluid at end.	do.....	1.217	Close inspection of photograph shows granular suspension; some lacquer on sides of beaker.
A.....	2	Gradual increase became solid at end of test.	No granular suspension but very dark.	-----	Heavy lacquer on sides of beaker.
B.....	2	Gradual increase until nearly solid at end of test.	do.....	0.495	Do.
Ucon LB-300-X.....	2	No noticeable increase after first ½ hour. Very fluid at end.	-----	2.072	No lacquer. Residues fluid. Marks on sides of beakers in photographs are droplets of residue and dust particles.
Do.....	1	No noticeable increase after first ½ hour. Very fluid at end.	-----	-----	Do.
Do.....	0.5	No noticeable increase after first ½ hour. Very fluid at end.	-----	0.634	Do.
Do.....	0.2	Still fluid when hot at end of test. Very viscous, however.	-----	-----	Lacquer deposits on beaker.

The results show that the polyoxyalkylene compound in concentrations from 0.5% to 2% by volume was superior to the other additives tested. The test, however, being qualitative in nature, did not permit an absolute rating of the additives.

Example 2

The effectiveness of the additive of the invention in an internal combustion engine was determined by means of the combustion chamber deposits cycle test (CCD Test) with no external load. This test reproduces conditions favorable to gum deposition in the intake system and intake valves of the engine, and is designed to simulate normal temperature city driving conditions. The gasoline fuel used in the test was a particularly high gum content thermal debutanizer bottoms, the same fuel used in the laboratory test of Example 1. 1.5 cc. per gallon of tetraethyl lead was added to the fuel. A solvent-refined motor oil was used in the engine in each test. The duration of the test was 30 hours, the time found sufficient in a blank run in which no additive was added to the fuel to give stuck valves and heavy inlet valve deposits.

The evaluation of the additive in each engine test was made by obtaining the following data:

1. Weight of inlet valve deposits.
2. Weight of piston top deposits.
3. Octane demand increase.
4. Compression pressures initially and finally in each combustion chamber.
5. Photographs of inlet and exhaust valves.
6. General description of engine.

Results of engine tests for fuels containing no additive, 0.01 and 0.5% polyoxyalkylene compound (Ucon LB-300-X) and 0.5% torch oil are shown in Table II.

The test data show that while 0.01% polyoxyalkylene compound was insufficient to prevent gum deposition, 0.5% gave excellent results. Torch oil at 0.5% concentration was even less effective than 0.01% polyoxyalkylene compound. Test runs 2 and 4 seem to run higher in gum deposition than the blank, but the most striking difference between the fuel containing 0.5% polyoxyalkylene compound and runs 2 and 4 lies in the inlet valve deposits and the appearance of the inlet valve chamber. The inlet valves in test run No. 3 were only slightly coated and the inlet valve chamber was free of gum deposits. Moreover, the combustion chamber deposits were less in run No. 3 than in the blank, as the piston top deposit weights show. The octane demand increase for test run No. 3 was only 6 numbers, compared with 10 numbers for the blank. Because of the short duration of runs Nos. 2 and 4, piston top weights cannot be fairly compared with those of run No. 3.

Example 3

Fuels containing the additive and the additive-alcohol composition of the invention were tested to determine the relative amounts of the deposit in the engine fuel induction system by the following procedure.

The equipment consisted of a Waukesha single cylinder knock testing engine equipped with a special glass intake manifold. This manifold system incorporated a 16¾ inch long Pyrex tube (22 mm. outside diameter) between the carburetor and the intake port where it was held by two special flanges. A Nichrome coil of 10 ohms resistance was wrapped around the glass manifold about 3 inches from the carburetor end so that the heated area was about 3½ inches long. This heating element was controlled by a 110 volt Variac and supplied localized

TABLE II

Additive	Fuel Additive conc'n, volume percent	Percent diolefin	ASTM gum, mg./100 cc.	Average inlet valve deposits, wt. in grams	Average piston top deposits, wt. in grams	Octane demand increase	Average compression pressure change, p. s. i.	Remarks
1. None.....	-----	1.06	52	0.911	8.085	10	-16.....	Usually heavy and gummy deposits in inlet valve chamber. Combustion zone not unusual in appearance.
2. Ucon (LB-300-X).....	0.01	1.10	64	1.082	3.909	-----	-6 at 11 hrs.	Lasted only 13 hours because of stuck inlet valve. Valve deposit was glossy. Heavy gum deposits in valve chamber.
3. Ucon (LB-300-X).....	0.5	0.97	41	0.440	5.724	6	+5.....	Clean inlet valve chamber. Glossy thin deposit on valves. Thinner but similar combustion zone deposits.
4. Torch Oil.....	0.5	1.10	64	1.150	3.262	-----	-60.....	Lasted only 12 hours because of stuck inlet valves. Heavy gum deposits in valve chamber.

heating to the special manifold. The temperature of the manifold was measured by a surface contact thermocouple located about 1/2 inch beyond the end of the coil, and the temperature was taken directly from a potentiometer. The intake air was drawn into the carburetor through a preheater consisting of a glow coil heater in the induction system surge. The heater was controlled from the engine panel board. The engine was operated for 10 hours, with the fuel being tested under the following conditions:

R. P. M. _____ 600.
Coolant temperature _____ 210° F.
Oil temperature _____ Approximately 150° F.
Air temperature:
Intake at carburetor _____ 95° ± 5.
1/2" beyond the coil _____ 170° ± 5.
Approximate fuel consumption _____ 1.2 lbs./hour (1.8 gal./10 hours).

The glass manifold was weighed both before and after the test. The difference in weight is the amount of gum deposited during the combustion of the fuel in the hot area of the manifold and is a measure of the effectiveness of the additive in preventing gum deposits. Four runs were made using the fuels set forth in the table below, with the results reported therein:

TABLE III

No.	Fuel	ASTM gum, g.	Grams of deposit in manifold at 10 hrs.
1	Caustic treated gasoline containing 3 cc. of tetraethyl lead per gallon.	10-15	0.70
2	Fuel No. (1) containing 0.25% by volume polyoxyalkylene compound (Ucon LB-300-X).		0.07
3	Commercial gasoline containing 4.5 cc. tetraethyl lead per gallon.	3.2	0.045
4	Fuel No. (3) containing 0.3% polyoxyalkylene compound (Ucon LB-300-X).		0.030

Runs Nos. 1 and 2 show that a polyoxyalkylene compound in a concentration as low as 0.25% by volume will reduce the gum deposit of a high gum fuel by a factor of 10. Run Nos. 3 and 4 show that even a gasoline fuel of very low ASTM gum rating can be improved by the addition of a polyoxyalkylene compound. In addition to the decrease in gum deposit, the appearance of the glass manifold in run No. 4 was much better than run No. 3.

The polyoxyalkylene compound added to fuel No. 4 was in the form of a methanol solution containing 25 parts of polyoxyalkylene compound to 75 parts methanol. This composition was added to the fuel in the ratio of one pint to each 10 gallons, i. e., a ratio of 1 part methanol solution to 80 parts fuel.

This polyoxyalkylene compound methanol composition was effective not only to reduce the gum deposit of a gasoline, but also to reduce its freezing point, and, in fact, is superior in this respect to several commercial products tested. This composition in the ratio of 1 part to 80 parts fuel was added to a gasoline containing 0.25% water by volume, and the freezing points of the water contained therein determined after standing from 1/2 hour to 68 hours. The data taken was then compared with the freezing points obtained when two comparable commercial freezing point depressants compositions were

added to the same fuels in the same proportions. The results are reproduced in the table below:

TABLE IV

Percent H ₂ O (volume)	Gasoline	Depressant	Freezing points of H ₂ O (° F.)			
			1/2 hr.	20 hrs.	44 hrs.	68 hrs.
0.25	Premium	25% Ucon LB-300-X, 75% Methanol.	-59	-86	-86	-86
0.25	Regular	do	-59	-85	-85	-86
0.25	do	Commercial A	+9	+17	+20	+20
0.25	do	Commercial B	-7	-23	-25	-24

These results show that the polyoxyalkylene compound-methanol composition is far superior to the other commercial compositions tested in freezing point lowering.

Example 4

In order to determine the optimum concentration of polyoxyalkylene compound in a composition thereof with methanol required to achieve maximum reduction in gum deposit, together with an excellent freezing point depression, using the minimum amount of polyoxyalkylene compound, compositions containing all proportions of polyoxyalkylene compound and methanol were prepared and the freezing points determined of water in gasoline containing these mixtures in an 80:1 ratio of gasoline to the mixture. The following results were obtained:

TABLE V

Percent H ₂ O (by volume)	Gasoline	(Volume percent) percent Ucon (LB-300-X)	(Volume percent) percent methanol	Freezing point of H ₂ O (1/2 hr. contact time), ° F.
0.25	Regular	0	100	-79
		25	75	-59
		50	50	-25
		75	25	+9
		100	0	+31
0.25	Premium	0	100	-80
		25	75	-59
		50	50	-27
		75	25	+8
		100	0	+31
0.50	Regular	0	100	-62
		25	75	-46
		50	50	-32
		75	25	+17
		100	0	+31

The results for fuel No. 4 of Example 3 show that a 1:80 mixture of 25% polyoxyalkylene compound 75% methanol and gasoline is sufficient for gum inhibition and the above results show that this mixture gives superior depression of the freezing point of water in the fuel. Such a gasoline composition contains 0.3% polyoxyalkylene compound, which approaches the minimum necessary to give a reduction in gum deposit. When larger amounts of polyoxyalkylene compound are present, a lower freezing point depression is obtained. A proportion of from 25 to 50% polyoxyalkylene compound to methanol gives optimum results as regards both freezing point depression and gum deposit reduction.

Example 5

Compositions were prepared containing polyoxyalkylene compound (Ucon LB-300-X) in varying proportions with isopropanol and these were dissolved in water-containing gasoline in a 1:80 ratio, and the freezing point of the

water thereafter determined. The following results were obtained:

TABLE VI

Percent H ₂ O (by volume)	Gasoline	(Volume percent) percent Ucon (LB-300-X)	(Volume percent) percent isopropanol	Freezing point H ₂ O, ° F.
0.25-----	Regular-----	25	75	+18
		50	50	25
		75	25	28
0.5-----	do-----	25	75	+27
		50	50	29
		75	25	31

These results show that isopropanol is satisfactory although not quite as effective as methanol in reducing the freezing point of water.

Compositions containing polyoxyalkylene compound dissolved in n-propanol and ethylene glycol were also tested, and were similar to isopropanol.

Example 6

Road tests were made using fuel No. 4 of Example 3. One series of tests using a 1946 Ford V-8 showed that the polyoxyalkylene compound present in this fuel effectively suppressed gum formation on the inlet valves of the engine. Some of the valves, after the engine had been run 31,675 miles, were cleaned and the engine then run with fuel No. 4. After 93 hours of idle (800 R. P. M., equivalent to 1480 miles of driving) and 281 road miles these valves showed no sign of additional gum formation.

Fuel No. 4 was also used to operate a police motor launch which operated at low speed (10 to 20 M. P. H.) or idled frequently (40% of its operating time). The polyoxyalkylene compound was found to be effective in removing gum formed in the carburetor. The carburetor, which showed badly gummed throttle section, was operated for 48 hours using fuel No. 4, and within this time approximately 70 to 80% of the gum present therein had been removed from the carburetor throat. The fuel tanks of this motor launch were made of copper which catalyzed gumming and the fuel was therefore especially susceptible to gum formation.

This application is a division of Serial No. 188,491, filed October 4, 1950.

I claim:

1. An additive for use with motor fuels derived from petroleum in order to produce a motor fuel composition characterized by a relatively low tendency to form gum deposits in the intake system and valves of an internal combustion engine and a relatively low freezing point comprising an oil-soluble monobutoxy poly-1,2-oxypropylene glycol having a viscosity of from about 200 to 1000 SUS at 100° F., and a water-miscible lower aliphatic hydrocarbon alcohol having from one to three carbon atoms in an amount within the range from 20%

to 80% by volume to facilitate the solution of the polyoxyalkylene compound in the fuel and together with the polyoxyalkylene compound to reduce the freezing point of water present in the fuel.

2. An additive for use with motor fuels derived from petroleum in order to produce a motor fuel composition characterized by a relatively low tendency to form gum deposits in the intake system and valves of an internal combustion engine and a relatively low freezing point comprising an oil-soluble monobutoxy poly-1,2-oxypropylene glycol having a viscosity of about 300 SUS at 100° F., and a water-miscible lower aliphatic hydrocarbon alcohol having from one to three carbon atoms in an amount within the range from 20% to 80% by volume to facilitate the solution of the polyoxyalkylene compound in the fuel and together with the polyoxyalkylene compound to reduce the freezing point of water present in the fuel.

3. An additive for use with motor fuels derived from petroleum in order to produce a motor fuel composition characterized by a relatively low tendency to form gum deposits in the intake system and valves of an internal combustion engine and a relatively low freezing point comprising an oil-soluble monobutoxy poly-1,2-oxypropylene glycol having a viscosity of about 300 SUS at 100° F., and a propyl alcohol in an amount within the range from 20 to 80% by volume to facilitate the solution of the polyoxyalkylene compound in the fuel and together with the polyoxyalkylene compound to reduce the freezing point of water present in the fuel.

4. An additive for use with motor fuels derived from petroleum in order to produce a motor fuel composition characterized by a relatively low tendency to form gum deposits in the intake system and valves of an internal combustion engine and a relatively low freezing point comprising an oil-soluble monobutoxy poly-1,2-oxypropylene glycol having a viscosity of about 300 SUS at 100° F., and methyl alcohol in an amount within the range from 20 to 80% by volume to facilitate the solution of the polyoxyalkylene compound in the fuel and together with the polyoxyalkylene compound to reduce the freezing point of water present in the fuel.

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