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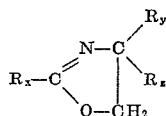
3,033,661

MOTOR FUEL CONTAINING SUBSTITUTED OXAZOLINE COMPOUNDS

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This invention relates to a motor fuel containing a multi-purpose additive comprising a substituted oxazoline compound of the general formula:



wherein R_x represents a saturated or unsaturated hydrocarbon radical containing 7 to 19 carbon atoms, and where R_y represents the radical $-(\text{CH}_2)_n\text{OH}$ where n is a small whole number such as 1 to 3, and where R_z represents a radical selected from the group consisting of R_y , hydrogen, or a lower alkyl radical having 1 to 3 carbon atoms.

Salts of these mildly basic compounds have been suggested by the prior art for incorporation into liquid hydrocarbons and particularly slushing oils, lubricating oils, and grease compositions to provide corrosion inhibiting properties. Exemplary of this art is a corrosion preventive composition utilizing an oxazoline salt of ricinoleic acid, as disclosed in Patent No. 2,587,955.

It has been found, however, in accordance with this invention, that the oxazoline compounds of the general formula assigned above are excellent motor fuel additives in themselves when incorporated into the fuel in amounts as small as 0.0025% by weight. Furthermore, the compounds used in the fuel of this invention fill the criteria of a commercially desirable gasoline additive in that they offer a host of improvements without offsetting these gains with other effects which are of harmful nature, and hence their selection for use is not merely a compromise of gaining certain advantages for the sacrifice of less important characteristics.

The fuel of this invention is particularly helpful in preventing a troublesome condition experienced by most cars operated today in urban areas. It is generally accepted in the art that harmful deposits accumulate in the carburetors of motor vehicles from contaminated intake air which the carburetor must breathe in by tremendous volumes for engine operation. The major contributor to this responsible contamination in the air is believed to be the combustion products exhausted from the large population of other cars operating in the same immediate area. The condition is, of course, aggravated by any blowby fumes from the operation of the car's own engine or by other under-hood fumes generated by the car's engine, in addition to any industrial fumes present in the air which the carburetor breathes. These sources of contamination are additive in nature and may cause the rapid buildup of deposits on the carburetor of the engine.

These deposits manifest themselves by causing rough idling of the engine and in many cases the occurrence of frequent stalling. Although the motor fuel itself is not responsible for these effects, it offers the most likely agency for introducing a material which may serve as a remedy for such an undesirable condition by preventing such deposit buildup in the carburetor.

It therefore is the aim of this invention to provide a fuel containing minor amounts of a class of oxazoline compounds which are effective in preventing deposits from forming in the carburetor of an internal combustion engine which would interfere with the normal operation of such an engine.

tion engine which would interfere with the normal operation of such an engine.

The further value of the fuels of this invention will be appreciated from the following discussion of ancillary benefits derived from their utilization in automotive engines.

Motor fuels containing these substituted oxazoline compounds show marked effectiveness in reducing surface ignition. It is well recognized in the prior art that the continued operation of the internal combustion engine results in the formation of combustion chamber deposits. These deposits produce a number of adverse effects, notable among which is a tendency for these deposits to become heated to incandescence, developing one or more hot spots within the combustion chamber. These spots ignite the fuel either before or after the position in the cycle at which the fuel would normally be ignited by the spark of the sparkplug. This condition, referred to commonly as surface ignition, manifests itself in various ways characterized as different forms of knocking or engine roughness and usually resulting in appreciable power loss. A number of chemical compounds have the facility for eliminating in some degree this adverse effect of chamber deposits. It is believed that these compounds either remove part of the deposits so formed or in some manner modify the chemical nature of these deposits so as to minimize their adversity, or alter the combustion characteristics of the fuel so as to minimize abnormal combustion. Obviously in selecting any additive for gasoline, it is highly desirable that the additive be effective in reducing surface ignition.

Characteristically, compounds which are desirable due to their effectiveness in reducing surface ignition often detract from their value by demonstrating a negative response to octane rating. With the current production costs for octane rating and the increasing pressure for octane improvement in fuels, many compounds, although effective in reducing surface ignition, cannot be justified due to their adverse effect on octane rating. Surprisingly, the oxazoline compounds are an exception to this generalization since their presence in the fuel not only reduces surface ignition but also produces an improvement in octane rating. These compounds as fuel additives thereby offer obvious economic as well as quality advantages.

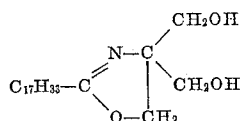
A further benefit that these compounds offer in motor fuels is related to their effectiveness in minimizing carburetor icing. The latter condition represents another troublesome problem attendant to automotive engines operated in cool, moist, atmospheric conditions. This icing problem is most likely to occur during engine warm-up when the engine is below normal operating temperature. Vaporization of the gasoline in the carburetor during this warmup period produces temperature reduction of the throttle plate and carburetor walls, causing the moisture present in the incoming air on cool, humid days to condense and freeze. Such ice formation restricts the narrow openings in the carburetor, manifesting itself in a rough idling condition and frequent engine stalls.

The oxazolines used in this invention may be prepared from amino hydroxy compounds through their fatty amides or by any other procedure. In preparing the oxazolines from the fatty acid amides, a suitable amino hydroxy compound is reacted with a desirable fatty acid or a mixture thereof at an elevated temperature to yield the amide. The temperature is then increased so as to split out water and form the oxazoline. Further information regarding the preparation of these substituted oxazolines using this procedure is contained in Patent Nos. 2,372,409 and 2,372,410.

Any of the compounds having the general formula

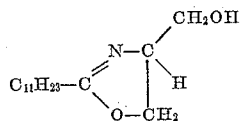
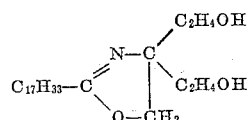
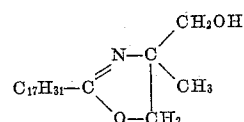
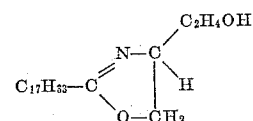
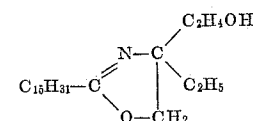
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offered above are suitable for the invention. The preferred compounds, however, are those in which R_x , as defined heretofore, contains 11 to 17 carbon atoms for maximum solubility in hydrocarbon fuels. Mixtures of compounds may be used, and this frequently will be the case since the R_x radical is derived from naturally occurring fats and oils and R_x will correspond to the fatty acid radicals in such fats and oils, such as tallow, cottonseed oil, soybean oil, tall oil, etc. Since the nature of R_x does not affect the results appreciably, its selection will be dictated largely by economics. The commercial grade of the compounds including small amounts of impurities or by-products are suitable. A particularly desirable compound for purposes of the invention is 2-heptadecynyl 4,4-bis-(hydroxy methyl) oxazoline and has the formula:



and is referred to hereinafter as "Oxazoline-T."

Other compounds useful in the invention and which are included as illustrative within the above formula are:



The hydrocarbon fuel base stocks to which these substituted oxazoline compounds are to be added may be any of those conventionally used in preparing a motor gasoline for internal combustion engines, such as catalytic distillate, motor polymer, alkylate, catalytic reformat, isomerate, naphthas, etc. It is intended that the gasoline of the invention may also contain tetraethyl lead in amounts up to 6 ml., generally up to 3 ml., but at least ½ ml. per gallon and a halide scavenging agent. In addition to the lead anti-detonant and a scavenger composition, the gasoline may also contain anti-oxidants, stabilizers, solvent oils, dyes, other additives such as boron compounds, and the like.

The amount of the substituted oxazolines to be added to the fuel may vary between 0.0025% by weight, which is the smallest amount that produces any significant effect with respect to carburetor cleaning and carburetor anti-icing, and 0.1% since amounts in excess of this concentration could not normally be justified economically. Amounts between 0.005% and 0.05% are usually to be preferred.

A clearer understanding of the benefits derived from the fuel of this invention will be obvious from the results obtained from the following testing programs.

Carburetor Cleanliness

A test was devised to determine an effective rating for promoting carburetor cleanliness with the gasoline of the invention as compared with the same gasoline not containing an oxazoline compound. In this test a contamination system was developed to simulate the conditions which contribute to carburetor deposits in the urban operation of motor vehicles. The system comprises the operation of a slave engine and a test engine. The exhaust gases from one bank of the slave engine (four cylinders) is metered to the crankcase of the test engine at a rate of 1.5 cubic feet per minute. All gases from the crankcase of the test engine are passed to the air intake of the carburetor of the test engine including, therefore, the exhaust gases from the slave engine and the blowby fumes of the test engine. The 1.5 cubic feet per minute rate of exhaust gases from the slave engine under these conditions constitutes approximately 8% of the total air intake of the test engine at idle manifold vacuum and speed. The operating conditions for the two engines are as follows:

	Test Engine	Slave Engine
Engines.....	'54 Olds, 10:1 C.R.	'55 Chrys, 11:1 C.R.
Spk. Plugs.....	Champion J-12	Champion N-8B
Carburetion.....	Standard four-barrel Rochester carburetor.	Standard four-barrel Carter carburetor.
Eng. Speed.....	Set up initially for each test at 850 r.p.m., except for accelerations to 3,000 r.p.m., unloaded.	700 r.p.m. ± 20 .
Jacket Temp.....	160° F. $\pm 5^\circ$	165° F. $\pm 5^\circ$.

The test cycle was 2 hours in time, which included four acceleration periods conducted on the unloaded test engine spaced at ½ hour intervals, the first acceleration period being 30 minutes after the test cycle was commenced. During each acceleration period the throttle was moved quickly to a wide open position and then quickly closed again to an idle position five separate times to permit a surge of carbureted gasoline to come into contact with the carburetor parts. The carburetor at the start of each test was in spotless condition. All experimental conditions were the same for the two test cycles except for the gasoline.

The base fuel in each test was the same and had the following composition and specifications.

Composition:

75% cat. distillate
25% SR naphtha

API gravity 62.4

Engler distillation:

IBP °F. 98
10% °F. 130
30% °F. 163
50% °F. 204
70% °F. 262
90% °F. 365
EP °F. 424

Reid vapor pressure..... 8.75

At the end of each test the carburetor was removed and disassembled and a numerical rating was assigned for amount of deposits and discoloration by a number of observers rating independently and uninformed of the gasoline they were rating. The rating assigned was based on a standardized scale ranging from 100 to 0, wherein 100 would be a rating of a perfectly clean carburetor throttle plate and barrel, and 0 rating would represent a throttle plate and barrel loaded with deposit.

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The results below represent the average rating by the observers:

Additive	Rating
Oxazoline-T at 0.05% by weight.....	93
None.....	60

Surface Ignition Suppression

To determine the effectiveness of the gasoline of this invention to suppress surface ignition, an ASTM-CFR single-cylinder engine having the compression ratio adjusted at 12:1 was employed. Preparatory to the test cycle, the engine was run open throttle at 900 r.p.m. for thirty minutes to stabilize the engine conditions for the test period. Following this, the engine was continued at open throttle continuously for three hours, during which time the total surface ignition count was observed electronically. All experimental conditions were the same for each test except the gasoline.

The base fuel in each test was the same and had the following composition and specifications.

Composition:	
59.8% ultraformate	
30.0% cat. distillate	
10.0% lt. naphtha	
0.2% solvent oil	
API gravity.....	49.7
Engler distillation:	
IBP.....°F..	80
10%.....°F..	155
30%.....°F..	202
50%.....°F..	263
70%.....°F..	310
90%.....°F..	377
EP.....	452
Reid vapor pressure.....	6.5
Tetraethyl lead (motor mix).....ml./gal..	3.0

The number of surface ignitions in the additive fuel is expressed as percentage of the surface ignitions of the blank fuel with the results as follows:

Additive	SI Rating of Fuel, percent	
	Total SI	Audible SI
None.....	100	100
Oxazoline-T at 0.1%.....	51	89

Octane Improvement

Two fuels having the compositions and physical characteristics as listed below were tested for octane rating by the F-1 and F-2 ASTM test methods with and without the oxazoline additive.

	Fuel A	Fuel B
Composition.....	84% Cat. Reformate 16% Lt. Naphtha	59% Lt. Alkylate 30% Benzene 11% Toluene
API Gravity.....	49.1	52.7
Engler Distillation:.....	° F.	° F.
IBP.....	114	120
10%.....	153	168
30%.....	206	186
50%.....	250	196
70%.....	296	208
90%.....	356	232
EP.....	446	296
Reid Vapor Pressure.....	6.96	5.05
Tetraethyl Lead (Motor Mix).....ml./gal..	3.1	3.1

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The results of these tests are indicated below:

	Octane Rating	
	F-1	F-2
Fuel A.....	99.56	90.85
Fuel A + Oxazoline-T at 0.05% by Weight.....	99.69	91.04
Fuel B.....	107.66	101.18
Fuel B + Oxazoline-T at 0.05% by Weight.....	107.61	101.34

In the 99+ octane fuel, typical of premium fuels today, both the F-1 and F-2 ratings are improved. The numerical improvements indicated in these results are not large; yet considering the high octane levels of these base fuels, any further increase in octane number, particularly in the F-2 method, is significant. The important thing is that the oxazoline compounds, contrary to the observation with many compounds that suppress surface ignition such as many phosphorus compounds do not lower the octane rating of the fuel.

Carburetor Anti-Icing

A test procedure was devised simulating the stop-and-go type of engine operation normally experienced by the motorist during the engine warmup period. The test was conducted in a 1955 Plymouth V-8 engine equipped with a two-barrel carburetor. Carburetor air was supplied at a constant rate of 70 cubic feet per minute by a specially designed air conditioner controlled at 42° F. and 90% relative humidity, which are temperature and humidity conditions considered highly conducive to carburetor icing. All test conditions were the same except for the gasoline.

The test consisted of running the same number of cycles on each fuel where in each cycle the engine was operated at 2200 r.p.m. for 15 seconds and then decelerated normally to an idle at 450 r.p.m. for a maximum of 30 seconds. Performance of the engine was observed during each idle period, and a numerical rating based on the degree of rough idling and engine stalls was assigned so that each fuel received a merit rating on a scale ranging from 100 to 0. By this scheme an engine operating with a smooth idle over the idle periods of every test cycle would receive a rating of 100, and an engine which stalled in less than 12 seconds in the idle period of every test cycle would receive a rating of 0.

The base fuel used had the following composition and specifications.

Composition:	
48.5% lt. cat. distillate	
28.9% Ultraformate	
9.7% lt. naphtha	
4.9% isopentane	
7.8% butane	
0.2 solvent oil	
API gravity.....	61.7
Engler distillation:	
IBP.....° F..	93
10%.....° F..	120
30%.....° F..	160
50%.....° F..	202
70%.....° F..	250
90%.....° F..	325
EP.....° F..	420
Reid vapor pressure.....	11.75
Tetraethyl lead (motor mix).....ml./gal..	3.0

The results of this test are as follows:

Additive	Rating
None.....	58
0.0035% Oxazoline-T by Weight.....	76
0.02% Oxazoline-T by Weight.....	92

Since these compounds are readily soluble in gasoline, they may be incorporated directly into bulk gasoline at a refinery by standard mixing equipment. If a motorist wishes to use the compound in a fuel not made with the same at the refinery, a preferred method for incorporating these compounds into the motor fuel is in solution in a solvent compatible with gasoline. An appropriate volume of this solution may then be added to a filled fuel tank of a motor vehicle to provide the desired concentration of oxazoline. For example, if 12 ounces of solvent with 5% by weight of the oxazoline compound is added to 10 gallons of gasoline, the latter will contain approximately 0.05% by weight of the oxazoline compound. Similarly, if 12 fluid ounces of solvent with 0.25%, 0.5%, and 10% by weight, respectively, of the oxazoline compound are added to 10 gallons of gasoline, the latter will contain approximately 0.0025%, 0.005%, and 0.1% by weight, respectively, of the oxazoline compound. The solvent may be any hydrocarbon such as benzene or preferably any lower aliphatic alcohol having 1 to 4 carbon atoms, especially methanol and iso-propanol.

A particular advantage that is to be gained by the use of the combination of a lower aliphatic alcohol and one or more of the foregoing class of oxazoline compounds is in reducing operational distress of the engine due to ice formation in the engine's fuel supply system. It is, of course, well known that commercial gasolines contain small amounts of water. Some moisture also tends to accumulate in the individual tanks of cars, and hence water is always present in the fuel passing through the engine's fuel supply system. During operation at atmospheric temperatures lower than 32° F., and particularly at temperatures below 20° F., this water content freezes and ice crystals have a tendency to form in the fuel system. Such ice formation restricts or blocks the small diameter fuel line or the fuel filter where small openings are present for the passage of the fuel to the engine; and due to this blockage or restriction to flow, the engine can no longer operate, or operation is seriously impaired.

In like manner, commercial hydrocarbon fuels for diesel engines and jet engines also contain certain amounts of water, and hence a problem of the same nature with regard to the fuel supply lines of these engines is experienced for these fuels. Therefore, since the class of oxazoline compounds described herein are soluble and compatible in both diesel fuel and jet fuel, the following discovery is intended to have application in these fuel compositions as well as in gasoline compositions for automotive engines.

The lower aliphatic alcohols or mixtures thereof, particularly methanol and isopropanol, have received widespread acceptance for use in gasoline to protect against the fuel system freezing problem described above. The alcohol serves as a freezing point depressant and its function in this regard varies linearly with concentration. For this reason, in order to prevent fuel system freezing at low temperatures, considerable amounts of the alcohol must be incorporated and the economics of obtaining such protection is unattractive. It has been found as a part of the present invention that the presence of only a small amount of one or more of the foregoing described class of oxazoline compounds in a gasoline composition containing a lower aliphatic alcohol, particularly methanol, isopropanol, or mixtures thereof, produces a significant coaction in protecting against the blockage or restriction of the engine's fuel system by ice so that the amount of alcohol which must be added to the fuel to obtain protection to any desired temperature level of operation may be greatly reduced. Fortuitously, the coaction of the additive combination is particularly effective in the temperature range where, as a practical consideration, protection against fuel line freezing is most desired; namely, from 20° F. down to -20° F. At ambient temperatures lower than -20° F. the coaction between the alcohol and oxazoline compound diminishes. Although

the actual mechanism is not clearly understood, the oxazoline compound in some manner acts as a conditioning agent in conjunction with the alcohol in reducing the tendency of the ice forming in the engine's fuel system from causing operational distress. The amount of alcohol to be added to the fuel to give fuel line protection will vary depending upon the alcohol used. The amount required increases with the molecular weight of the alcohol. At least 0.1% by weight alcohol is required, however, to offer any significant improvement. Usually amounts over 2% cannot be justified and a preferred range will be 0.1% to 1% by weight. The amount of oxazoline compound may also vary, but at least 0.001% by weight oxazoline is required to produce the desired coaction with respect to fuel line freezing protection. Larger amounts may be used without adversely affecting the coaction and it may therefore be desirable, particularly in gasoline compositions, to use amounts of 0.0025% by weight or higher to gain the further advantages accompanying the presence of oxazoline compounds, such as carburetor cleaning, carburetor anti-icing, and surface ignition suppression. It will be obvious, therefore, that the concentrate of alcohol and oxazoline compound in the proportions previously described will provide the desired quantity of the two materials to the fuel when added in the manner and amount already stated. It is to be understood, however, that the alcohol and oxazoline compound may be added to the base hydrocarbon fuel separately if so desired.

To demonstrate the coaction of the oxazoline compounds with lower aliphatic alcohols in preventing operational distress due to ice formation in the fuel system, a fuel handling system simulating that associated with an automotive engine was devised and adapted to be run in a cold box having a volume of approximately 2 cubic feet. This cold box is cooled with Dry Ice and a blower is present within the box so that the cold air may be circulated and the temperature maintained uniform. In the fuel system provided in the box a fuel tank is connected to a fuel filter by approximately 190 inches of 5/16-inch copper tubing, a fuel line length, similar to the fuel line in an automobile. A U-tube section is located in the fuel line immediately after the fuel tank, similar to the type provided in the fuel system of a car as a water trap. The fuel filter has a glass bowl so that ice formation at this point in the system may be visually detected. An electric pump is connected to the fuel filter by a short section of copper tubing. The outlet side of the pump is connected to a single-barrel carburetor by another short section of tubing in which a flowmeter (Rotameter) is located to measure the flow rate of fuel through the system. A flow valve is adapted to the carburetor to return fuel to the fuel tank. Windows are provided in the cold box at the areas where the fuel filter and the flowmeter are located so that each of these elements may be observed by the operator during the test.

A series of gasolines were tested in the above equipment to determine the temperature at which operational distress would occur due to freezing conditions within the fuel system. One set of samples comprised a composition of a base gasoline containing different alcohols in varying amounts. For comparison, another set of samples comprised a composition of the same base gasoline containing the combination of an alcohol and an oxazoline compound in varying amounts. The base gasoline for each sample was a commercial regular grade of gasoline containing 1% by weight water. Distress condition for the fuel system was determined by noting when the fuel flow as indicated on the flowmeter fell below a predetermined value, together with noting ice formation in the glass bowl of the fuel filter. In testing each composition, the temperature was reduced gradually in the box so as to avoid supercooling of the fuel mixture. The temperature at which distress conditions were experienced for the base fuel without any additive was found to be 29° F. From the test run on the base fuel samples

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containing additives, the amount of additive required to give protection against fuel system freezing to 20° F., 10° F., 0° F., -10° F., and -20° F. was determined and the results are indicated below:

Protection to ° F.	Methanol, Wgt. Percent	Combination		Isopropanol, Wgt. Percent	Combination	
		Methanol, Wgt. Percent	Oxazoline-T, Wgt. Percent		Isopropanol, Wgt. Percent	Oxazoline-T, Wgt. Percent
20-----	0.17	0.04	0.001	0.31	0.08	0.001
10-----	0.40	0.15	0.001	0.70	0.25	0.001
0-----	0.60	0.25	0.001	1.10	0.45	0.001
-10-----	0.80	0.40	0.001	1.50	0.75	0.001
-20-----	1.00	0.90	0.001	1.90	1.70	0.001
20-----	0.17	0.04	0.0025	0.31	0.08	0.0025
10-----	0.40	0.15	0.0025	0.70	0.25	0.0025
0-----	0.60	0.25	0.0025	1.10	0.45	0.0025
-10-----	0.80	0.40	0.0025	1.50	0.75	0.0025
-20-----	1.00	0.90	0.0025	1.90	1.70	0.0025

To confirm the above results, a test was conducted on a standard 1956 Buick Century automobile operated in a cold room. In this test the gasoline introduced into the car's fuel system contained 0.4% by weight water. The distress condition in this test was determined by engine failure as the temperature was gradually reduced. The distress temperature for the base fuel without any additive was found to be 25° F. The amount of methanol required to be added to the gasoline to give protection to 0° F. was determined and compared to the amount of methanol and Oxazoline-T that must be added to the base fuel to give protection to 0° F. The results are reported below:

Protection to ° F.	Methanol, Wgt. Percent	Combination	
		Methanol, Wgt. Percent	Oxazoline-T, Wgt. Percent
0-----	0.55	0.25	0.0025

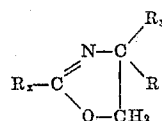
It is to be understood that various modifications of the foregoing invention will occur to those skilled in the art upon reading the above description. All such modifications are intended to be included as may be reasonably covered by the appended claims.

The present application is a continuation-in-part of application Serial No. 717,549, filed February 26, 1958, and it is intended that the entire contents of said latter application be incorporated herein by reference.

I claim:

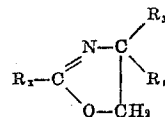
1. A liquid hydrocarbon fuel adapted to prevent freezing of fuel lines containing from 0.1% to 2% by weight a lower molecular weight alkyl alcohol and at least 0.001% by weight a substituted oxazoline compound of the formula:

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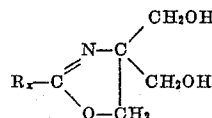
wherein R_x represents an aliphatic hydrocarbon radical containing 7 to 19 carbon atoms and where R_y represents the radical $-(\text{CH}_2)_n\text{OH}$ where n is a small whole number and R_z represents a radical selected from the group consisting of R_y , hydrogen, and a lower alkyl radical having 1 to 3 carbon atoms.

2. A gasoline adapted to prevent freezing of fuel lines containing $\frac{1}{2}$ to 6 cc. of tetraethyl lead per gallon, 0.1% to 2% by weight a lower molecular weight alkyl alcohol and at least 0.001% by weight a substituted oxazoline compound of the formula:



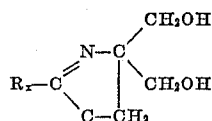
wherein R_x represents an aliphatic hydrocarbon radical containing 7 to 19 carbon atoms and where R_y represents the radical $-(\text{CH}_2)_n\text{OH}$ where n is a small whole number and R_z represents a radical selected from the group consisting of R_y , hydrogen, and a lower alkyl radical having 1 to 3 carbon atoms.

3. A gasoline adapted to prevent freezing of fuel lines containing $\frac{1}{2}$ to 6 cc. of tetraethyl lead per gallon, a halide scavenging agent, 0.1% to 1% by weight methanol, and at least 0.001% by weight a substituted oxazoline compound of the formula:



wherein R_x represents an aliphatic hydrocarbon radical containing 11 to 17 carbon atoms.

4. An additive concentrate consisting essentially of 95% to 99.5% by weight of a lower aliphatic alcohol and 5.0% to 0.5% by weight of a substituted oxazoline compound of the formula:



wherein R_x represents an aliphatic hydrocarbon radical containing 11 to 17 carbon atoms.

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