

PATENT SPECIFICATION

NO DRAWINGS

822,278



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COMPLETE SPECIFICATION

Gasoline Containing Organic Boron Compounds

We, THE STANDARD OIL COMPANY, of Midland Building, Cleveland 15, Ohio, United States of America, a Company incorporated under the Laws of the State of Ohio, United

5 States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to gasoline containing organic boron compounds as hereinafter explained.

15 It is well known that during the operation of an initially clean internal combustion engine, deposits form progressively and accumulate on the surfaces within the combustion zone, i.e. on the cylinder head, piston top, spark plug, and the intake and exhaust valves. As the operation continues, the amount of the deposit reaches a level after which there is no appreciable further increase in the amount of the deposits. The engine is then regarded as having deposit equilibrium and old deposits flake off as fast as new deposits form.

25 The deposit problem is aggravated when tetraethyl lead is contained in the fuel because the deposits are then no longer essentially carbonaceous, but comprise appreciable quantities of lead and lead compounds generally mixed in with the carbonaceous material. Despite the fact that one or more organic halides are included in the fuel with the tetraethyl lead to serve as a scavenging agent, the lead is not all removed. A carbonaceous deposit with lead is often more tenacious and troublesome than a pure carbonaceous deposit. The fact that this deposit is partially metallic in character is thought to give it a catalytic activity which modifies the action of the deposit in affecting engine operation. Furthermore, since the lead is intimately mixed in which the carbonaceous material, the lead in the deposit is difficult to reach by means of scavengers or material reactive therewith to eliminate or modify it, especially in engines

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Price 4s 6d.

which have been in operation and which have such deposits built up in them over a long period of time.

These deposits have a number of adverse effects upon engine operation, but with available fuels they cannot be avoided and are inherent in present day operation of internal combustion engines. One adverse effect of the deposits is manifested most clearly to the operator of the engine, particularly an automobile engine, by the fact that the presence of the deposits in the engine requires a fuel having a higher octane rating in order not to knock than is required by a new or clean engine. This requirement for a fuel of higher octane number, as the engine becomes progressively dirtier, persists until the deposits reach equilibrium, and is known as octane requirement increase (ORI). Another factor to be considered is the reduction in the volume of the combustion zone equivalent to the volume of the deposits and the consequent increase in compression ratio which aggravates the above manifestation.

Another adverse effect of the deposits is characterized by uncontrolled ignition, either pre-ignition or post-ignition. This is explained by the fact that the deposits, or portions of them, become heated to incandescence and ignite the hydrocarbon either before or after the portion in the cycle at which the charge would be ignited by the spark of the spark plug. This manifests itself in various ways characterized as "roughness," "rumble," various forms of knocking, and a general lack of smoothness in engine operation.

Another adverse effect of the deposits is on the spark plug, particularly when the deposits contain lead which results in spark plug fouling with resultant loss in power or reduced spark plug life.

Still another adverse effect of the deposits is the result of their thermal insulating property. They are poorer conductors than the metal parts of the engine and as a result they interfere with proper cooling and temperature

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maintenance of the engine parts. This is particularly true in the case of the exhaust valves which become overheated and burn or otherwise deteriorate as the result of the deposits.

5 A serious adverse effect of the deposits is the channeling of exhaust valves. This is explained by the fact that the deposits flake off as fast as they are formed once the deposit equilibrium is reached, and the flakes leave through the exhaust valves. The glowing deposits expelled from the silencer have been seen by all who drive at night. As these deposits leave through the exhaust valve they may lodge between the valve and the seat, thereby interfering with the proper closing of the valve. The hot exhaust gases passing between the valve and the seat result in channeling. Failure of the valve to seal the combustion chamber properly results in loss of acceleration, power and mileage. Somewhat analogous results occur in connection with the inlet valves, but because these valves are cooler the character of the deposits is often different, and while not as severe, is nevertheless a problem in engine operation. The action of fuel and the deposits of valve life and condition has been largely overlooked or minimized in evaluating fuels, and since the valve action is vital to proper engine operation, this phase of the matter deserves special attention.

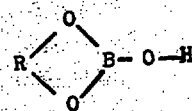
Organic boron compounds, if they are stable under many conditions of use, are usually of relatively high molecular weight and have a relatively low boron content. As such, they are not adapted for many uses where their suitability depends on a high proportion of boron in the compound. In addition, organic boron compounds which do have a substantial boron content, as a class, are generally unstable and do not satisfactorily resist oxidation or hydrolysis. The compounds employed according to the invention have the advantage of a relatively high boron content and, at the same time, relatively good stability. They are particularly resistant to oxidation and are resistant to hydrolysis to form boric acid, a property not possessed by the common organic esters of boric acid.

50 In addition, the organic boron compounds of the invention are moderately soluble in hydrocarbons as well as water and do not precipitate from gasoline in which they are dissolved even though such gasoline is stored for long periods in contact with water. This is to be contrasted with many boron compounds which hydrolyze so readily that they would be eliminated from gasoline when the latter is stored in contact with water as is usually the case at bulk storage plants and in the tanks of individual cars.

Thus, the boron compounds of the invention have such desirable characteristics as solubility, volatility and chemical structure as to render them soluble in the fuel and effective

in the combustion chamber.

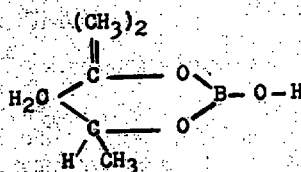
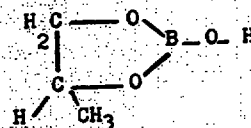
The boron compounds which are employed in the fuel of the invention to meet the above objects are of the general formula:



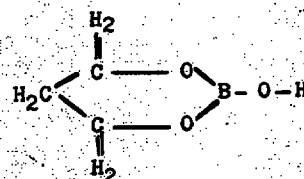
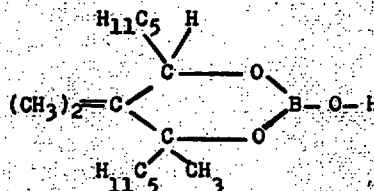
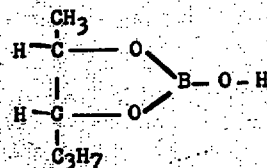
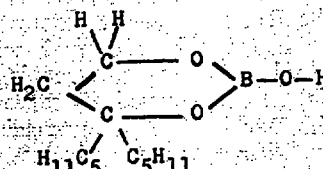
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where R is an alpha or beta alkylene radical having from 3 to 20 carbon atoms.

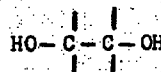
Examples of such boron compounds are:



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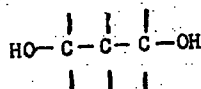
The compounds used in the fuel of the invention may be made by reacting orthoboric acid with the corresponding alpha glycol having the formula



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or the betaglycol having the formula



in which formulae the unattached valencies are linked to hydrogen or to straight chain or branched alkyl radicals, to provide a total of 3 to 20 carbon atoms in the glycol. The glycol and boric acid are reacted in equimolar quantities.

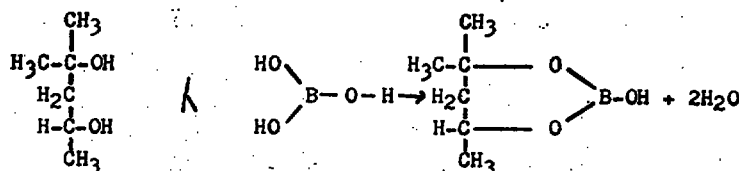
The materials are reacted at mildly elevated temperatures to split off the water of reaction and to remove it, or at least part of it, from the reaction system; thus in certain cases it is possible to separate remaining water after completion of the reaction, for example by simple decantation from a solid boron compound product. The water split off is preferably separated overhead, such as by blowing with nitrogen or air or by distillation. All the water can be removed by simple boiling. However, because of the low boiling points of many of the compounds a certain amount thereof may be lost due to volatilisation during the removal of water. For this reason the complete removal of water from the reaction system may not be preferable unless

it is done by means of fractional distillation or azeotropic fractional distillation, which permits the removal of the water with a minimum loss of the reaction product. The reaction proceeds readily, 2 mols of water being split off for each mol of glycol and boric acid. The water of reaction removed overhead by means previously described may be accurately measured so that the formation of the compounds may be readily controlled when the glycol and boric acid are reacted in equimolar proportions.

The following Examples illustrate the production of the compounds which may be used in accordance with the present invention:

EXAMPLE 1.

320 Grams of dry boric acid and 620 grams of 2-methyl, 2,4-pentanediol (a 1:1 molar ratio) were mixed together. Upon mixing, the temperature dropped approximately 8 to 10° C., and upon additional stirring, the temperature then rose rapidly to and slightly above the initial temperature, at which time the mixture became solid. The mixture was then heated and became completely liquid at about 70 to 75° C. Upon cooling a solid was formed which was separated from the aqueous mother liquor. The reaction proceeds as follows:



The crystalline product was recrystallised twice from straight-run naphtha (a hydrocarbon having a boiling range of 43 to 127° C.) and once from normal heptane. The product was a white crystalline solid and had a melting point of 74 to 76° C. It may be recrystallised from water. The compound has the formula $\text{C}_6\text{H}_{13}\text{BO}_3$ and may be named 1-hydroxy, 3,3,5-trimethyl, 1-bora, 2,6-dioxyacyclohexane or 1,1,3-trimethyltrimethylene acid borate. The compound has a solubility of 11 grams in 100 grams of water, 58 grams in 100 grams of benzene, 156 grams in 100 grams of isopropanol, and about 2½ to 5 grams in 100 grams of gasoline, depending upon the composition of the latter, all as measured at 86° F.

The compound was analysed and found to have the following composition:

	Calculated	Found by Analysis
% Carbon	50.05	50.01
% Hydrogen	9.10	9.21
% Boron	7.52	7.43
Molecular Weight	143.97	155.

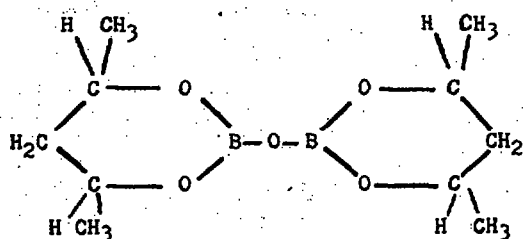
The boron determination was made by titration in the presence of mannitol, and the molecular weight was done cryoscopically in benzene. Infra red analysis confirmed the presence of the hydroxyl group, and repetition under conditions permitting recovery of the water of reaction showed that two moles of water were formed.

EXAMPLE 2.

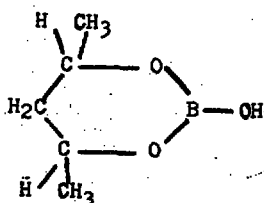
The compound of Example 1 can also be prepared by contacting with water the anhydride of Example 1 of Application No. 14673/58 (No 822,279) as described therein.

EXAMPLE 3.

52 Grams pentanediol-2,4 is reacted with 31 grams of dry orthoboric acid under conditions the same as those of Example 1 of Application No. 14673/58 (No. 822,279). The product was a clear, colourless, viscous liquid having a boiling point of 114° C. at 1 mm. Hg. It has the formula



The yield, which was 61 grams, was vacuum distilled at 1 mm. Hg. to give a yield of 55.8 grams. On reaction with water, the compound gives a product that is a crystalline solid, having a melting point of 76° C. and which has the formula



Fuel containing the boron compound should be and is sufficiently stable to be stored in contact with moisture for three months without loss of boron content.

The gasoline base stocks to which the boron compounds are added may be any of those conventionally used in making motor gasoline (gasoline for use in an automobile).

The gasoline of the invention also contains tetraethyl lead in amounts up to 6 cc., but usually from $\frac{1}{2}$ cc., per United States gallon, and a halide scavenging agent. The latter may be 1 theory of ethylene dichloride and $\frac{1}{2}$ theory of ethylene dibromide (the so-called "motor mix" or MM) or 1 theory of ethylene dibromide (the so-called "aviation mix" or AM). Reference to MM and AM hereinafter is intended to refer to a gasoline containing the scavenging agent in the amount above recited. By "theory" is meant the stoichiometric amount of the ethylene dihalide for combination with all of the lead as lead halide. The AM is preferred since in combination with the boron compound there results a better overall effect in engine performance. The gasoline may contain any of the usual adjuncts such as anti-oxidants, dyes, solvent oils or alcohols.

The amount of the boron compound added to the fuel may vary and the amount is preferably expressed in terms of boron. Generally an amount of the compound to provide 0.002% by weight of boron (based on the total fuel) is the smallest amount that will give any significant effect. Amounts between 0.004% and 0.008% by weight are preferred. Use of amounts in excess of 0.1% usually cannot be justified economically.

The fuel containing an amount of the compound of Example 1 to provide 0.004% boron was extensively tested. This compound was selected in order that all the tests may be comparative. Reference hereinafter to "Boron" is intended to refer to a gasoline containing this compound in the above amount specified.

The base stock in all tests was the same, namely a mixture of straight run naphtha and catalytic distillate in the ratio of 1:3 with 3 cc of tetraethyl lead per gallon to give an octane number of 94.

Two different scavenging agents were employed, namely the MM and AM as defined previously.

The gasolines containing Boron gave a significant reduction in octane requirement increase. This property of boron in gasoline is not particularly important today in view of the high octane number gasolines generally available which satisfy the demands of most cars as far as octane number is concerned. For this reason the effect of the boron compound was evaluated more critically in other respects.

Inasmuch as the deposits in an engine are the cause of most of the difficulties, a test was devised to determine the amount of the deposits formed with gasoline of the invention as compared with the same gasoline not containing boron. In this test conditions were employed which encourage deposit formation, namely the mild or low duty use analogous to city driving. A Chevrolet engine was used, running on a cycle of one minute idle and five minutes at 2,000 rpm at 11.8 brake horsepower. This light load and relatively low speed is similar to city driving interrupted by traffic light stops. The test was continued for 100 hours and the engine dismantled and the amount of deposit ascertained. All experimental conditions were the same except for the gasoline. The total combustion chamber deposits per engine in this test are expressed in grams and are as follows:

Additive	Scavenger	
	MM	AM
Boron	57.6	57.6
None	60.5	67.1

The amount of the deposit on the piston top was separately ascertained, with the following results:

Additive	Scavenger	
	MM	AM
Boron	29.5	20.9
None	34.2	28.3

The amount of the exhaust valve tulips was separately ascertained inasmuch as these deposits have an important bearing on the operation of the engine and the reduction in them is significant.

Additive	Scavenger	
	MM	AM
Boron	0.55	0.30
None	3.10	1.55

5 It will be noticed that the presence of Boron gave a lower deposit when used with both scavengers, although the lowest deposits were obtained with the AM scavenger.

10 To determine the effect of Boron on pre-ignition and related manifestations, a high compression Oldsmobile engine having a compression ratio of 10:1 was employed. In some cars on the road today the compression ratio is so low as not to make pre-ignition a problem, but there is a trend to higher compression ratios, and many cars are troubled

by pre-ignition. In this test the engine was run for 28 minutes under idling conditions, which would bring up fresh deposits. Following this the engine was accelerated for two minutes under a load simulating climbing a hill. The number of "wild pings" was observed electronically and was counted, following which the ignition switch was turned off and the seconds noted that the car continued to run with the ignition off. During this running time the fuel was being ignited by the heat of the deposits. Also the number of "wild pings" were similarly counted during this after-running period. All conditions were the same for each test except the fuel. The results are as follows:

	Fuel	"Wild Ping" with switch on	Seconds of After-running	"Wild Ping" with switch off
35	MM	145	44	155
	MM plus Boron	137	37.5	131
	AM	12.5	36.5	93.5
	AM plus Boron	0	27	63

40 From the above data it will be seen that Boron improves the motor mix in every instance, although the aviation mix gives such good results with the switch on so as to leave little room for improvement. Even here Boron improves the operation. The ability to eliminate "wild ping" by the combination of AM and Boron is especially noteworthy.

45 The effects described above and the manifestations of deposits in the general field of pre-ignition are subtle in detection and expression. They are characterized as both audible and inaudible, with and without rumble. A general lack of smoothness in operation accompanies any of these manifestations which is difficult to characterize quantitatively in figures. However, subjective appraisal by persons comparing fuels with and without the boron compound, even in lower compression engines, noted an increase in smoothness of operation as one of the characteristics most readily noted by the motorist.

50 In a further test, four fuels, i.e. the same base with MM and AM, each with and without the Boron, were subjected to a test on the Pennsylvania Turnpike, employing twelve 55 1954 model cars, two each of Ford, Buick, Century, Oldsmobile 88, Chevrolet, Chrysler New Yorker, and Cadillac. Each car was run 10,000 miles on the same gasoline, and each car was run on two gasolines, and each gasoline was tested in each type of car for the same number of miles, to give a total of 240,000 miles. The test was set up statistically to eliminate variations between cars, the details of the method being given in "Experimental Designs" by Cochran and Cos, Section 11.51. The cars were examined before and after the 10,000 mile run on a single gasoline. The valves were replaced at the end of 10,000 miles. The cars were driven at a

uniform 60 miles per hour and were driven 400 miles continuously each day with an intermediate stop for refueling. All conditions were the same except for the fuels. This was a heavy duty test in which the exhaust valves would be subject to the most strenuous operating conditions and which therefore were examined with care and will be considered first. The average final statistical ratings in grams of the deposits on the exhaust valve heads are as follows:

Fuel	Deposits
MM	1.26
MM plus Boron	1.23
AM	1.23
AM plus Boron	1.14

In this instance the Boron seems to be more effective in reducing exhaust valve head deposit with AM.

Similar data for exhaust valve tulips are as follows:

Fuel	Deposits
MM	1.60
MM plus Boron	1.44
AM	1.04
AM plus Boron	1.09

In this instance the Boron appears to be more effective with the MM, probably because the AM is already about as effective in reducing exhaust valve tulip deposits as is reasonable to expect.

Another important observation from the standpoint of the operation of the car is valve channeling. Channels worn on the valve faces permit the fuel to be expelled through these channels during the compression stroke and



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to permit a part of the hot gases to be expelled during the power stroke, with a resultant loss of power and mileage. The extent of channeling is determined by removing all the valves from the car and examining the number of degrees around the circumference of all of the valves of a single car that are channeled. The final statistical ratings are as follows:

	Fuel	Degree of Valve Channeling
10	MM	134
	MM plus Boron	84
	AM	13
	AM plus Boron	15

	Car	Fuel	Total Deposit
30	Chevrolet	MM	13.2
		MM plus Boron	12.7
	Ford	AM	16.6
		AM plus Boron	15.5

Similar improvements in cylinder heat deposit weight were obtained.

35 Another factor that was noted is oil mileage, with the following results:

	Fuel	Miles Per Quart of Oil
40	MM	1506
	MM plus Boron	1788
	AM	2007
	AM plus Boron	1319

45 The addition of the Boron compound significantly improves the oil mileage with the MM but not with the AM.

As indicative of the engine wear in operating on different gasolines, the cylinder bore was measured before and after the test. Average statistical ratings were calculated and are listed in the following table in which the increase in cylinder bore diameter is expressed in units of 10^{-4} inches.

	Fuel	Cylinder Bore Increase
55	MM	6.27
	MM plus Boron	6.48
	AM	4.24
	AM plus Boron	2.67

60 In this instance the Boron does not reduce the wear in the case of MM but the increase is small. Boron significantly reduces wear in the case of AM, which is already much lower than MM.

65 Each car was measured for acceleration after being driven for 10,000 miles on a single gasoline and this acceleration was expressed in terms of the number of seconds it required a car to increase from 22 miles an hour to 60 miles an hour on the flat open road with full open throttle. A decrease in the amount of time shows that the car is able to develop greater power and shows better mechanical condition of the car. The statistical results are as follows:

15 In this instance the Boron reduces the amount of channeling in the MM formulation. The AM formulation, however, is so good in this respect that little improvement can be expected. While the above are the statistical results, in a single car in which AM and AM plus Boron was used, a 1954 Ford, the run of AM gave valve channeling of 120° and the run on AM plus Boron gave 0° valve channeling.

20 The total combustion chamber deposits in grams in this heavy duty test were reduced as in the light duty tests, typical examples being:

	Fuel	Time to Accelerate
75	MM	14.62 seconds
	MM plus Boron	14.56 seconds
	AM	14.92 seconds
	AM plus Boron	14.70 seconds

80 From the above data it will be seen that the Boron improves the ability of the car to accelerate with both scavenging agents. The improvement is most notable in the case of addition of AM to Boron, although the final acceleration is not quite as good as the acceleration of MM without Boron.

85 The mileage obtained by the various gasolines was noted, with the following results:

	Fuel	Miles per Gallon
90	MM	15.87
	MM plus Boron	15.95
	AM	16.01
	AM plus Boron	15.93

95 d From the above data it will be seen that the addition of Boron to MM increases the mileage but that this was not the case on the addition of Boron to AM. However, the mileage of AM plus Boron, which has all the advantages inherent in the use of AM, is superior to use of MM alone, which is the fuel heretofore commercially available to the motorist.

100 The spark plugs were examined at the end of the tests and no fouling was observed. The ability of the Boron to increase acceleration and mileage shows that Boron has an advantageous effect on power output, and this may be attributed in part to inhibition of spark plug fouling.

105 From the above data it will be noted that in some instances Boron coacts with the MM to give an improvement and in other cases it coacts with AM to give an improvement. The action is not always consistent. The scavenger to be selected will depend upon the items of improvement believed to be the most important. It is believed that the factors in



which Boron improves AM are more important and that the final results for this combination are more significant to the motorist. For this reason this combination is preferred, despite the greater cost of the AM scavenger. If one is to use AM as the best method of reducing valve channeling, the addition of Boron thereto gives improvements in many respects over AM alone, such as in acceleration and engine wear.

In instances, however, where the manufacturer will not want to increase his cost by substituting AM for MM, the use of Boron with MM will result in the many improvements noted in the data.

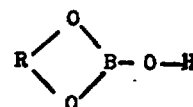
In addition, the cars driven on the Turnpike were examined for other properties, such as silencer deposits, silencer corrosion, inlet valve deposits and piston ring weight loss, all the details of which it is believed unnecessary to include. In most instances there was an improvement. In those cases where there was not, the adverse effect was so small as not to outweigh the desirable properties described above.

In addition to the above tests, the fuel with MM and the fuel with AM plus Boron were extensively tested in two large groups of employee cars. In these tests the MM with Boron and AM without Boron were not tested because these tests were run to evaluate a proposed substitution of AM plus Boron as a new fuel to replace MM. Each employee did not know whether the fuel he was using was the regular fuel or the proposed new fuel. Because of the difficulty of keeping accurate controls, details of the test are not included, but they showed in general an increase in mileage, less valve deposits, lower octane requirement, reduction in ring wear, and an increased alkalinity of oil after the equivalent of 1500 miles driving. Inasmuch as the employee did not know which fuel he was using, opinion surveys can be considered as valid, and these showed more comments favourable for the AM plus Boron than for MM. In addition, a survey from many

thousands of motorists showed that two out of three detected an immediate improvement upon changing from MM to AM plus Boron. The most predominant improvement rated was a smoothness of operation, despite the fact that the octane number was the same.

WHAT WE CLAIM IS:

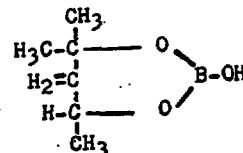
1. Gasoline to which has been added up to 6 cc. per United States gallon of tetraethyl lead, a halide scavenging agent, and at least one boron compound of the general formula:



wherein R is an alpha or beta alkalene radical having 3 to 20 carbon atoms, the amount of said compound added to the gasoline being equivalent to 0.002 to 0.1% by weight boron.

2. Gasoline as claimed in claim 1, in which the scavenging agent is one theory of ethylene dibromide per theory of tetraethyl lead.

3. Gasoline as claimed in claim 1 or 2, in which the boron compound has the formula



4. Gasoline as claimed in claim 1 or 2, in which the boron compound is one of the boron compounds hereinbefore specifically referred to.

ELKINGTON AND FIFE,
Consulting Chemists and
Chartered Patent Agents,
Bank Chambers, 329 High Holborn, *mg.*
London, W.C.1,
Agents for the Applicants.

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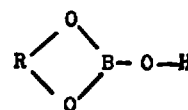
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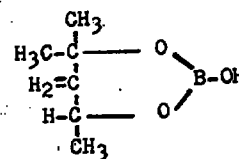
1. Gasoline to which has been added up to 6 cc. per United States gallon of tetraethyl lead, a halide scavenging agent, and at least one boron compound of the general formula:



wherein R is an alpha or beta alkalene radical having 3 to 20 carbon atoms, the amount of said compound added to the gasoline being equivalent to 0.002 to 0.1% by weight boron.

2. Gasoline as claimed in claim 1, in which the scavenging agent is one theory of ethylene dibromide per theory of tetraethyl lead.

3. Gasoline as claimed in claim 1 or 2, in which the boron compound has the formula



4. Gasoline as claimed in claim 1 or 2, in which the boron compound is one of the boron compounds hereinbefore specifically referred to.

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