PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

Improvements in and relating to Fuels for Internal Combustion Engines.

We, "SHELL" REFINING AND MARKETING COMPANY LIMITED, a British Company of St. Helen's Court, Great St. Helen's, London, E.C.3, ROBERT GRAHAM, PH.D., of 4, Meadows Way, Upton-by-Chester, and ALUN LEWIS, B.SC., of 84, Overleigh Road, Handbridge, Chester, both British Subjects, 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 :—

This invention relates to fuels for internal combustion engines containing tetra-alkyl lead anti-knock agents and to compositions for the production of such fuels.

Fuels for internal combustion engines frequently contain a tetra-alkyl lead anti-knock agent such as tetra-ethyl lead and to such fuels it is usual to add also a further compound for the purpose of scavenging from the combustion chamber the lead compounds produced in the combustion of the fuels. These further compounds are commonly termed "scavengers" and typical compounds of this class are halohydrocarbons such as ethylene dibromide and ethylene dichloride. These halohydrocarbons are used with a view to avoiding the deposition of lead oxides and metallic lead in the combustion chamber by the formation of lead halides volatile at the combustion temperature which would be swept out with the gaseous products of combustion. In actual practice, however, the formation of lead oxyhalides and lead oxides as well as the volatile lead halides cannot be avoided and deposits of lead compounds are found in the combustion chamber even when such scavengers are employed. Among other deleterious effects such combustion deposits tend to increase the octane requirement of the engine. Further adverse effects which accompany the use of leaded gasolines containing conventional scavengers are spark plug fouling and corrosion, for example, of exhaust valves.

[*Price 2s. 8d.*]

It has now been found that the above-mentioned defects arising from the use of conventional leaded gasolines can be avoided by the use of an additional and different type of scavenger which appears to function by converting the lead present in the fuel into compounds of such high melting points that they cannot fuse on the surfaces of the combustion chamber and of such a fine state of sub-division that they are swept out of the combustion chamber with the gaseous products of combustion.

The additional scavengers used in accordance with the present invention are completely esterified organic esters of oxyacids of phosphorus or arsenic containing at least one carbocyclic radical. They are used in conjunction with the halohydrocarbon type scavengers and their use results in a reduction of exhaust valve corrosion and of spark plug fouling and considerably less increase in octane requirement over the running life of the engine.

Accordingly the present invention provides fuels for internal combustion engines which comprise hydrocarbons of the gasoline boiling range containing a minor proportion of a tetra-alkyl lead, a minor proportion of a completely esterified organic ester of an oxy-acid of phosphorus or arsenic containing at least one carbocyclic radical and a minor proportion of a halohydrocarbon scavenger.

Further the present invention provides compositions suitable for addition to hydrocarbon fuels for internal combustion engines which comprise a mixture of a tetra-alkyl lead, a completely esterified organic ester of an oxyacid of phosphorus or arsenic containing at least one carbocyclic radical and a halohydrocarbon scavenger. These compositions can be blended into gasolines to give the fuels of the present invention and they may contain the usual dyes used in tetra-alkyl lead fluids in order to indicate their poisonous nature or for identification purposes. Other ingredients such as sol-

vents, oxidation inhibitors and so on may also be present if desired.

The organic esters suitable for use in accordance with this invention are preferably derived from the oxyacids of phosphorus, such as ortho-, pyro- or meta-phosphoric acid and phosphorous or hypophosphorous acid, by replacement of all the hydrogen atoms in the acid by organic radicals, preferably hydrocarbon radicals, at least one of which is a carbocyclic radical. The organic radicals, including the carbocyclic radical, may contain substituent groups or atoms if desired. These organic esters are generally oily liquids or low melting point solids soluble in petroleum hydrocarbons. They may be used singly or in admixture. Typical examples of such compounds are the triphenyl-, tricresyl-, tri-(4-ethylphenyl)-, tri-(4-tertiary butyl phenyl)-, trixylenyl-, tri-2-naphthyl-, tri-(4-isopropyl-1-naphthyl)-, tribenzyl-, triphenylethyl-, tricyclohexyl-, tri-(trichlorophenyl)- and tri-(4-chlorobenzyl)-orthophosphates, di-isoamyl cyclohexyl-orthophosphate, dibutyl phenyl-orthophosphate, 2-naphthyl diphenyl-orthophosphate, 2-naphthyl-3¹-methylphenyl-pheuyl-orthophosphate and -triphenylphosphite. Commercial tricresyl orthophosphate is particularly useful as a cheap and readily available compound for use as a scavenger in accordance with this invention.

Compounds analogous to the phosphorus compounds recited above wherein there is an arsenic atom in place of the phosphorus atom can also be employed in accordance with this invention although in general the phosphorus compounds are preferred because of their easy preparation and cheapness. Typical of such analogous compounds are tri-*o*-, -*m*- or -*p*-cresyl arsenite and diethyl phenylarsonate.

As mentioned above the novel scavengers of the present invention are used in conjunction with halohydrocarbon scavengers. These halohydrocarbon scavengers include all the halohydrocarbons known to be useful as scavengers for leaded fuels, for example, ethylene dibromide or dichloride, acetylene tetrabromide, 2,2,3-trichlorobutane, 2-chloro-2,3-dibromobutane, and the dibromotoluenes. Mixtures of these halohydrocarbons, such as a mixture of ethylene dibromide and ethylene dichloride, may also be used. In general halohydrocarbons having a vapour pressure in the range 0.1 mm. to 100 mm. mercury at 50° C. are capable of being so used. It is desirable that the combined scavenger contains not more than about 90% by weight of the halohydrocarbon. With higher percentages the disadvantages of that type of scavenger are not avoided.

In general the quantity of scavengers required in the fuel is that necessary to convert (theoretically) all the lead present as tetra-alkyl lead into the corresponding lead halide and lead salt of the oxyacid concerned. (Such a quantity is throughout this Specification and in the claims hereinafter appearing referred as "1 theory.") Greater or smaller quantities may be employed in some circumstances depending upon the particular components of the fuel and the type of engine in which it is intended to be used. In fact one of the merits of the present invention is that it allows greater variation in the quantity of scavenger employed than is permissible when a halohydrocarbon is used as the sole scavenger. Even small excesses of halohydrocarbon scavengers are detrimental in such circumstances whereas with the scavengers of this invention a substantial excess can be tolerated without adverse results. The preferred range of total scavenger content of the fuels of this invention is from 0.9 to 1.2 theories.

The use of the scavengers of this invention not only results in a significant modification in the natute of the combustion chamber deposits as compared with those produced when the conventional halohydrocarbon scavengers are used alone, but also results in a considerable decrease in corrosion, for example of the exhaust valves, and in improved spark plug electrode condition.

In order to illustrate the effect of the ester scavengers of this invention used in conjunction with a conventional halohydrocarbon scavenger a Franklin 4 AC-176 engine was operated upon a 100/130 grade aviation gasoline fuel containing 4 mls. of tetra-ethyl lead per gallon and 1 theory of ethylene dibromide, using a simulated cruise test, and the elapsed time before spark plug fouling, as evidenced by mis-firing, was noted. The test was then repeated under the same operating conditions, but 0.2 theories of various scavengers were incorporated in the above fuel in addition to the 4 mls. tetra-ethyl lead and 1 theory ethylene dibromide. The results obtained are presented in Table I:

TABLE I.

Added Scavenger (0.2 Theory).	% Increase in Plug Fouling Time.
None	0
Ethylene dibromide	25
Triphenyl phosphite	225
Tri-*p*-cresyl phosphite	>480
Tri-*o*-cresyl orthophosphate	>1100

A surprising and unexpected feature of the present invention is the fact that a carbocyclic radical is necessary in the ester scavenger to obtain substantial improvements in performance. Thus, it has been established that the corresponding esters containing only non-cyclic organic radicals fail to achieve the freedom from spark plug fouling attained by the present invention. Thus, in the Franklin engine tests described above using the same fuel containing 4 mls. of tetra-ethyl lead per gallon and 1 theory of ethylene dibromide the addition of 0.2 theories of various ester scavengers gave the following results:

TABLE II.

Added Scavenger (0.2 theory).	Time, in hours, before plug fouling occurred.
Tributyl phosphite	9½
Triethyl phosphite	11¼
Isoamyl octyl-orthophosphate	10½
Tri-*o*-cresyl orthophosphate	Ran for over 29 hours without fouling
Tri-*p*-cresyl phosphite	Ditto

Further the ester scavengers containing no carbocyclic radical adversely affect the anti-knock rating of fuels in which they are incorporated whereas the ester scavengers of this invention have practically no such effect. Thus the addition of 0.2 theories of tricresyl orthophosphate to a gasoline containing 5.5 ml. tetraethyl lead and 1 theory of ethylene dibromide per gallon and having an F2 Octane Number of 99 caused no decrease in the said Octane Number. The addition of 0.2 theories of tributyl orthophosphate to such a gasoline, however, lowered the Octane Number to 98, a significant drop in an aviation fuel.

It is well-known that internal combustion engines running on leaded fuel show an increase with time in the octane rating of the fuel required if freedom from knocking is to be maintained. The modification in the nature of the combustion chamber deposits resulting from the use of the leaded fuels of this invention leads to a much lower increase in this octane requirement. This advantage was illustrated in tests in a Petter engine which was run firstly on a gasoline containing 1.05 mls. of tetraethyl lead per gallon and 1 theory of ethylene dibromide and secondly on the same fuel containing additionally 0.2 theories of tricresyl orthophosphate. Each fuel was tested under identical conditions and the duration of each test was 70 hours. The octane requirement of the engine at the commencement and at the end of each test was determined. The results are set out in Table III.

TABLE III.

Scavenger in fuel.	Increase in octane requirement after 70 hours.
1 theory ethylene dibromide	15.8
1 theory ethylene dibromide plus 0.2 theories tricresyl orthophosphate	9

Both the fuels and the compositions suitable for addition to fuels provided by this invention may advantageously contain further compounds known to exert a stabilising and/or anti-oxidising effect on hydrocarbons of the gasoline boiling range. In this connection, 2,4-dimethyl-6-tertiarybutyl phenol is a particularly useful additive, but other stabilising compounds such as hydroquinone, 2,6-ditertiary-butyl-4-methyl phenol, N-phenyl-α-naphthylamine and N,N¹-dibutyl-*p*-phenylendiamine may be employed.

While the invention has been described with special reference to tetra-ethyl lead, it can be applied equally well to other tetra-alkyl lead anti-knock agents, such as ethyl-trimethyl lead, diethyl-dimethyl lead, triethylmethyl lead and tetramethyl lead. The invention is not, however, applicable to gasolines containing iron carbonyl as the anti-knock agent.

The fuels of this invention may be compounded by simply mixing the ingredients in any order. Thus the ester scavenger may be added to a leaded gasoline which already contains a halohydrocarbon scavenger. Alternatively the ester scavenger may be incorporated in a tetra-alkyl lead fluid containing a halohydrocarbon scavenger and the whole composition blended into the fuel. Again the ester scavenger may be added to an

unleaded gasoline and the tetra-alkyl lead fluid containing a halohydrocarbon scavenger added subsequently.

What we claim is :—

1. A fuel for internal combustion engines which comprises hydrocarbons of the gasoline boiling range containing a minor proportion of a tetra-alkyl lead, a minor proportion of a completely esterified organic ester of an oxyacid of phosphorus or arsenic containing at least one carbocyclic radical and a minor proportion of a halohydrocarbon scavenger.

2. A fuel as claimed in Claim 1 wherein the tetra-alkyl lead is tetra-ethyl lead.

3. A fuel as claimed in Claim 1 or 2 wherein the organic ester is an organic ester of orthophosphoric acid or of phosphorous acid.

4. A fuel as claimed in any of the preceding claims wherein the organic radicals of the ester are hydrocarbon radicals at least one of which is carbocyclic.

5. A fuel as claimed in Claim 4 wherein the ester is tricresyl orthophosphate or trixylenyl orthophosphate.

6. A fuel as claimed in any of the preceding claims wherein the halohydrocarbon scavenger is ethylene dibromide, ethylene dichloride or a mixture thereof.

7. A fuel as claimed in any of the preceding claims wherein the organic ester plus halohydrocarbon scavenger is present to the extent of 0.9 to 1.2 theories.

8. A fuel as claimed in any of the preceding claims which contains additionally a further compound known to exert a stabilising and/or anti-oxidising effect on hydrocarbons of the gasoline boiling range.

9. Fuels containing a tetra-alkyl lead, a completely esterified organic ester of an oxyacid of phosphorus or arsenic containing at least one carbocyclic radical and a halohydrocarbon scavenger substantially as hereinbefore described.

10. A composition suitable for addition to hydrocarbon fuels for internal combustion engines which comprises a mixture of a tetra-alkyl lead, a completely esterified organic ester of an oxyacid of phosphorus or arsenic containing at least one carbocyclic radical and a halohydrocarbon scavenger.

11. A composition as claimed in Claim 10 wherein the tetra-alkyl lead is tetra-ethyl lead.

12. A composition as claimed in Claim 10 or 11 wherein the organic ester is an organic ester of orthophosphoric acid or of phosphorus acid.

13. A composition as claimed in Claim 12 wherein the organic ester is tricresyl orthophosphate or trixylenyl orthophosphate.

14. A composition as claimed in any of Claims 10 to 13 inclusive wherein the halohydrocarbon scavenger is ethylene dibromide, ethylene dichloride or a mixture thereof.

15. A composition as claimed in any of Claims 10 to 14 inclusive, which contains additionally a dye, solvent or oxidation inhibitor.

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London, E.C.3.

PROVISIONAL SPECIFICATION.

Improvements in and relating to Fuels for Internal Combustion Engines.

We, "SHELL" REFINING AND MARKETING COMPANY LIMITED, a British Company, of St. Helen's Court, Great St. Helen's, London, E.C.3, ROBERT GRAHAM, PH.D., of 4, Meadows Way, Upton-by-Chester, and ALUN LEWIS, B.SC., of 84, Overleigh Road, Handbridge, Chester, both British Subjects, do hereby declare this invention to be described in the following statement :—

This invention relates to fuels for internal combustion engines containing tetra-alkyl lead anti-knock agents and to compositions for the production of such fuels.

Fuels for internal combustion engines frequently contain a tetra-alkyl lead anti-knock agent such as tetra-ethyl lead and to such fuels it is usual to add also a further compound for the purpose of scavenging from the combustion chamber the lead compounds produced in the combustion of the fuels. These further compounds are commonly termed "scavengers" and typical compounds of this class are organic halides such as ethylene dibromide and ethylene dichloride. These organic halides are used with a view to avoiding the deposition of lead oxides and metallic lead in the combustion chamber by the formation of lead halides volatile at the combustion temperature which would be swept out with the gaseous products of combustion. In actual practice, however, the formation of lead oxyhalides and lead oxides as well as the volatile lead halides cannot be avoided and deposits of lead compounds are found in the combustion chamber even when such scavengers are employed.

unleaded gasoline and the tetra-alkyl lead fluid containing a halohydrocarbon scavenger added subsequently.

What we claim is :—

1. A fuel for internal combustion engines which comprises hydrocarbons of the gasoline boiling range containing a minor proportion of a tetra-alkyl lead, a minor proportion of a completely esterified organic ester of an oxyacid of phosphorus or arsenic containing at least one carbocyclic radical and a minor proportion of a halohydrocarbon scavenger.

2. A fuel as claimed in Claim 1 wherein the tetra-alkyl lead is tetra-ethyl lead.

3. A fuel as claimed in Claim 1 or 2 wherein the organic ester is an organic ester of orthophosphoric acid or of phosphorous acid.

4. A fuel as claimed in any of the preceding claims wherein the organic radicals of the ester are hydrocarbon radicals at least one of which is carbocyclic.

5. A fuel as claimed in Claim 4 wherein the ester is tricresyl orthophosphate or trixylenyl orthophosphate.

6. A fuel as claimed in any of the preceding claims wherein the halohydrocarbon scavenger is ethylene dibromide, ethylene dichloride or a mixture thereof.

7. A fuel as claimed in any of the preceding claims wherein the organic ester plus halohydrocarbon scavenger is present to the extent of 0.9 to 1.2 theories.

8. A fuel as claimed in any of the preceding claims which contains additionally a further compound known to exert a stabilising and/or anti-oxidising effect on hydrocarbons of the gasoline boiling range.

9. Fuels containing a tetra-alkyl lead, a completely esterified organic ester of an oxyacid of phosphorus or arsenic containing at least one carbocyclic radical and a halohydrocarbon scavenger substantially as hereinbefore described.

10. A composition suitable for addition to hydrocarbon fuels for internal combustion engines which comprises a mixture of a tetra-alkyl lead, a completely esterified organic ester of an oxyacid of phosphorus or arsenic containing at least one carbocyclic radical and a halohydrocarbon scavenger.

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14. A composition as claimed in any of Claims 10 to 13 inclusive wherein the halohydrocarbon scavenger is ethylene dibromide, ethylene dichloride or a mixture thereof.

15. A composition as claimed in any of Claims 10 to 14 inclusive, which contains additionally a dye, solvent or oxidation inhibitor.

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This invention relates to fuels for internal combustion engines containing tetra-alkyl lead anti-knock agents and to compositions for the production of such fuels.

Fuels for internal combustion engines frequently contain a tetra-alkyl lead anti-knock agent such as tetra-ethyl lead and to such fuels it is usual to add also a further compound for the purpose of scavenging from the combustion chamber the lead compounds produced in the combustion of the fuels. These further compounds are commonly termed "scavengers" and typical compounds of this class are organic halides such as ethylene dibromide and ethylene dichloride. These organic halides are used with a view to avoiding the deposition of lead oxides and metallic lead in the combustion chamber by the formation of lead halides volatile at the combustion temperature which would be swept out with the gaseous products of combustion. In actual practice, however, the formation of lead oxyhalides and lead oxides as well as the volatile lead halides cannot be avoided and deposits of lead compounds are found in the combustion chamber even when such scavengers are employed.

Moreover, not only do these organic halides fail to scavenge all the lead from the combustion chamber, but their use fails to eliminate serious corrosion problems.

It has now been found that the above mentioned defects arising from the use of organic halides as scavengers can be avoided by the use of a different type of scavenger which functions by converting the lead present in the fuel into compounds of such high melting points that they cannot fuse on the surfaces of the combustion chamber and of such a fine state of sub-division that they are swept out of the combustion chamber with the gaseous products of combustion.

The scavengers used in accordance with the present invention are organic esters of oxyacids of phosphorus containing at least one carbocyclic radical.

Accordingly the present invention provides fuels for internal combustion engines which comprise hydrocarbons of the gasoline boiling range containing a small amount of a tetra-alkyl lead and a small amount of an organic ester of an oxyacid of phosphorus containing at least one carbocyclic radical.

Further the present invention provides compositions suitable for addition to fuels for internal combustion engines which comprise a mixture of a tetra-alkyl lead and an organic ester of an oxyacid of phosphorus containing at least one carbocyclic radical. These compositions can be blended into gasolines to give the fuels of the present invention and they may contain the usual dyes used in tetra-alkyl lead fluids in order to indicate their poisonous nature or for identification purposes. Other ingredients such as solvents, inhibitors and so on may also be present if desired.

The organic phosphorus compounds suitable for use in accordance with this invention are derived from the oxyacids of phosphorus, such as ortho-, pyro- or meta-phosphoric acid and phosphorous or hypophosphorous acid by replacement of the hydrogen atoms in the acid by organic radicals, preferably hydrocarbon radicals, at least one of which is a carbocyclic radical. They are generally oily liquids or low melting point solids soluble in petroleum hydrocarbons. They may be used singly or in admixture. Typical examples of such compounds are the triphenyl-, tricresyl-, tri-(4-ethylphenyl)-, tri-(4-tertiary butyl phenyl)-, trixylenyl-, tri-2-naphthyl-, tri-(4-isopropyl-1-naphthyl)-, tribenzyl-, triphenyl ethyl-, tricyclohexyl-, tri-(trichlorophenyl)-, and tri-(4-chlorobenzyl)-orthophosphates, di-isoamyl cyclohexyl-ortho-phosphate, dibutyl phenyl-ortho-phosphate, 2-naphthyl diphenyl-orthophosphate, 2-naphthyl-3¹-methylphenyl-phenyl-orthophosphate and triphenyl-phosphite. Commercial tricresylorthophosphate is particularly useful as a cheap and readily available compound for use as a scavenger in accordance with this invention.

In general the quantity of organic phosphorus compound required in the fuel is that necessary to convert (theoretically) all the lead present as tetra-alkyl lead into the corresponding lead salt of the phosphorus oxyacid concerned, but greater or smaller quantities may be employed in some circumstances depending upon the particular components of the fuel and the type of engine in which it is intended to be used.

The use of the scavengers of this invention not only results in a significant reduction in the weight of combustion chamber deposits as compared with that arising when the conventional organic halide scavengers are used, but also results in a considerable decrease in corrosion, for example of the exhaust valves, and in improved spark plug electrode condition. These advantages were illustrated in tests in a Petter engine (the word "Petter" is a Registered Trade Mark) which was run firstly on a gasoline containing 2·5 millilitres of tetra-ethyl lead per gallon together with sufficient ethylene dibromide to convert (theoretically) all the lead to lead bromide and secondly under identical conditions except that in place of the ethylene dibromide the fuel contained sufficient tricresyl-orthophosphate to convert (theoretically) all the lead to lead orthophosphate. The length of each run was 40 hours after which the engine was opened up and examined. The results are compared in the following Table. There was no significant difference in the power produced by the engine in the two runs.

	Ethylene dibromide as scavenger.	Tricresyl orthophosphate as scavenger.
Exhaust valve condition	Poor	Light powdery deposit which left valve "as new" on being brushed off.
Wgt. of deposit on Piston Crown	4.83 grms	3.25 grms.
Cylinder head	9.00 "	5.62 "
Nature of deposits	Fused, hard and difficult to remove	Unfused, powdery and easily brushed off.
Condition of spark plug electrode	Poor	Good.

Both the fuels and the compositions suitable for addition to fuels provided by this invention may advantageously contain further compounds known to exert a stabilising and/or anti-oxidising effect on such fuels and compositions. In this connection, 2,4-dimethyl-6-tertiarybutyl phenol is a particularly useful additive, but other stabilising compounds such as hydroquinone and 2,6-ditertiary-butyl-4-methyl phenol may be employed.

Further it is possible to use the scavengers of this invention in conjunction with conventional scavengers such as ethylene dibromide, or dichloride, acetylene tetrabromide, 2,2,3-trichlorobutane, 2-chloro-2,3-dibromobutane and the dibromo-toluenes.

While the invention has been described with special reference to tetra-ethyl lead, it can be applied equally well to other tetra-alkyl lead anti-knock agents, such as ethyl-trimethyl lead, diethyl-dimethyl lead, triethylmethyl lead and tetramethyl lead.

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SPECIFICATION NO. 683405

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By a direction given under Section 17(1) of the Patents Act 1949 this application proceeded in the name of "Shell" Refining & Marketing Company Limited, a British company of St. Helens Court, Great St. Helens, London, E.C.3.

THE PATENT OFFICE,
4th November, 1952

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ERRATUM

SPECIFICATION NO. 683,405

Page 4, line 46, *for* "hydrocarbon duels" *read* "hydrocarbon fuels".

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