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(54) Title: USE OF ALCOHOLS IN FUELS FOR SPARK IGNITION ENGINES

(57) Abstract: The use of at least one C<sub>4</sub> alcohol to increase the vapour pressure of a fuel for spark-ignition engines comprising base fuel and ethanol in which use the at least one C<sub>4</sub> alcohol is added to a fuel comprising base fuel and ethanol to produce a resultant fuel mixture comprising 15-50 % by volume of base fuel, 15-65 % by volume of ethanol and 15 to 50 % C<sub>4</sub> alcohol. The resultant fuel mixture exhibits an adequately high vapour pressure, for example, which meets the specification, and also exhibits a good start behaviour even at winter temperatures.

### USE OF ALCOHOLS IN FUELS FOR SPARK IGNITION ENGINES

The invention relates to the use of alcohols in fuels for spark-ignition engines and to the manufacture of fuels for spark ignition engines. In addition to ethanol, these fuels also comprise C<sub>4</sub> alcohol.

5 Fuels for spark-ignition engines are engine fuels which are suitable for engines with spark ignition. They usually contain a mixture of different hydrocarbons with different boiling points, which typically lie within a range from 26 °C to 210 °C at atmospheric pressure. However, this range is not specifically predetermined, and may be dependent on the actual composition of the hydrocarbons, the additives and other components, as well as  
10 on the environmental conditions. Typically, the hydrocarbon components of the fuels contain C<sub>4</sub> to C<sub>10</sub> hydrocarbons.

The requirements for spark-ignition fuels which must be fulfilled in their production are multifaceted. They are the result of laws and regulations, the distribution chain, and their use in the engine. Added to these are production-specific conditions and  
15 different drive concepts, which make a differentiation of the spark-ignition fuel into different types necessary. In Germany, minimum requirements are imposed by decrees in the form of DIN Standards. These must be identifiable by appropriate identification at the filling station. The Fuel Quality Decree introduced in 1993 and updated in 2004 and 2006, now only allows fuels which fulfil all the requirements of the DIN Standard EN228. In the  
20 USA, corresponding specifications are imposed by ASTM 4814.

In order to achieve optimum driving conditions, the fuel should fulfil various different requirements of the vehicle. In specific terms, this means: After a long cold night the engine should start just as easily as it does in summer, that is, as if the well-heated engine is restarted after a brief intermediate stop. Also, the engine should not stall when  
25 idling, and should not work in an erratic manner under high loads. Even under unfavourable conditions there should be no interfering residues in the aspiration system, combustion chamber, or in the oil sump.

The fuel should be adapted to these different, and in part contradictory, requirements in such a way that a trouble-free, reliable, and mutually-beneficial interplay is  
30 possible. Brand manufacturers in Germany therefore mix what are referred to as “mineral oil substitute components”, such as alcohols and ethers, to the base fuels in limited proportions, as well as chemical active substances up to 0.5 percent by weight.

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Particular requirements are specified for fuels with alcohol contents from 0 to 85 % by volume of ethanol, for use in what are referred to as Flex-Fuel Vehicles. With what are referred to as E85 fuels, with 70 to 85% by volume of ethanol, the vapour pressures for the cold start behaviour is particularly critical. Fuels with a high alcohol content, such as ethanol or mixtures of different alcohols are known. Mention may be made by way of example of the Applications HK 106 428, US 2006/0137243, US 2004/0107634, and US 2004/123518.

It is also known to mix a refinery-typical "super benzine" or gasoline having a Research Octane Number (also referred to as RON) of 95 in summer or winter quality with 70 to 85% by volume of bioethanol. A draft specification CWA 15293 in accordance with the CEN Workshop Agreement of May 2005 and the Draft Standard E DIN 51625 (October 2007, Beuth Verlag) recommends a minimum of 35 kPa vapour pressure for the summer months (Class A) and 50 kPa vapour pressure for winter months (Class B). Depending on the vapour pressure of the base super benzine or gasoline fuel, a vapour pressure of the gasoline/ethanol mixture may not correspond to the draft specification. In these cases, the ethanol concentration in the mixture must be adjusted downwards. This is undesirable.

An aspect of the invention is therefore to provide a fuel with high alcohol concentration which exhibits a sufficiently high vapour pressure, for example, a pressure which meets the specification. This is intended to enhance cold start behaviour, for example to guarantee that the fuel exhibits good cold start behaviour even under winter temperatures.

This aspect is resolved by the use of at least one C<sub>4</sub> alcohol according to the invention described herein.

The base fuel is a conventional gasoline, for example, an unleaded gasoline, for example such as is offered in Germany and elsewhere in Europe with a RON of 95.

The minimum standards for unleaded gasoline, which has been on offer at filling stations since 1984 as Normal, and additionally since 1985 as Euro-Super and since 1989 as SuperPlus, were specified in 1993 in the European Standard DIN EN 228. The composition of spark-ignition fuels from German refineries in the qualities Normal, Super, and Super-Plus, is to be found, for example, in the DGMK Research Report 502-1 by the

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German Scientific Association for Mineral Oil, Natural Gas, and Coals (also referred to as DGMK). In this analysis the main constituents of the fuel were determined, specifically as

both the mean values as well as the ranges. The basis for this analysis was the winter product from the winter 2001/2002. Reference is made herein expressly to this report. Specifically, the content was determined of 50 hydrocarbons with 3 to 6 carbon atoms, 51 aromatics, and 3 oxygen containing compounds. In addition, the total content of all  
5 representatives with the same carbon content in each case was determined for all paraffins, naphthenes, cyclic and acyclic olefins and aromatics. Separately tested was the content of 10 diolefins and 15 polycyclic aromatics. The spark-ignition fuels referred to were derived from 14 different refineries.

10 The resultant fuel according to the invention contains 15 to 50% by volume of such a base fuel.

The inclination of the gasoline to evaporation – its volatility – is the central precondition for use as a spark-ignition fuel, and at the same time is an essential quality feature. Because gasoline is a mixture of many hydrocarbons it does not have a defined boiling point, but a boiling range, which lies usually between 30 °C and 200 °C.

15 The fuel according to the invention comprises 15 to 65% by volume of ethanol and 15 to 50% by volume of C<sub>4</sub> alcohol. In total, naturally, including any other components, if present, the sum total of 100% by volume is always derived.

In principle, due to a high content of alcohols the properties of the fuel are altered in comparison with a normal fuel. One such property is, for example, the volatility.

20 The volatility is characterised by the boiling curve in a temperature band of, in most cases, 30 to 200 °C, maximum from 26 to 210 °C, and by the vapour pressure. The boiling curve describes the proportion of evaporated fluid at different temperatures; the vapour pressure is the result of the fuel components which at a defined temperature in a closed container convert from the fluid phase into the vapour phase.

25 The function of “evaporated gasoline fractions/temperature” provides what is referred to as the boiling curve, the location and characteristics of which allow persons skilled in the art to reach conclusions regarding the behaviour of the fuel in the engine. In principle, the volatility of the spark-ignition fuel must be established in such a way that in all situations an ignitable fuel-air mixture is available in the combustion chamber. Under  
30 specific operating conditions, such as, for example, with a particularly cold or particularly hot engine, this precondition is hard to fulfil, such that quality differences in the fuels become identifiable in the light of this criterion.

The ignitability of the mixture is influenced not only by the fuel, but also by the engine concept. That is to say, there is a difference as to whether the engine is being driven with “rich” or “lean” mixtures.

For winter operation, the volatility of the spark-ignition fuel is in general adapted to the cold ambient temperatures. For a reliable cold start, the fuel should be as volatile as possible. A low boiling curve in the lower range and a high vapour pressure facilitate starting and warm-up of the engine, since a too low volatility would lead to the mixture becoming lean due to insufficient evaporation and excessive fuel condensation on the walls of the intake manifold.

The requirements on the gasoline with a hot engine are precisely the reverse. Under unfavourable conditions, components of the fuel system can become so hot that a too large part of the fuel evaporates (“vapour phase formation” in the fuel pump), boils out of the float chamber of the carburettor, or forms vapour cushions in the injection system. As a result, the fuel delivery is interrupted and the mixture is over-enriched, which has a negative effect on driving behaviour. These disturbances arise more often, the hotter the fuel system becomes (pump, carburettor, and fuel injection), and the lower the fuel delivery volume and system pressure are. On the fuel side, an excessively high volatility makes itself evident to disadvantageous effect. High ambient temperatures in the summer increase the likelihood of faults. For this reason, the fuel manufacturers blend their summer qualities in such a way that the boiling curve in the lower range is high and the vapour pressure is low compared to winter quality fuel. This adjustment must not go so far, however, that difficulties arise with the cold start.

Depending on the base fuel, in particular with mixtures of fuels with a high ethanol content, the minimum vapour pressure to be maintained according to the draft specification often cannot be maintained, in particular for the winter product. The consequence would be that the ethanol content would have to be lowered in order to achieve the minimum vapour pressure.

Surprisingly, it has been found that, by mixing fuel comprising base gasoline and ethanol (for example E85) with at least one C<sub>4</sub> alcohol, for example, such as may be present in other fuels having a high concentration of C<sub>4</sub> alcohols, an increase in the mixture vapour pressure can be achieved which lies above that of the individual fuels. In the

laboratory, vapour pressures (according to EN 13016-1) of various different ethanol and butanol mixtures in the super gasoline were tested out.

The results are represented in Tables 1 and 2. The number following the letters characterising the individual combination in each case indicate the content of this  
5 combination in % by volume.

The requirements referred to heretofore are fulfilled with the fuel according to the invention, and in particular the cold start properties are improved.

In principle, it can be determined that with mixtures of a base fuel with a relatively high proportion of ethanol, an atypical increase in the vapour pressure occurs if at least one  
10 C<sub>4</sub> alcohol is added to this mixture, such that its content amounts to at least 15 % by volume in the resultant fuel mixture. Such an increase in the vapour pressure would not be foreseeable for the person skilled in the art. The effect is noted among fuels which comprise after mixing, 15 to 50 % by volume base fuel, 15 to 65 % by volume ethanol, and 15 to 50 % by volume C<sub>4</sub> alcohol. The effect is especially marked among fuels which  
15 comprise after mixing, 20 to 46 % by volume base fuel, 20 to 60 % by volume ethanol, and 20 to 50 % by volume C<sub>4</sub> alcohol and most particularly amongst fuels which comprise after mixing, 19.5 to 44 % by volume base fuel, 21 to 59.5 % by volume ethanol, and 21 to 49 % by volume C<sub>4</sub> alcohol. This effect is particularly perceptible when the C<sub>4</sub> alcohol is n-butanol (also referred to as 1-butanol), secondary butanol (also referred to as 2-butanol) or  
20 isobutanol (also referred to as 2-methyl-1-propanol), or a mixture of at least two of these butanols.

From an analysis of the boiling behaviour (distillation according to EN ISO 3405), a positive effect was determined on mixing a fuel containing ethanol with a fuel containing butanol. The distillation curves, running in plateau fashion in the range of about 20 to 90  
25 % of evaporated volume, show in the mixtures according to the invention, a constant rise and therefore a path which is similar to that of a spark-ignition fuel.

The effects referred to are particularly favourable with regard to the vapour pressure and also the boiling behaviour if the C<sub>4</sub> alcohol is an isobutanol.

With the ethanol contained in the fuel according to the invention, it is in principle  
30 not of consequence for the properties of the fuel from what sources the ethanol derives. For preference, however, an ethanol is to be used which is at least predominantly of natural origin, such as from biomass, because on combustion, this ethanol develops carbon dioxide

which is environmentally neutral. For the same reason it is preferable for the C<sub>4</sub> alcohol used to be likewise of predominantly natural origin.

The overall content of alcohols of the fuel according to the invention is at least 50 % by volume, but for preference at least 70 % by volume.

5 As already mentioned, the base fuel is a fuel such as unleaded gasoline for example which is commercially available at the present time. This consists of a mixture of hydrocarbons with a Carbon-number of predominantly from 4 to 12. Its main components are predominantly paraffins, naphthenes, olefins, and aromatics. In addition to this, the base fuel may comprise components containing oxygen. In this situation, ethers are  
10 preferred. The ether content can for preference amount to up to 15 % by volume related to the base fuel. As ethers well-suited is tertiary butyl methyl ether (also referred to as MTBE), as well as tertiary butyl ethyl ether (also referred to as ETBE) or mixtures thereof.

The invention further relates to a method for the manufacture of the resultant fuel according to the invention, as well as its use as a fuel for spark-ignition engines, in  
15 particular for spark-ignition engines in motor vehicles.

For preference, the resultant fuel according to the invention is manufactured from already existing fuel mixtures. One of these mixtures is an E85 fuel already described, with a proportion of 70 to 85 % by volume of ethanol and 15 to 30 % by volume of base fuel (mixture (I)). The other mixture comprises 30 to 50 % by volume of base fuel and 50  
20 to 70 % by volume of C<sub>4</sub> alcohol (mixture (II)). The preferred mixing ratio of mixture I : II is 1:1. Mixture I may be an E70 or E85 gasoline and mixture II may be a B50 or a B70 gasoline. Preferred are mixtures of a mixture (I) having 70 % by volume of ethanol with a mixture (II) having 50 % by volume of C<sub>4</sub> alcohol, in particular isobutanol. Preferably, the ratio of mixture (I) to mixture (II) is in the range from 40:60 to 60:40, but in particular  
25 50:50 or 1:1 respectively. Mixture (I), however, may comprise up to 85 % by volume of ethanol and mixture (II) up to 70 % by volume of C<sub>4</sub> alcohol.

Basically, however, a C<sub>4</sub> alcohol can not only be added, as described, as a mixture with a base fuel to a mixture of a base fuel with a high content of ethanol, but it is also possible for C<sub>4</sub> alcohol to be added in a high concentration (up to 100%) to the latter  
30 mixture. In the final analysis, the concentration ratios in the resultant mixtures are determinant. The invention therefore further relates to the use of at least one C<sub>4</sub> alcohol to increase the vapour pressure of a fuel for spark-ignition engines comprising base fuel and

ethanol in which use the at least one C<sub>4</sub> alcohol is added to a fuel comprising base fuel and ethanol to produce a resultant fuel mixture comprising 15-50 % by volume of base fuel, 15-65 % by volume of ethanol and 15 to 50 % C<sub>4</sub> alcohols. The % by volume data relate in each case to the resultant fuel mixture.

5 The resultant fuel mixture preferably has a water concentration of less than 800 ppm by weight, for example in the range 100 to 400 ppm by weight.

The invention will now be illustrated by way of example only, by reference to the data in Tables 1 and 2. Vapour pressures measured in the laboratory according to EN 13016-1, of various different ethanol and butanol mixtures in super gasoline were  
10 measured and are shown in Tables 1 and 2.

Differences between expected vapour pressures, as calculated according to the relative volume amounts of the components, and actual vapour pressures are also shown. The expected vapour pressure was calculated according to the relative proportions by volume of the components. For example, the expected vapour pressure of a 70 vol %  
15 iB50/30 vol % E70 mixture was calculated as 70% of the vapour pressure of iB50 plus 30 % of the vapour pressure of E70. The data in the Tables show that the use of at least one C<sub>4</sub> alcohol increases the vapour pressure of the fuel comprising base fuel and ethanol beyond that which would be expected according to the summing in proportion to their amounts, of the vapour pressures of the various gasoline/ethanol and gasoline/butanol  
20 mixtures. Thus, for example, 1B50 was found to have a vapour pressure of 56.1 kPa, 2B50 a vapour pressure of 55.7 kPa, iB50 a vapour pressure of 55.6 kPa, iB70 a vapour pressure of 40.7 kPa, E70 a vapour pressure of 56.1 kPa and E85 a vapour pressure of 40.0 kPa. The vapour pressures of various 50:50 (vol/vol) mixtures of these butanol and ethanol  
25 mixtures were measured and found to be greater than the average of the vapour pressures of the component mixtures. Thus, the C<sub>4</sub> alcohol had increased the vapour pressure of the fuel mixture.

Also shown in Tables 1 and 2 is the difference in vapour pressure between expected and measured for the various ratios of E70/iB50 mixtures and E85/iB70 mixtures, the expected value being calculated according to the relative proportions by volume of the two  
30 mixtures.

The increase in vapour pressure caused by the C<sub>4</sub> alcohol is shown in the Tables.

Table 1

		Absolute measurement results				
B50/B70 concentration [%]	E70/E85 concentration [%]	E70+1B50 Vapour pressure [kPa]	E70+2B50 Vapour pressure [kPa]	E70+iB50 (2 <sup>nd</sup> ) Vapour pressure [kPa]	E85+iB70 Vapour pressure [kPa]	E70+iB50 (1st) Vapour pressure [kPa]
100	0	56.1	55.7	55.6	40.7	58.9
70	30			58.1	42.1	
60	40			58.5	42.3	
50	50	58.8	58.2	58.5	42.2	61.1
40	60			58.4	42.1	
30	70			58.0	41.7	
0	100	56.1	56.1	55.8	40.0	57.9

		Differences in relation to calculated vapour pressure according to relative volume proportions				
B50/B70 concentration [%]	E70/E85 concentration [%]	E70+1B50 Vapour pressure [kPa]	E70+2B50 Vapour pressure [kPa]	E70+iB50 (2 <sup>nd</sup> ) Vapour pressure [kPa]	E85+iB70 Vapour pressure [kPa]	E70+iB50 (1st) Vapour pressure [kPa]
70	30			2.46	1.65	
60	40			2.84	1.91	
50	50	2.70	2.30	2.83	1.88	2.70
40	60			2.71	1.84	
30	70			2.30	1.51	

E = Ethanol  
 1B = 1-butanol  
 2B = 2-butanol  
 iB = Isobutanol  
 50-85 = Alcohol content [% by volume]  
 gasoline = RON95 unleaded gasoline, "Super"

Table 2

	Ethanol [% by volume]	C4 alcohol [% by volume]	Gasoline [% by volume]	Total [% by volume]	Vapour pressure difference between expected and measured [kPa]	Vapour pressure absolute measurement [kPa]
<b>1B50</b>	0	50	50	100	-	56.1
<b>E70 + 1B50 (1:1)</b>	35	25	40	100	2.70	58.8
<b>E70</b>	70	0	30	100	-	56.1
<b>2B50</b>	0	50	50	100	-	55.7
<b>E70 + 2B50 (1:1)</b>	35	25	40	100	2.30	58.2
<b>E70</b>	70	0	30	100	-	56.1
<b>iB50 (2nd)</b>	0	50	50	100	-	55.6
<b>E70 + iB50 (2nd)</b>	21	35	44	100	2.46	58.1
<b>E70 + iB50 (2nd)</b>	28	30	42	100	2.84	58.5
<b>E70 + iB50 (2nd)</b>	35	25	40	100	2.83	58.5
<b>E70 + iB50 (2nd)</b>	42	20	38	100	2.71	58.4
<b>E70 + iB50 (2nd)</b>	49	15	36	100	2.30	58.0
<b>E70 (2nd)</b>	70	0	30	100	-	55.8
<b>iB70</b>	0	70	30	100	-	40.7
<b>E85 + iB70</b>	25.5	49	25.5	100	1.65	42.1
<b>E85 + iB70</b>	34	42	24	100	1.91	42.3
<b>E85 + iB70</b>	42.5	35	22.5	100	1.88	42.2
<b>E85 + iB70</b>	51	28	21	100	1.84	42.1
<b>E85 + iB70</b>	59.5	21	19.5	100	1.51	41.7
<b>E85</b>	85	0	15	100	-	40.0
<b>iB50 (1st)</b>	0	50	50	100	-	58.9
<b>E70 + iB50 (1st)</b>	35	25	40	100	2.70	61.1
<b>E70 (1st)</b>	70	0	30	100	-	57.9

E = Ethanol  
 iB = Isobutanol  
 1<sup>st</sup> = First measurement series  
 1B = 1-butanol  
 50-85 = Alcohol content [% by volume]  
 2<sup>nd</sup> = Second measurement series  
 2B = 2-butanol  
 gasoline = RON95 unleaded gasoline, "Super"

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Further, any prior art reference or statement provided in the specification is not to be taken as an admission that such art constitutes, or is to be understood as constituting, part of the common general knowledge in Australia.

5 Where the terms "comprise", "comprises", "comprised" or "comprising" are used in this specification, they are to be interpreted as specifying the presence of the stated features, integers, steps or components referred to, but not to preclude the presence or addition of one or more other feature, integer, step, component or group thereof.

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The Claims defining the invention are as follows:

1. Use of at least one C<sub>4</sub> alcohol to increase the vapour pressure of a fuel for spark-ignition engines comprising base fuel and ethanol in which use the at least one C<sub>4</sub> alcohol is added to a fuel comprising base fuel and ethanol to produce a resultant fuel mixture comprising 15-50 % by volume of base fuel, 15-65 % by volume of ethanol and 15 to 50 % C<sub>4</sub> alcohol.
2. Use according to claim 1, in which said resultant fuel mixture comprises:
  - 20 to 46 % by volume of a base fuel;
  - 20 to 60 % by volume of ethanol; and
  - 20 to 50 % by volume of C<sub>4</sub> alcohol.
3. Use according to claim 1, in which said resultant fuel mixture comprises:
  - 19.5 to 44 % by volume of a base fuel;
  - 21 to 59.5 % by volume of ethanol; and
  - 21 to 49 % by volume of C<sub>4</sub> alcohol.
4. Use according to any one of claims 1 to 3, in which the C<sub>4</sub> alcohol is one or more of n-butanol, secondary butanol, and isobutanol.
5. Use according to any one of claims 1 to 3, in which the C<sub>4</sub> alcohol is isobutanol.
6. Use according to any one of claims 1 to 5, in which the ethanol is predominantly of natural origin.
7. Use according to any one of claims 1 to 6, in which the C<sub>4</sub> alcohol is predominantly of natural origin.
8. Use according to any one of claims 1 to 7, in which the base fuel comprises predominantly a mixture of hydrocarbons with a Carbon-number predominantly from 4 to 12.
9. Use according to claim 8, in which the mixture of hydrocarbons comprises predominantly paraffins, naphthenes, and aromatics.
10. Use according to claim 8 or claim 9, in which the base fuel comprises 0-15 % by volume of ether.
11. Use according to claim 10 in which the ether is tertiary butyl methyl ether and/or tertiary butyl ethyl ether.

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12. Use according to any one of claims 1 to 11, in which the alcohol content of the resultant fuel mixture is at least 50 % by volume.
13. Use according to claim 12, in which the alcohol content of the resultant fuel mixture is at least 70 % by volume.
14. Use according to any one of claims 1 to 13, wherein the resultant fuel mixture comprises a mixture of mixture I, comprising 15-30 % by volume of a base fuel and 70-85 % by volume of ethanol with a mixture II, comprising 30-50 % by volume of base fuel and 50-70 % by volume of C<sub>4</sub> alcohol.
15. Use according to claim 14 in which mixture I and mixture II are mixed in the ratio of 1:1 by volume.
16. Use according to claim 14 or claim 15, in which mixture I is an ethanol fuel of up to 85 % by volume of ethanol gasoline blend and mixture II is a butanol fuel blend of up to 70 % by volume of butanol gasoline blend.
17. Use according to any one of claims 1 to 16, substantially as hereinbefore described with reference to any of the accompanying Examples.