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PROCESS OF DELEADING GASOLINE

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Fig. 2

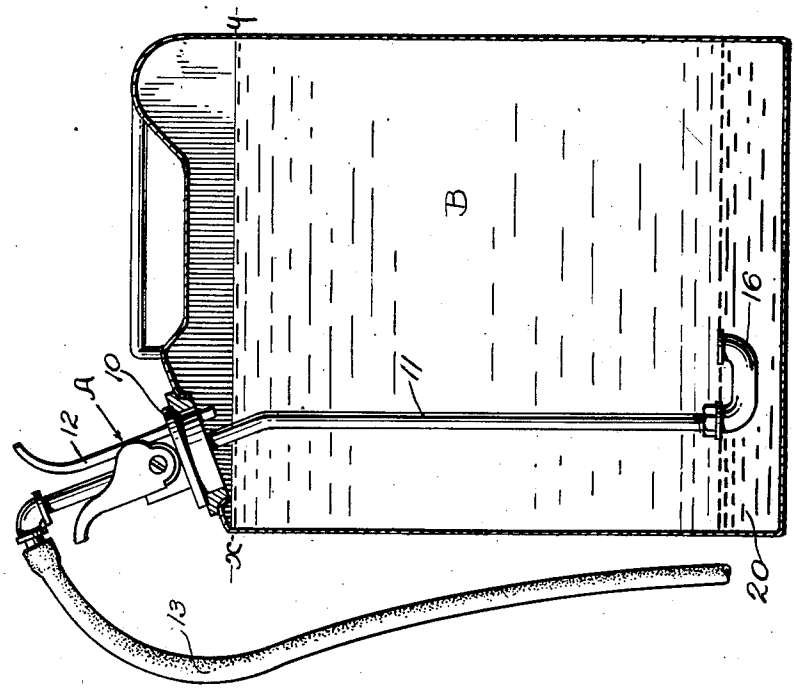
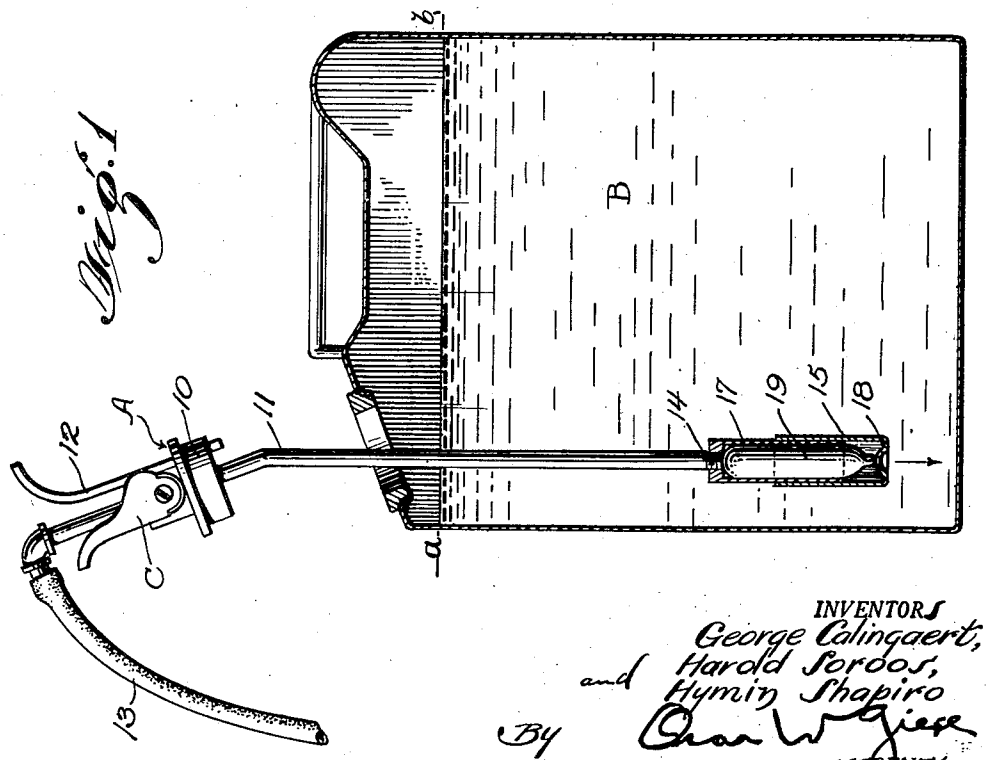


Fig. 1



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PROCESS OF DELEADING GASOLINE

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7 Claims. (Cl. 196—44)

The invention described herein, if patented, may be manufactured and used for or by the Government for governmental purposes, without the payment to us of any royalty thereon.

The invention relates to a process of deleading gasoline by means of stannic chloride and to equipment for use in carrying out the process.

More particularly the invention contemplates the treatment of a batch of leaded motor gasoline with a definite quantity of stannic chloride in a frangible capsule followed by a water wash and decanting by siphoning.

It is a matter of common knowledge that practically all gasoline commercially available at the present time contains tetraethyl lead. For certain purposes the use of leaded gasoline is inadvisable and in such cases an effort must be made to find straight or white gasoline, which frequently involves considerable difficulty. In the armed forces of the United States, particularly in foreign theatres of war, unleaded gasoline is ordinarily not available because it cannot be supplied separately in any practical way. When unleaded gasoline is needed for some special purpose the Army personnel must delead the leaded gasoline that is used for vehicles, planes, etc.

One of the important instances in which lead-free gasoline is required is in cooking units of all descriptions, from the Army field range to the single burner portable stove of very small size. Army medical regulations forbid the use of leaded gasoline in connection with cooking equipment because of the danger of contamination of food being prepared, cooked or eaten nearby by the lead dust in the vapors coming from the burners. The tendency of fine particles of metallic lead and lead oxide to clog the burners would also make the use of leaded gasoline in the stove units very burdensome if not impossible.

In order that Army units, particularly those in the field, may be able to provide themselves with lead-free gasoline when it is needed, the process presented herein has been devised, which can be practiced with a minimum of materials and equipment and which produces within a comparatively short time a supply of satisfactorily deleaded gasoline.

Another object of the invention is to provide a method of preparing solutions adapted for general use when an active chemical is to be added to a solvent or other substance.

A further object of the invention is to provide apparatus for the practice of the process of deleading gasoline disclosed herein and which is also adapted to facilitate the making of any solution or mixture, especially those in which a violent or unpleasant reaction would occur if the materials were brought into contact in the open.

Other objects of the invention will be apparent from a consideration of the detailed description of the invention, taken in connection with the accompanying drawing, in which

Fig. 1 is a sectional view of the standard gasoline can as used by the Army with the combined closure, ampoule breaker, stirrer and siphon tube;

Fig. 2 is a sectional view of the can and closure assembly in which the ampoule breaker has been replaced by a siphon end fitting, and part of the solution has been siphoned off.

The apparatus devised for the novel method of deleading gasoline consists of an assembly A including a closure or stopper 10 for a standard blitz can B of five gallons capacity as ordinarily used for gasoline in war theatres. The stopper assembly A has locking means C provided for securing it in the outlet of the can B. Extending through the body of the closure 10 is a rigid tubular member 11 which serves a plurality of purposes to be described presently. A vent tube 12 also extends through the stopper 10.

At the upper end of the member 11 a flexible hose 13 is attached, forming a siphon extension of the tubular passage in the member 11 for decanting the contents of the can. The lower end of the member 11 is screw-threaded at 14 to receive either the ampoule breaker 15 as shown in Fig. 1 or the U-shaped extension 16 as shown in Fig. 2. The ampoule breaker 15 consists of a holder or chamber made up of telescoping members 17 and 18. The member 17 is a skeleton or cup-shaped member screwed on the threads 14 of the member 11 and open at the lower end, and 18 is a cup-shaped member or cap telescoped on the member 17. A frangible capsule or ampoule 19 of the chemical to be added is provided, stannic chloride being employed in the process of deleading gasoline, and the ampoule breaker 15 is used in adding the contents of the capsule to the contents of the can. The ampoule 19 is inserted in the chamber formed by the telescoping members, which can be closed only part way with the ampoule in place. The assembly is grasped by the upper end at or near the stopper 10 and the holder 15 is inserted in the can and pressed or struck sharply against the bottom of the can, causing the holder to collapse or close into a more completely telescoped form so as to break the ampoule. The contents of the ampoule

mingle with the contents of the can, and the assembly may be employed to stir the mixture to insure a thorough distribution of the chemical.

If it is desired to decant the solution after a settling period the assembly A can be employed as a siphon. In the particular instance described in connection with the novel process of deleading gasoline, the addition of the stannic chloride is followed by washing with water to remove the excess stannic chloride and the solution is allowed to stand before decanting. It is necessary to leave in the can approximately two quarts of material, which includes water and the precipitates and enough gasoline, about one quart, to insure that no sludge is withdrawn. To decant the deleaded gasoline, the ampoule breaker is unscrewed from the member 11 and the U-shaped pipe extension 16 substituted therefor. The closure may be locked in place by the mechanism C during washing and decanting operation and the length of the member 11 and extension 16 is predetermined to leave the proper amount of residue 20 in the can.

In decanting other solutions the position of the end of the extension 16 would be determined by the relative volumes of the material to be withdrawn and the residue.

If means other than the ampoule breaker is employed to introduce the chemical, the assembly with the siphon extension in place may be used as a stirrer before the closing element is secured in place.

For deleading a batch of gasoline, the following procedure is recommended. With the ampoule breaker 15 attached to the bottom end of the siphon tube 11 and held in the inverted position with the cap 18 removed, a glass ampoule 19 of the deleading agent is inserted into the breaker and the cap is replaced. The ampoule of deleading agent contains 22 cc. of stannic chloride, which is sufficient to delead 5 gallons of gasoline containing up to 3.0 cc. of tetraethyl lead per gallon, the maximum amount likely to be found in motor gasoline in the field. For deleading aviation gasoline, two ampoules of the deleading agent should be used for complete deleading. The tube 11 is then inverted so that the ampoule breaker is in the downward position, and is inserted into a blitz can containing five gallons of gasoline to be deleaded, as illustrated in Figure 1. Holding the tube 11 in the hand near the upper end, the operator brings it down sharply, causing the members 17 and 18 to telescope more completely and breaking the ampoule. Without bringing the breaker above the surface, the gasoline is stirred for two minutes by means of the tube so as to disperse the $SnCl_4$ uniformly throughout the gasoline. A small amount of white fumes may arise from the gasoline during stirring. There is no need to be alarmed at this as a small portion of the reagent in coming into contact with moisture is merely being hydrolyzed, and will not take fire. The ampoule breaker is then removed, and the treated gasoline is allowed to stand for 15 minutes to allow completion of the reaction.

After the standing period, one quart of water is added to the gasoline, the regular cap or the stopper assembly A with the ampoule breaker removed is replaced, and the can and contents are shaken for four minutes. This can probably be done in the field most conveniently by having two men, facing each other, grasp the can B by the handles and bottom, and alternately invert and right the can. This shaking need not be continuous. The can and contents are then allowed to stand for 5 minutes in order for the sludge to settle.

In case the blitz can is so full of gasoline that a quart of water cannot be added without overflow of the gasoline, the can should be tilted slightly by placing a block of wood or other suitable object under the bottom, so that the side nearest the opening is raised an inch or two. In Figure 1 the line a—b denotes the original level of the gasoline, and in Figure 2 the line x—y indicates the level after the water has been added.

While the can is standing after shaking with water, the capsule breaker 15 is unscrewed from the siphon tube, if it has not already been done, and in its place is screwed a small U-pipe 16. The siphon is then inserted into the blitz can and clamped in position, as illustrated in Figure 2. After the 5-minute standing period, the gasoline is now ready for siphoning, which is done by stoppering the air vent 12 of the blitz can and blowing into the flow pipe. The siphon tube is of such length that it will drain all but two quarts of the contents of the can (one quart each of gasoline and water). The siphoned gasoline may now be used for the purpose in mind.

When the stannic chloride is added to the leaded gasoline the following reaction takes place:

$$SnCl_4 + (C_2H_5)_4Pb \rightarrow (C_2H_5)_2SnCl_2 \downarrow + (C_2H_5)_2PbCl_2 \downarrow$$

The diethyllead dichloride and diethyltin dichloride produced are insoluble in gasoline and settle to the bottom of the can.

This reaction is not completed so as to precipitate all the tetraethyl lead except in the presence of an excess of stannic chloride. No more excess stannic chloride than is necessary should be added, however, because the excess stannic chloride promotes the polymerization of the unsaturated hydrocarbons in the gasoline, producing gum formations which are undesirable because they will clog burners, and other devices, in which the deleaded gasoline may be used. It will be obvious, as indicated in the formula, that one mole of stannic chloride combines with one mole of tetraethyl lead. It has been found that a proportion of 2.50 moles of stannic chloride to one mole of tetraethyl lead produces the most satisfactory results, a maximum precipitation of lead with a minimum of gum formation. The use of substantially more than this amount of stannic chloride is to be avoided.

It is necessary to remove the excess stannic chloride from the solution in the gasoline as soon as all the tetraethyl lead has been precipitated, to prevent the formation of excess amounts of the hydrocarbon polymers. To accomplish this enough water is added, in the batch specifically described, one quart, to precipitate the tin as the hydroxide, as

$$SnCl_4 + 4H_2O \rightarrow Sn(OH)_4 \downarrow + 4HCl$$

All the precipitates together with the excess water and other heavy components settle to the bottom of the can and the deleaded gasoline can then be separated by decanting or any desired method.

The siphoned deleaded gasoline will analyze less than 0.3 g. Pb, 0.1 g. Sn, and 0.03 g. HCl per gallon, and up to 300 mg. gum per 100 cc. depending on the particular gasoline used. The siphoned gasoline may have a cloudy appearance because of a small amount of very finely divided suspended sludge. If this is objectionable, the gasoline

may be clarified by filtration through chamois, or by simply allowing the gasoline to stand for 12 hours, then decanting.

Having thus described the invention what we claim and desire to secure by United States Letters Patent is:

1. The process of deleading gasoline by adding stannic chloride to gasoline containing tetraethyl lead and separating the resultant precipitate.

2. The process of deleading gasoline which comprises adding an excess of stannic chloride to gasoline containing tetraethyl lead, removing the unreacted stannic chloride with a water wash and separating the water wash and the resultant precipitate from the gasoline.

3. The process of deleading gasoline which comprises adding an excess of stannic chloride to gasoline containing tetraethyl lead and after an interval adding water to remove the excess of stannic chloride and separating the deleaded gasoline from the water and the resultant sludge.

4. The process of deleading gasoline which comprises adding stannic chloride to gasoline containing tetraethyl lead in the proportion of about 2½ moles of stannic chloride to one mole of tetraethyl lead, washing the thus treated gasoline with water, following the reaction, and separating the deleaded gasoline from the water wash and resultant sludge.

5. The process of deleading gasoline which comprises adding stannic chloride to gasoline containing tetraethyl lead in the proportion of about 2½ moles of stannic chloride to one mole of tetraethyl lead, washing the gasoline with water to remove the excess stannic chloride and separating the deleaded gasoline from the water and resultant sludge.

6. The process of removing tetraethyl lead from gasoline comprising precipitating the tetraethyl lead with stannic chloride.

7. The method of removing tetraethyl lead from gasoline comprising the steps of precipitating the tetraethyl lead from the gasoline with an excess of stannic chloride, removing the unreacted stannic chloride with a water wash and separating the resultant precipitate and water wash from the gasoline.

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