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(21) International Application Number: PCT/GB99/01523 (22) International Filing Date: 13 May 1999 (13.05.99) (30) Priority Data: 9810408.6 15 May 1998 (15.05.98) GB (71) Applicant (for all designated States except US): MACFARLAN SMITH LIMITED [GB/GB]; Wheatfield Road, Edinburgh EH11 2QA (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): RENNER, Bernd [DE/DE]; Alter Postweg 131e, D-21220 Seevetal (DE). SCHMIDT, Ulrich [DE/DE]; Neue Strasse 13, D-21635 Jork (DE). (74) Agent: FITZPATRICKS; 4 West Regent Street, Glasgow G2 1RS (GB).		(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>With amended claims.</i>
(54) Title: HYDROCARBON LIQUID FORMULATION (57) Abstract A liquid hydrocarbon is rendered unpalatable by introducing thereto a denaturant suitable for discouraging the ingestion of the hydrocarbon liquid, but hitherto presenting difficulties in achieving that purpose, particularly with paraffinic liquids such as lamp oil, white spirit, kerosene and other hydrocarbon solvents, wherein the denaturant is a denatonium compound, preferably denatonium benzoate, or an analogue or derivative thereof exhibiting similar organoleptic effects, and using a sufficient amount of a liquid stabiliser miscible with said hydrocarbon liquid, e.g. an alkanol or derivative thereof, to obtain a stable solution or suspension of said denaturant in said liquid hydrocarbon.		

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Hydrocarbon Liquid Formulation

Technical Field

This invention relates to a denaturant formulation suitable for discouraging the ingestion of hydrocarbon liquids particularly paraffinic liquids such as lamp oil, white spirit, kerosene and other hydrocarbon solvents.

Background Art

Liquid hydrocarbons are widely used for various technical purposes and are often available for household purposes, where unguarded use or storage may present risks to children and domestic animals who may confuse them with potable liquids. This is especially so in third world countries where near starving children will consume virtually any liquid in their desperation to quench a thirst. The inadvertent consumption of such liquids by vulnerable individuals is also a high risk because they are available as essentially colourless transparent liquids which are visually indistinguishable from water. The physiological damage arising from deliberate or inadvertent consumption of these liquids is serious and may be fatal. However, it is not practical to keep such substances permanently under secure conditions to avoid unauthorised access or uninformed intervention. Therefore it is desirable to denature the liquids in some way to render them undrinkable or at least to discourage ingestion thereof.

Aversive agents are known which impart a highly bitter taste to substances when mixed therewith. These cause such a bitter taste if an attempt is made to swallow the substance that the substance is rejected immediately, which usually avoids absorption or ingestion of amounts having any lasting harmful effect. Reference may be made to earlier patents including GB-A-955 309, for quaternary salts and compositions

containing them, and GB-B-2 212 814 relating to aversive agents for liquid hydrocarbons.

Denatonium benzoate otherwise known as lignocaine benzyl benzoate is cited in the Guinness Book of Records as the most bitter substance known to man, and its preparation and properties are described in GB-A-955 309. It is available commercially under the Registered Trade Mark "**BITREX**". Other denatonium salts are known in the literature. Denatonium saccharide is a bittering agent analogous to denatonium benzoate and is described in US-A-4 652 577.

Liquid hydrocarbons of the type of concern here are predominantly organic liquids of an aliphatic character (alkanes) and mainly recognisable as of the liquid paraffin type but the invention is not restricted to a particular petroleum distillate fraction commonly called paraffin oil. The problem may be explained more generally by an understanding that aliphatic liquids are useful because of their relative chemical inertness. This, for example, makes white spirit useful as a diluent in enamel paints or as a cleaning liquid for removal of certain paints from surfaces without chemically attacking the surface. This predictable non-reactive character and stability, whilst providing desirable properties for marketing products based on such liquids, also presents a problem when seeking to include a denaturant or aversive agent therein. Thus the most preferred denaturant, denatonium benzoate, is extremely insoluble in apolar liquids. Therefore even though that denaturant would exhibit an acceptable level of aversive activity at concentrations as low as 10 ppm it has not proved possible to introduce it directly to liquid hydrocarbons and maintain a satisfactory level of concentration therein for a sufficient period of time even to satisfy a typical shelf life before eventual use. As cited in GB-B-2 212 814, the denaturant does

not form a stable solution in kerosine or the like liquids and tends to migrate rapidly to the container walls.

This problem was addressed in GB-B-2 212 814, by introducing the desired denaturant, and at least one
5 surfactant to the liquid hydrocarbon. This had the reported effect of permitting the desired aversion agent which has a high hydrophilic character but low lipophilic character to function in the desired manner without any inhibition of the rapid organoleptic reaction.

10 The need to introduce surfactants inevitably adds some expense to the product. Furthermore, there is now some doubt as to the effectiveness thereof with all target products, particularly with the higher levels of purity now achievable in comparison with the equivalent products routinely produced
15 commercially over a decade ago. Presently there is tighter control on permissible levels of incidental aromatic contaminants in current commercial products, < 0.1% being achievable for some white oils of the n-paraffin type. It is postulated that the effectiveness of the previous proposal may
20 have been enhanced by such incidental contaminants which facilitated the "solvation" of the small amounts of the preferred denaturant on account of its aromatic components.

Having researched this problem it is now proposed to adopt a different approach to achieving the objective of
25 introducing a sufficient amount of hydrophilic denaturant or aversive agent into a liquid hydrocarbon of an essentially aliphatic character to deter ingestion of physiologically harmful amounts thereof.

Disclosure of Invention

30 According to the present invention this objective is achievable by utilising a stabiliser in the form of a co-solvent which is at least miscible with the target hydrocarbon

but insoluble in water and has sufficient affinity with the selected aversion agent (preferably denatonium benzoate) to permit introduction of effective amounts thereof to the target hydrocarbon with satisfactory stability of the final

5 formulation.

Suitable co-solvents for the purposes of the invention include certain aliphatic or alicyclic alkanols, including diols and diol mono-ethers, esters, and mixtures thereof. A preliminary screening test to confirm or determine suitability of a co-solvent from amongst these classes of solvents would include determining if the solvent under consideration is miscible with the hydrocarbon to be denatured and if it is also capable of dissolving the desired denaturant at a satisfactory level, say 0.1%. Preliminary work suggests that substituted alkanols would be acceptable.

Thus, according to the present invention, there is provided a method for rendering a liquid hydrocarbon unpalatable with an aversive agent having poor solubility in said liquid hydrocarbon, comprising selecting a stabiliser in the form of a co-solvent from amongst aliphatic or alicyclic alkanols and diols, optionally substituted, with ester, ether, ketonic, alicyclic and/or aromatic groups, singularly or as mixtures, said co-solvent being miscible with the liquid hydrocarbon to be denatured and being a solvent in which the aversive agent is soluble, dissolving the aversive agent in the co-solvent and introducing an effective amount of the solution into the liquid hydrocarbon.

Preferred stabilisers include diols and diol mono-ethers, esters, and mixtures thereof particularly branched chain lower secondary alkanol esters of lower fatty acids, especially those formed using a secondary diol. An especially suitable co-solvent is 2,2,4-trimethylpentan-1,3-diol-monoisobutanoate

(CAS-Nr.: 25265-77-4, $C_{12}H_{24}O_3$) which proved to be uniquely suitable for the purpose of the invention.

Alkanols which have not been derivatised have also been observed to be useful for the purposes of the invention with varying degrees of stability. Thus whilst preliminary tests suggest that aliphatic or alicyclic alkanols, i.e. C_2 - C_{20} and above such as 1-butanol and isopropanol, would show varying degrees of stability, it is preferable to select C_6 - C_{20} alkanols, such as 1-octanol, 1-hexadecanol, and mixtures thereof with esters such as 1-octanol/ethyl acetate 1:1 w/w which are now found to be effective in enabling introduction of the preferred aversive agent "BITREX®" at the desired level.

In accordance with the invention a base oil to be denatured is selected, a preferred denaturant such as the aversive agent denatonium benzoate is provided, a stabilising co-solvent therefor is selected from amongst alkanols and aliphatic esters of the aforesaid type, the denaturant is introduced to the co-solvent and dissolved therein e.g. by stirring or similar agitation, to provide a doping medium and thereafter an amount of said doping medium sufficient to have an aversive organoleptic effect is added to the base oil to denature same.

Modes for Carrying Out the Invention

The preparatory work leading to the realisation of the present invention included study of many liquids considered to be potentially suitable for use as co-solvents for the purposes of the invention. The study lead to evaluation of a number of liquids of apparently generally similar chemical and physical properties.

The following Table I summarises the results of this evaluation stage, and in particular assesses the success in

- obtaining a clear stable formulation of the preferred denaturant (denatonium benzoate/ "BITREX"®) in a target commercial product to be denatured (lamp oil). Those combinations marked as "FAIL" were observed to exhibit
- 5 turbidity and insufficient stability in the amount of denaturant introduced to the lamp oil. In each case, the only critical variation lay in the selection of the co-solvent. Apart from the attempted use of rape seed oil, it is assumed that the co-solvent is essentially a single chemical entity.
- 10 The results clearly showed that use of 2,2,4-trimethylpentan-1,3-diol-monoisobutanoate is highly effective in achieving the objective of the invention.

Co-solvent	Assessment
2-Ethylhexyllaurate	FAIL
Isopropyllaurate	FAIL
Isopropyldecanoate	FAIL
2-Ethylhexyl-2-ethylhexanoate	FAIL
Rapeseed Oil	FAIL
2,2,4-trimethylpentan-1,3-diol-monoisobutanoate	PASS

- The successful co-solvent in Table I is commercially
- 15 available under the trade mark TEXANOL® from Eastman Chemical Company (USA).

As an illustrative example of procedure, one may prepare a 40ppm "BITREX®" doped lamp oil by simply stirring in a

previously prepared 0.1% solution of "BITREX®" in the selected co-solvent TEXANOL® to a level of 4% (all amounts described herein are w/w). A similar procedure was found effective in denaturing, white spirit, dearomatised white spirit, and isoparaffin.

Similar tests were conducted using 0.1% Bitrex® in the co-solvents shown in Table II below.

Co-solvent	Pass/Fail	
	Lamp-oil	White Spirit
Texanol®	Pass	Pass
1-Octanol	Pass	Pass
1-Octanol/Ethyl acetate 1:1 w/w	Pass	Pass
Ethyl DL-2-hydroxyhexanoate	Pass	Pass
1-Butanol	Pass	Pass
Isopropanol	Pass	Pass
Ethanol	Pass	Pass
1-Hexadecanol	Pass	np
Ethyl DL-2-hydroxycaproate	Pass	np
Ethyl Acetate	Fail	np
Dichloromethane	Fail	np
Methyl Hexanoate	Fail	np

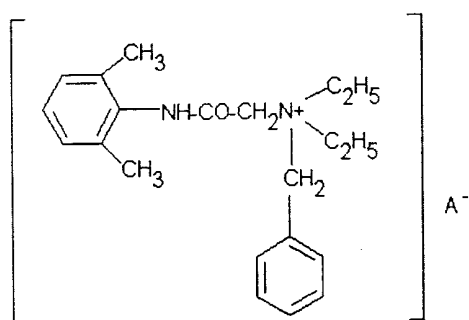
- Pass = clear solution, Fail = cloudy solution,
- np = Test not performed

Industrial Applicability

The fact that it has proved possible to incorporate the preferred denaturing substance at levels effective in achieving an aversive effect in apolar liquid hydrocarbons which are essentially free of aromatic content even upon storage of the treated product without the need to use surfactants or the like additives is surprising and of significant commercial value.

CLAIMS

1. An apolar aliphatic hydrocarbon-based liquid containing,
as an aversion agent, an effective amount of a compound of the
5 formula:-



or an analogue or derivative thereof exhibiting similar
organoleptic effects, wherein A^- is an anion, and a sufficient
amount of a liquid stabiliser whereby a stable solution or
suspension of said agent is formed, said liquid stabiliser
10 being miscible with said hydrocarbon liquid.

2. An apolar aliphatic hydrocarbon-based liquid according to
claim 1, wherein the liquid stabiliser is an alkanol or
derivative thereof.

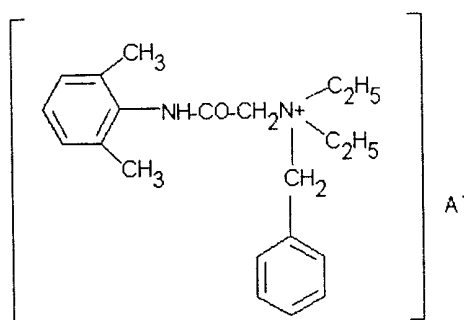
3. An apolar aliphatic hydrocarbon-based liquid according to
15 claim 1 or claim 2, wherein the liquid stabiliser comprises at
least one liquid selected from higher (C_6 and above) aliphatic
or alicyclic alkanols, including diols, optionally substituted
with ester, ether, ketonic, alicyclic and/or aromatic groups,
singularly or together.

4. An apolar aliphatic hydrocarbon-based liquid according to claim 1 or claim 2, wherein the liquid stabiliser is selected from C₆-C₂₀ aliphatic or alicyclic alkanols, including diols, optionally substituted with ester, ether, ketonic, alicyclic and/or aromatic groups, singularly or together.

5. An apolar aliphatic hydrocarbon-based liquid according to claim 1 or claim 2, wherein the liquid stabiliser is selected from 1-butanol, isopropanol, 1-octanol, and a 1:1 (w/w) mixture of 1-octanol and ethyl acetate.

10 6. An apolar aliphatic hydrocarbon-based liquid according to claim 1 or claim 2, wherein the liquid stabiliser is selected from ethyl-DL-2-hydroxyhexanoate and 2,2,4-trimethylpentan-1,3-diol-monoisobutanoate.

15 7. A method of rendering an apolar aliphatic hydrocarbon-based liquid unpalatable comprising, providing such a liquid, selecting an aversive agent of the formula:

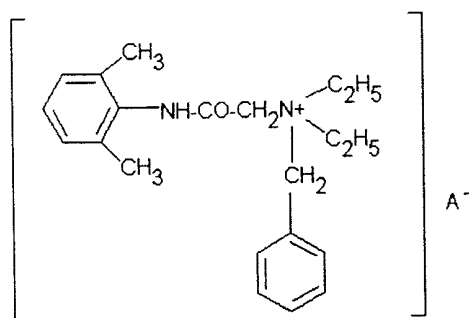


wherein A⁻ is an anion, and providing 2,2,4-trimethylpentan-1,3,-diol monoisobutanoate as a stabiliser for said agent, introducing a predetermined amount of said agent to said stabiliser to form a doping medium therefrom, and adding an

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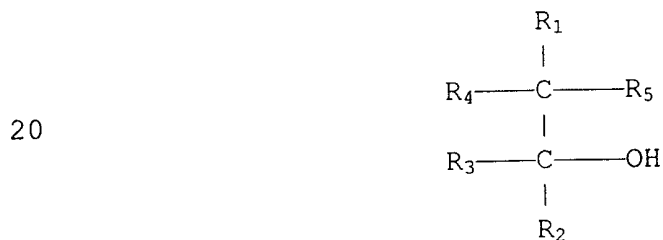
effective amount of that doping medium to said liquid whereby said liquid is rendered unpalatable.

8. A method of rendering an apolar aliphatic hydrocarbon-based liquid unpalatable comprising, providing such a liquid,
5 selecting an aversive agent of the formula:



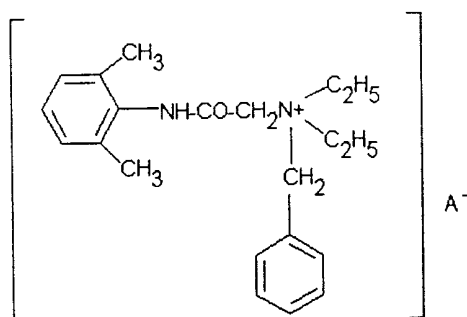
- wherein A^- is an anion, and providing as a stabiliser for said agent at least one liquid selected from higher (C_6 and above) aliphatic or alicyclic alkanols, including diols, optionally substituted with ester, ether, ketonic, alicyclic and/or
10 aromatic groups, singularly or together, introducing a predetermined amount of said agent to said stabiliser to form a doping medium therefrom, and adding an effective amount of that doping medium to said liquid whereby said liquid is rendered unpalatable.

- 15 9. A doping medium comprising at least one stabiliser conforming to the generic formula:-



- where R_1 , R_2 , R_3 , R_4 and R_5 are independently selected from the
25 group consisting of hydrogen, and a substituted or an

unsubstituted, straight or branched chain aliphatic hydrocarbon, wherein each hydrocarbon optionally comprises one or more substituents selected from the group comprising alkanols, esters, ethers, ketones, alicyclic entities and aromatic entities, wherein the number of carbon atoms comprised within R₁, R₂ and R₃ combined is not less than four and does not exceed eighteen, and an aversive agent of the formula:-



wherein A⁻ is an anion in an amount effective for use in a treatment process rendering an apolar aliphatic hydrocarbon liquid unpalatable.

10. A doping medium according to Claim 9, wherein said stabiliser comprises at least one available, i.e. underivatised, hydroxyl group.

11. A doping medium according to Claim 9, wherein said stabiliser is a secondary alkanol ester.

12. A doping medium according to Claim 9 or Claim 10, wherein said stabiliser is a diol.

13. A doping medium according to Claim 9 wherein said stabiliser is 2,2,4-trimethylpentan-1,3,-diol monoiso-butanoate.

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14. A doping medium as claimed in Claim 9 wherein said stabiliser is 1-octanol and ethyl acetate.

15. Use of a doping medium according to any one of claims 9 to 14, in a treatment process rendering an apolar aliphatic hydrocarbon liquid unpalatable.

16. Use according to Claim 15 wherein the hydrocarbon liquid contains less than 0.1% aromatic substances before treatment.

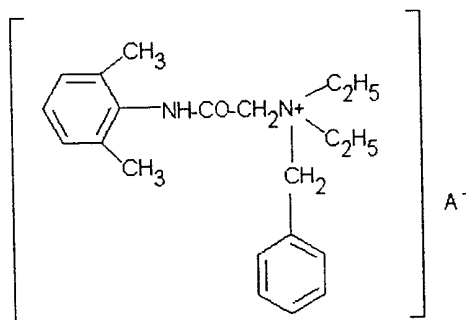
17. Use according to Claim 15 or claim 16, wherein the hydrocarbon liquid is a white oil.

18. Use according to Claim 16 wherein the hydrocarbon liquid is a de-aromatised mixture of n-paraffins.

19. Use according to any one of claims 15 to 18, wherein the aversive agent is sufficiently stabilised to maintain a level of at least 10ppm.

20. Use according to Claim 19 wherein the aversive agent is sufficiently stabilised to maintain a level of at least 40ppm.

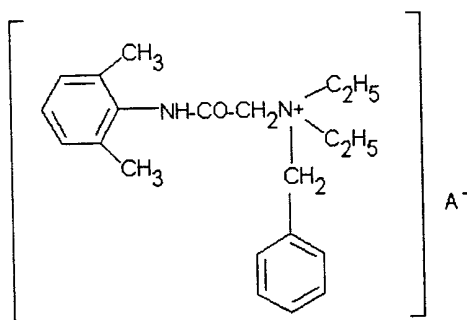
21. An apolar aliphatic hydrocarbon liquid containing a compound of the formula:



wherein A⁻ is an anion, as an aversive agent, and as a stabiliser, a sufficient amount of 2,2,4,-trimethylpentan-1,3-

diol-monoisobutanoate to sustain an organoleptically detectable aversive effect by said aversive agent for at least 6 months.

22. An apolar aliphatic hydrocarbon liquid containing a compound of the formula:

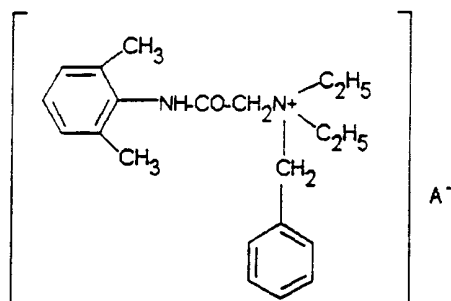


wherein A⁻ is an anion, as an aversive agent, and 2,2,4-trimethylpentan-1,3-diol-monoisobutanoate as a stabiliser being in the range of from 0.1 to 25% (w/w).

AMENDED CLAIMS

[received by the International bureau on 2 November 1999 (02.11.99);
original claims 2 and 3 cancelled; original claim 1 amended;
original claim 5 amended and renumbered as claim 3 original claims
4 and 6-22 renumbered as claims 2 and 4-20 (6 pages)]

1. An apolar aliphatic hydrocarbon-based liquid containing,
as an aversion agent, an effective amount of a compound of the
5 formula:-



or an analogue or derivative thereof exhibiting similar
organoleptic effects, wherein A^- is an anion, and a sufficient
amount of a liquid stabiliser whereby a stable solution or
suspension of said agent is formed, said liquid stabiliser
10 being miscible with said hydrocarbon liquid, and comprising at
least one liquid selected from higher (C_6 and above) aliphatic
or alicyclic alkanols, including diols, optionally substituted
with ester, ether, ketonic, alicyclic and/or aromatic groups,
singularly or together.

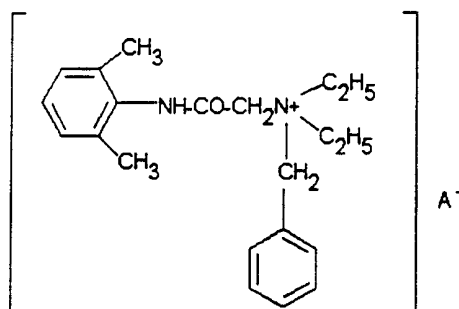
- 15 2. An apolar aliphatic hydrocarbon-based liquid according to
claim 1, wherein the liquid stabiliser is selected from C_6 - C_{20}
aliphatic or alicyclic alkanols, including diols, optionally
substituted with ester, ether, ketonic, alicyclic and/or
aromatic groups, singularly or together.

- 20 3. An apolar aliphatic hydrocarbon-based liquid according to
claim 1 or claim 2, wherein the liquid stabiliser is selected

from 1-octanol, and a 1:1 (w/w) mixture of 1-octanol and ethyl acetate.

4. An apolar aliphatic hydrocarbon-based liquid according to claim 1 or claim 2, wherein the liquid stabiliser is selected from ethyl-DL-2-hydroxyhexanoate and 2,2,4-trimethylpentan-1,3-diol-monoisobutanoate.

5. A method of rendering an apolar aliphatic hydrocarbon-based liquid unpalatable comprising, providing such a liquid, selecting an aversive agent of the formula:

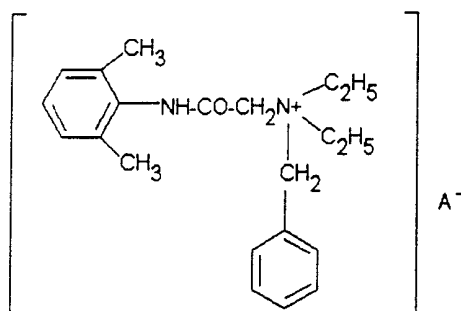


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wherein A^- is an anion, and providing 2,2,4-trimethylpentan-1,3,-diol monoisobutanoate as a stabiliser for said agent, introducing a predetermined amount of said agent to said stabiliser to form a doping medium therefrom, and adding an effective amount of that doping medium to said liquid whereby said liquid is rendered unpalatable.

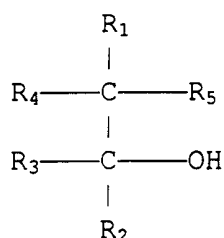
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6. A method of rendering an apolar aliphatic hydrocarbon-based liquid unpalatable comprising, providing such a liquid, selecting an aversive agent of the formula:



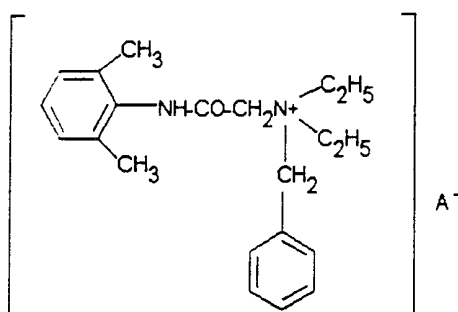
wherein A^- is an anion, and providing as a stabiliser for said agent at least one liquid selected from higher (C_6 and above) aliphatic or alicyclic alkanols, including diols, optionally substituted with ester, ether, ketonic, alicyclic and/or aromatic groups, singularly or together, introducing a predetermined amount of said agent to said stabiliser to form a doping medium therefrom, and adding an effective amount of that doping medium to said liquid whereby said liquid is rendered unpalatable.

7. A doping medium comprising at least one stabiliser conforming to the generic formula:-



where R_1 , R_2 , R_3 , R_4 and R_5 are independently selected from the group consisting of hydrogen, and a substituted or an unsubstituted, straight or branched chain aliphatic hydrocarbon, wherein each hydrocarbon optionally comprises one or more substituents selected from the group comprising

alkanols, esters, ethers, ketones, alicyclic entities and aromatic entities, wherein the number of carbon atoms comprised within R₁, R₂ and R₃ combined is not less than four and does not exceed eighteen, and an aversive agent of the
 5 formula:-



wherein A⁻ is an anion, in an amount effective for use in a treatment process rendering an apolar aliphatic hydrocarbon liquid unpalatable.

8. A doping medium according to Claim 7, wherein said
 10 stabiliser comprises at least one available, i.e. underivatised, hydroxyl group.

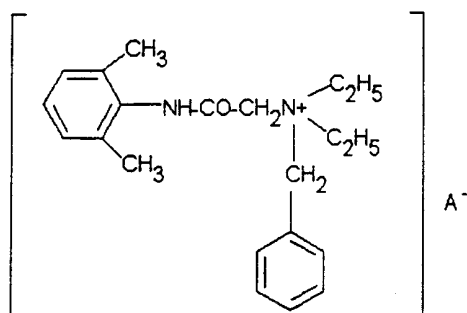
9. A doping medium according to Claim 7, wherein said stabiliser is a secondary alkanol ester.

10. A doping medium according to Claim 7 or Claim 8, wherein
 15 said stabiliser is a diol.

11. A doping medium according to Claim 7 wherein said stabiliser is 2,2,4-trimethylpentan-1,3,-diol monoiso-butanoate.

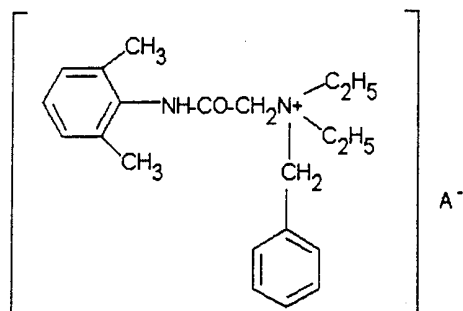
12. A doping medium as claimed in Claim 7 wherein said
 20 stabiliser is 1-octanol and ethyl acetate.

13. Use of a doping medium according to any one of claims 7 to 12, in a treatment process rendering an apolar aliphatic hydrocarbon liquid unpalatable.
14. Use according to Claim 13 wherein the hydrocarbon liquid contains less than 0.1% aromatic substances before treatment.
15. Use according to Claim 13 or claim 14, wherein the hydrocarbon liquid is a white oil.
16. Use according to Claim 14 wherein the hydrocarbon liquid is a de-aromatised mixture of n-paraffins.
17. Use according to any one of claims 13 to 16, wherein the aversive agent is sufficiently stabilised to maintain a level of at least 10ppm.
18. Use according to Claim 17 wherein the aversive agent is sufficiently stabilised to maintain a level of at least 40ppm.
19. An apolar aliphatic hydrocarbon liquid containing a compound of the formula:



- wherein A⁻ is an anion, as an aversive agent, and as a stabiliser, a sufficient amount of 2,2,4,-trimethylpentan-1,3-diol-monoisobutanoate to sustain an organoleptically detectable aversive effect by said aversive agent for at least 6 months.

20. An apolar aliphatic hydrocarbon liquid containing a compound of the formula:



wherein A⁻ is an anion, as an aversive agent, and 2,2,4-trimethylpentan-1,3-diol-monoisobutanoate as a stabiliser
5 being in the range of from 0.1 to 25% (w/w).

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 99/01523

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C10L1/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C10L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	EP 0 884 377 A (SHELL INT. RES.) 16 December 1998 (1998-12-16) the whole document ---	1,2,5
X	US 4 005 038 A (MINKOFF) 25 January 1977 (1977-01-25) example 1 ---	9-13
X	GB 955 309 A (EDINBURGH PHARMACEUTICAL IND.) 15 April 1964 (1964-04-15) cited in the application page 1 - page 2 page 5 ---	1,2
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6 September 1999

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